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DATA GAPS INVESTIGATION SAMPLING AND ANALYSIS PLAN REVISION 1 FOR SITE 19 DERECKTOR SHIPYARD MARINE SEDIMENT NS NEWPORT RI

08/01/2011

TETRA TECH NUS

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

SAMPLING AND ANALYSIS PLAN
(Field Sampling Plan and Quality Assurance Project Plan)
Revision 1 - August 2011

Former Derecktor Shipyard Marine Sediment
Data Gaps Investigation
Naval Station Newport
Newport, Rhode Island

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

Prepared by:
Tetra Tech
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Prepared under:
Contract Number N62470-08-D-1001
CLEAN Contract Task Order No. WE61

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

Document Title: Sampling and Analysis Plan, Former Derecktor Shipyard Offshore Data Gaps Investigation, Naval Station Newport, Newport, Rhode Island, May 2011

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

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

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

Preparation Date (Month/Year): May 2011, Revised August 2011

Investigative Organization's Project Manager:

Signature/Date
Stephen S. Parker, Tetra Tech, Inc.

Investigative Organization's Project Quality Assurance Manager:

Signature/Date
Tom Johnston, PhD, Tetra Tech, Inc.

Lead Organization's Project Manager:

Signature/Date
Winoma Johnson, PE, NAVFAC Mid-Atlantic

Lead Organization QA Officer:

Signature/Date
Government Chemist

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

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

Preparation Date (Month/Year): May 2011

Investigative Organization's Project Manager: 5/26/11
Signature/Date
Stephen S. Parker, Tetra Tech, Inc.

Investigative Organization's Project Quality Assurance Manager: 5-26-11
Signature/Date
Tom Johnston, PhD, Tetra Tech, Inc.

Lead Organization's Project Manager: JOHNSON.WINOMA.A.1229628980 Digitally signed by JOHNSON.WINOMA.A.1229628980 DN: c=US, o=U.S. Government, ou=DoD, ou=PKI, ou=USN, cn=JOHNSON.WINOMA.A.1229628980 Date: 2011.05.27 07:35:00 -04'00'
Signature/Date
Winoma Johnson, PE, NAVFAC Mid-Atlantic

Lead Organization QA Officer: Digitally signed by TUCKERJONATHAN.P.1239524180 Date: 2011.05.26 16:22:42 -04'00'
Signature/Date
Government Chemist

EXECUTIVE SUMMARY

This Sampling and Analysis Plan (SAP) has been prepared to support sampling activities for the marine sediment adjacent to the Former Robert E. Derecktor (Derecktor) Shipyard located at the Naval Station Newport (NAVSTA), Newport, Rhode Island. It presents the technical approach to map out sediment that may have been affected by releases and various industrial practices at Derecktor Shipyard (the Site). Tasks discussed include field sampling procedures and laboratory analytical methods.

The site was used by Robert E. Derecktor Shipyards of Rhode Island Inc. from 1979 until 1992 to repair, maintain, and construct private and military ships. Repair and maintenance operations consisted of sandblasting and painting, hull inspections, and other on-board ship repairs. The operations were conducted in several buildings on-shore as well as on two floating dry docks and a large ferry (the Greenport Ferry) moored in Coddington Cove adjacent to the shipyard. Construction included cutting and welding steel, sandblasting, priming and painting, and assembling ships. Supporting the ship maintenance and construction operations were an engineering department, and electrical and pipe shop, and a vehicle maintenance shop. Shipyard operations reportedly generated large quantities of hazardous wastes, including waste oil, paints, solvents, thinners, concentrated bases, and other waste solids and liquids; housekeeping practices and hazardous material handling practices at the facility reportedly were poor; and waste materials, including spent sandblast grit and oily liquids from the dry dock, were known to be disposed of on the property.

Marine sediment sampling conducted between 1997 and 1999 identified polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs) and metals in surface sediments near the shoreline and piers. Lower concentrations of contaminants were found in the outer portions of Coddington Cove. Risk assessments were conducted and Preliminary Remediation Goals (PRGs) were established from those risk assessments. Additional sampling conducted in 2004 found similar contaminants in the same locations, but at much lower concentrations, particularly around Pier 1 and near the former Building 234. An FS was drafted, but it was determined after the draft was submitted that the data available was inadequate to support costing and selection of remedial alternatives.

The contaminants in the marine sediment, particularly near Pier 1 and the bulkhead shoreline are likely to be a result of contaminant discharges from ships and from shipyard operations on shore and from vessels and floating dry docks at the site. Subsurface soil and groundwater contamination at the site is limited, and primary sediment contaminants are not present in groundwater, and therefore it is believed that the primary route of contaminant transport from the shipyard operations into the marine sediment is via overland runoff of spilled materials discharging directly into Coddington Cove through the storm drainage

system, and direct release of contaminated materials into the cove from the shoreline, the pier and other work areas.

This investigation of the sediment at the Former Derecktor Shipyard will focus on areas previously sampled, but not well defined: past sample locations are sparse and do not quantify sediment that exceed PRGs to a level that is adequate for development of remedial alternatives in an FS. New stations are based on analysis of the historic dataset and focus on the areas where concentrations were previously found to be highest. The investigation will provide supplemental data to map contaminants of concern (COCs) for which PRGs are established. In addition, other target analytes will be sought for informational purposes as requested by regulatory oversight groups. The sampling to be conducted in these areas will document areas to be identified for remediation under CERCLA.

Areas to be investigated as part of this Data Gaps Investigation include areas where PRGs were exceeded in the past, and areas between these previous sample location, as well as where contaminants were not previously sought due to access issues (under piers and inactive ship berths).

Because the result is to map areas for remediation alternatives, a grid approach was taken for this investigation. Different density of samples have been planned for different areas that are to be investigated: In locations where little or no contamination is suspected to be present samples will be taken on a 200 foot horizontal interval, while in locations of greater concern or greater potential for contamination samples will be taken on a 100 foot horizontal interval. At all locations, samples will be taken through coring at depths of 0-1 foot, 1-2 feet, and 2-4 feet below the sediment surface.

This investigation will provide additional sediment analytical data at the Former Derecktor Shipyard to determine extent of COCs exceeding PRGs, and presence of other target analytes identified by USEPA and RIDEM during planning meetings. The sediment data will fill in gaps identified in the existing dataset. The additional data will be used to support remedial decision-making in portions of the study area that are affected by site related contaminants. Data will be adequate to support remedy selection and design. Potential remedial actions will be fully evaluated in a revision pending for the existing Feasibility Study (FS) for this site prior to selection in a proposed plan and record of decision.

SAP Worksheets

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ACRONYMS

ADC	Acoustic Doppler Current (profiler)
BPRG	Baseline Preliminary Remediation Goal
CCRF	Coddington Cove Rubble Fill (area)
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
CFR	Code of Federal Regulations
CLEAN	Comprehensive Long-Term Environmental Action Navy
COC	Contaminant of Concern
CSM	Conceptual Site Model
CTO	Contract Task Order
DGI	Data Gaps Investigation
DI	Deionized (water)
DL	Detection Limits
DPT	Direct Push Technology
DQO	Data Quality Objective
DSY	Derecktor Shipyard
ERA	Ecological Risk Assessment
ERDC	Engineer Research and Development Center
FOL	Field Operations Leader
FS	Feasibility Study
GPS	Global Position System
HASP	Health and Safety Plan
HQ	Hazard Quotient
HMW	High Molecular Weight
HSM	Health and Safety Manager
IDW	Investigation Derived Waste
IR	Installation Restoration
LOD	Limits of Detection
LOQ	Limits of Quantitation
MIS	Multi-Incremental Sampling
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NAVFAC	Naval Facilities Engineering Command
NAVSTA	Naval Station Newport
ND	None detected, or not detected

NIRIS	Naval Installation Restoration Information Solution
NOAA	National Atmospheric and Oceanic Administration
NOEC	No Observed Effect Concentration
NOS	National Ocean Survey
OSHA	Occupational Safety and Health Administration
PA	Preliminary Assessment
PAH	Polycyclic Aromatic Hydrocarbon
PAL	Project Action Limit
PCB	Polychlorinated Biphenyls
PDF	Portable Document Format
PM	Project Manager
PPE	Personal Protective Equipment
PRG	Preliminary Remediation Goal
PWD	Public Works Department
QAM	Quality Assurance Manager
QC	Quality Control
RIDEM	Rhode Island Department of Environmental Management
RPM	Regional Project Manager
RPRG	Recommended Preliminary Remediation Goal
SAP	Sampling and Analysis Plan
SASE	Study Area Screening Evaluation
SDG	Sample Delivery Group
SOP	Standard Operating Procedure
SSO	Site Safety Officer
TBD	To Be Determined
TBT	Tributyltin
TOC	Total Organic Carbon
UFP-SAP	Uniform Federal Policy, Sampling Analysis Plan
USEPA	United States Environmental Protection Agency

SAP Worksheet #2 -- SAP Identifying Information

(UFP-QAPP Manual Section 2.2.4)

Site Name/Number: IR Site 19, Former Derecktor Shipyard, Naval Station Newport, Newport Rhode Island
Operable Unit: NA
Contractor Name: Tetra Tech
Contract Number: N62470-08-D-1001
Contract Title: Naval Facilities Engineering Command (NAVFAC) Mid-Atlantic Comprehensive Long-Term Environmental Action Navy (CLEAN)
Work Assignment Number: Contract Task Order (CTO) WE61

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

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

3. This SAP is a project-specific SAP.

4. List dates of scoping sessions that were held:

Scoping Session	Date
DQO 1 (Discussion of Path Forward)	October 27, 2010
DQO 2 (Conceptual Sampling Plan Review)	November 11, 2010
DQO 3 (Conceptual Sampling Plan Review 2)	December 14, 2010

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

Title	Date
NA	

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

U.S. EPA, Regulatory Oversight
RIDEM, Regulatory Oversight
NAVSTA, Property Holder
NAVFAC Mid-Atlantic, Responsible Party Representative
Tetra Tech Contractor

7. Lead organization

NAVFAC Mid-Atlantic

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

Not applicable.

SAP Worksheet #3 -- Distribution List

(UFP-QAPP Manual Section 2.3.1)

Name of SAP Recipients	Title	Organization	Telephone Number (Optional)	E-Mail Address or Mailing Address	Document Control Number (Optional)
Winoma Johnson	Remedial Project Manager (RPM)	NAVFAC Mid-Atlantic	757-341-2008	winoma.johnson@navy.mil	NA
Darlene Ward	IR Program Manager	NAVSTA Newport Environmental	401-841-6376	darlene.ward@navy.mil	NA
Dave Barclift	Ecological Risk Assessor	NAVFAC	215-897-4913	david.barclift@navy.mil	NA
Kymberlee Keckler	Remedial Project Manager	US EPA Region 1 Federal Facilities	617-918-1385	keckler.kymberlee@epa.gov	NA
Gary Jablonski	Remedial Project Manager	RIDEM Div Site Remediation	401-222-2797	gary.jablonski@dem.ri.gov	NA
Stephen Parker	Project Manager (PM)	Tetra Tech	978-474-8434	stephen.parker@tetratech.com	NA
Lucy Guzman	Project Chemist	Tetra Tech	978-474-8400	lucy.guzman@tetratech.com	NA
Tom Johnston	Program Quality Assurance Manager (QAM)	Tetra Tech	412-921-8615	tom.johnston@tetratech.com	NA
Michael Horton	Field Operations Leader (FOL)	Tetra Tech	978-474-8454	mike.horton@tetratech.com	NA
Susan Dunnihoo	Laboratory PM	Analytical Resources, Inc. (ARI)	206-695-6207	sue@arilabs.com	NA
Kelly Perkins	Laboratory PM	Katahdin Analytical Services, Inc. (Katahdin)	207-874-2400	kperkins@katahdinlab.com	NA
Joseph Carabillo	Laboratory PM	Test America, Burlington Laboratory (Test America)	802-923-1038	joseph.carabillo@testamerica.com	NA
Stephen Siegel	Laboratory PM	EMSL Analytical, Inc. (EMSL)	856-296-5329	ssiegel@emsl.com	NA
Ann Skradski	Laboratory PM	GEL Laboratories, LLC (GEL)	843-556-8171	team.skradski@gel.com	NA
TBD	Site Safety Officer (SSO)	Tetra Tech	TBD	TBD	NA
Bonnie Capito	Librarian/Records Manager/ Navy Admin Record	NAVFAC Atlantic	757-322-4785	bonnie.capito@navy.mil	NA (Cover Letter Only)
Glenn Wagner	Administrative Record Manager	Tetra Tech	412-320-2211	glenn.wagner@tetratech.com	NA

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

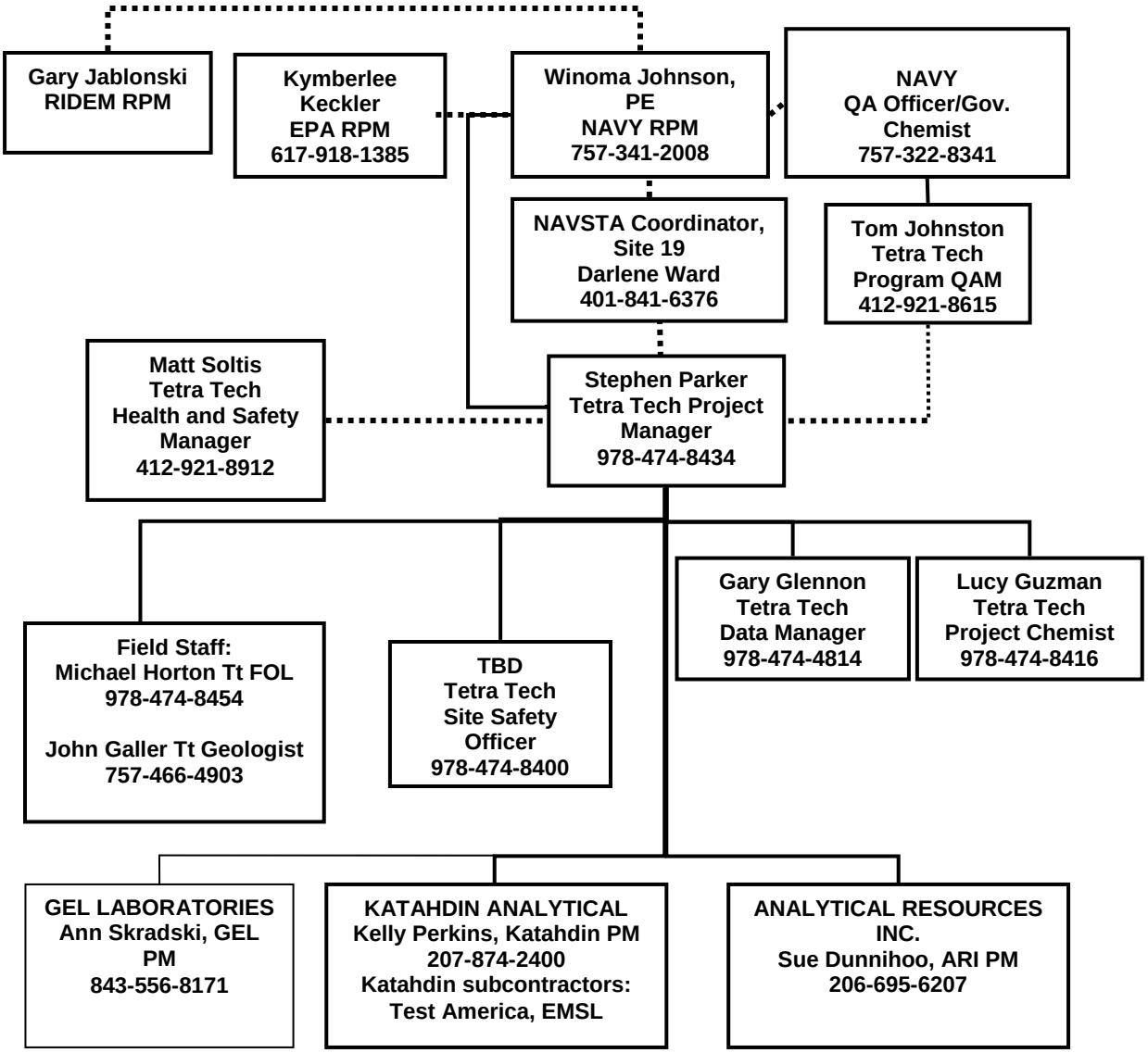
(UFP-QAPP Manual Section 2.3.2)

Name	Organization/Title/Role	Telephone Number	Signature/E-Mail receipt	SAP Section Reviewed	Date SAP Read
Stephen Parker	Tetra Tech/PM	978-474-8434			
Lucy Guzman	Tetra Tech/Project Chemist	978-474-8416			
Michael Horton	Tetra Tech/FOL	978-474-8454		All	
John Galler	Tetra Tech./Project Geologist	757-466-4903		All	
Sue Dunnihoo	ARI	206-695-6207		WS# 15, 19, 20, 23, 24, 25, 28, 30	
Kelly Perkins	Katahdin	207-874-2400		WS# 15, 19, 20, 23, 24, 25, 28, 30	
Joseph Carabillo	Test America	802-923-1038		WS #19, 20, 30	
Stephen Siegel	EMSL	856-296-5329		WS #15, 19, 20, 30	
Ann Skradski	GEL	843-556-8171		WS# 15, 19, 20, 23, 24, 25, 28, 30	
TBD	Tetra Tech/SSO	978-474-8400		All	

SAP Worksheet #5 -- Project Organizational Chart

(UFP-QAPP Manual Section 2.4.1)

Lines of Authority ——— ..... Lines of Communication



SAP Worksheet #6 -- Communication Pathways

(UFP-QAPP Manual Section 2.4.2)

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or E-Mail	Procedure
Changes in schedule	Tetra Tech FOL Tetra Tech PM Navy RPM	Michael Horton Stephen Parker Winoma Johnson	978-474-8454 978-474-8434 757-341-2008	FOL informs PM verbally within same day; PM informs RPM via e-mail within 24 hours and prepares schedule concurrence letter within 7 calendar days, if deemed necessary by the RPM and PM.
Issues in the field that result in changes in scope of field work or SAP Amendments	Tetra Tech FOL Tetra Tech PM Navy RPM	Michael Horton Stephen Parker Winoma Johnson	978-474-8454 978-474-8434 757-341-2008	FOL informs PM verbally within same day; PM informs RPM via e-mail within 24 hours; PM sends a concurrence letter to RPM, if warranted, within 7 calendar days and RPM signs the letter within 5 business days of receipt. Scope change is to be implemented before work is executed. Document the change on a FTMR form (within 2 business days) or SAP amendment (within timeframe agreed to by project team).
Recommendations to stop work and initiate work upon corrective action	Tetra Tech FOL Tetra Tech PM Tetra Tech QAM Tetra Tech SSO Navy RPM	Michael Horton Stephen Parker Tom Johnston TBD Winoma Johnson	978-474-8454 978-474-8434 412-921-8615 978-474-8400 757-341-2008	Responsible party verbally informs the PM, FOL, and subcontractors within 1 hour on recommendation to stop work and within 24 hours of recommendation to restart work. Responsible party follows verbal notification with an e-mail to the Project Team within 24 hours.

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or E-Mail	Procedure
Analytical data quality issues	Laboratory PM's Tetra Tech Project Chemist Tetra Tech PM Navy RPM	Kelly Perkins Susan Dunnihoo Ann Skradski Lucy Guzman Steve Parker Winoma Johnson	207 874-2400 206-695-6207 843-556-8171 978-474-8416 978-474-8434 757-341-2008	The Laboratory PM will notify (verbally or via e-mail) the Tetra Tech Project Chemist within one business day of when an issue related to laboratory data is discovered. The Tetra Tech Project Chemist will notify (verbally or via e-mail) the data validation staff and the Tetra Tech PM within one business day. Tetra Tech Project Chemist notifies Tetra Tech PM verbally or via e-mail within 48 hrs of validation completion that a non-routine and significant laboratory quality deficiency has been detected that could affect this project and/or other projects. The Tetra Tech PM verbally advises the NAVFAC RPM within 24 hours of notification from the Project Chemist. The NAVFAC RPM takes corrective action that is appropriate for the identified deficiency. Examples of significant laboratory deficiencies include data reported that has a corresponding failed tune or initial calibration verification. Corrective actions may include a consult with the NAVFAC Navy Chemist.
Field Work Orientation Meeting	Tetra Tech Project Staff	All Tetra Tech Project Staff	All Tetra Tech Project Staff	Review SAP, HASP, field SOPs, data acquisition sample collection and handling procedures, logistics and operational issues.
Site access and the need to clear utilities	Tetra Tech FOL Tetra Tech PM Navy RPM NAVSTA Coordinator	Michael Horton Stephen Parker Winoma Johnson Darlene Ward	978-474-8454 978-474-8434 757-341-2008 401-841-6376	Anticipated issues are addressed in planning meetings. If issues arise or if plans change during the work efforts, the FOL notifies NAVSTA Coordinator and Tetra Tech PM, who notifies Navy RPM. All meet to resolve and reset the planned activities.

FTMR - Field Task Modification Request

SAP Worksheet #7 -- Personnel Responsibilities and Qualifications Table

(UFP-QAPP Manual Section 2.4.3)

Name	Title/Role	Organizational Affiliation	Responsibilities
Winoma Johnson	RPM	NAVFAC Mid-Atlantic	Oversees project implementation, including scoping, data review, and evaluation.
Kymberlee Keckler	USEPA RPM	USEPA Region I	Participates in scoping, data review, evaluation, and review of the SAP. Oversees project execution for USEPA.
Gary Jablonski	RIDEM RPM	RIDEM	Participates in scoping, data review, evaluation, and review of the SAP. Oversees project execution for RIDEM.
Stephen Parker	PM	Tetra Tech	Oversees financial, schedule, and technical management of the project.
Darlene Ward	NAVSTA Coordinator	Tetra Tech	Coordinates access and local activities for the project.
Michael Horton	FOL	Tetra Tech	As FOL, supervises, coordinates, and performs field sampling activities.
John Galler	Project Geologist	Tetra Tech	Evaluates core recoveries; assimilates geological data, prepares geological interpretation and text.
TBD	SSO/Project Scientist	Tetra Tech	As SSO, will be responsible for on-site project-specific health and safety training and monitoring site conditions related to personnel safety. Details of the SSO's responsibilities are presented in the site-specific Health and Safety Plan (HASP). As Project Scientist, will be responsible for assisting the FOL in field activities.
Tom Johnston	QAM	Tetra Tech	Ensures quality aspects of the CLEAN program are implemented, documented, and maintained.
Lucy Guzman	Project Chemist	Tetra Tech	Participates in project scoping, reviews laboratory scopes of work, and coordinates and oversees the work performed by the subcontracted laboratory. Oversees production of data validation deliverables and compliance with Worksheets #34 to #36 requirements. Responsible for ensuring adherence to all quality assurance/quality control (QA/QC) requirements as defined in this SAP.
Matt Soltis	HSM	Tetra Tech	Oversees Tetra Tech CLEAN Program Health and Safety.
Susan Dunnihoo	Laboratory PM	ARI	Ensures that the laboratory scope of work is followed, coordinates analyses with laboratory chemists, provides QA on data packages, and communicates with Tetra Tech staff.
Ann Skradski	Laboratory PM	GEL	

Name	Title/Role	Organizational Affiliation	Responsibilities
Kelly Perkins	Laboratory PM	Katahdin	Ensures that the laboratory scope of work is followed, coordinates analyses with laboratory chemists, oversees the work performed by the subcontracted laboratory, provides QA on data packages, and communicates with Tetra Tech staff.
Joseph Carabillo Stephen Siegel	Laboratory PM Laboratory PM	Test America EMSL	Ensures that the laboratory scope of work is followed, coordinates analyses with laboratory analysts, provides QA on data packages, and communicates with Tetra Tech's subcontracted laboratory.

SAP Worksheet #8 -- Special Personnel Training Requirements Table

(UFP-QAPP Manual Section 2.4.4)

Each site worker will be required to have completed appropriate Hazardous Waste Operations and Emergency Response (HAZWOPER) training specified in Occupational Safety and Health Administration (OSHA) 29 Code of Federal Regulations (CFR) 1910.120 (e). Project-specific safety requirements are addressed in greater detail in the site-specific HASP.

SAP Worksheet #9a – DQO Participants Sheet (Discussion of Path Forward)

(UFP-QAPP Manual Section 2.5.1)

Project Name:	**Data Gaps Investigation**	Site Name:	**Derecktor Shipyard**
Projected Date(s) of Sampling:	**June 2011**	Site Location:	**Naval Station Newport, Newport Rhode Island**
Project Manager:	**Stephen Parker**		

Date of Session: **10/27/10**

Scoping Session Purpose: **Discussion of How to Move Forward With Assessment of Derecktor Shipyard Offshore Area**

Name	Title	Affiliation	Phone #	E-Mail Address	Project Role
D. Barclift	Ecological Risk Assessor	NAVFAC Atlantic	215-897-4913	david.barclift@navy.mil	Advisory
P. Crump	Staff Scientist	RIDEM	401-222-2797	Pamela.crump@dem.ri.gov	Regulatory
K. Finkelstein	Biologist	NOAA	617-918-1499	ken.finkelstein@noaa.gov	Trustee
T. Finlayson	PM	Gannett Fleming	781-380-7750	rtfinlayson@gfnet.com	Support to USEPA
A. Gavaskar	Sediment Transport scientist	NAVFAC Atlantic	757-322-4730	arun.gavaskar@navy.mil	Advisory
G. Jablonski	RPM	RIDEM	401-222-2797	gary.jablonski@dem.ri.gov	State RPM
M. Horton	FOL	Tetra Tech	978-474-8454	mike.horton@tetratech.com	FOL
N. Johnson		NAVFAC Mid-Atlantic	757-341-2015	nina.johnson@navy.mil	Supervisory
W. Johnson	RPM	NAVFAC Mid-Atlantic	757-341-2008	winoma.johnson@navy.mil	RPM
K. Keckler	RPM	US EPA	617-918-1385	Keckler.kymberlee@epa.gov	RPM
G. Kemp	Engineer	Gannett Fleming	603-625-9116	gkemp@gfnet.com	Support to USEPA
C. Mueller	IR Program Manager	NAVSTA Environmental	401-841-7561	cornelia.mueller@navy.mil	Base Manager
K. Munney	Biologist	US Fish and Wildlife	603-223-2541	kenneth_munney@fws.gov	Trustee
B. Olsen	Section Chief, Federal Facilities	US EPA	617-918-1365	olson.bryan@epa.gov	Supervisory
S. Parker	PM	Tetra Tech	978-474-8434	stephen.parker@tetratech.com	PM

Comments/Decisions:

Navy provided a proposed approach to seek COCs for which PRGs were established and agreed on in the past.

EPA requested Tributyltin (TBT) be sought as well, and compared to the NOEC calculated based on the Baseline ERA. Navy agreed.

EPA requested samples below the proposed 3 feet of depth. Navy agreed.

MIS Sampling was discussed and tabled until the next meeting.

There was general agreement that locations which did not have PRG exceedances would not need to be sampled again.

Navy agreed to deliver a proposed sampling plan for discussion on November 5, 2010. The EPA and RIDEM would provide comments for discussion on November 15, 2010. The parties would have a conference call on November 22 to discuss in detail.

Full meeting minutes were emailed to participating parties on November 9, 2010 and are provided in Appendix C.

Action Items:

Navy to provide revised plan 11/5/10

EPA and RIDEM to provide comments prior to 11/15/11

Consensus Decisions:

Seek TBT with the COCs

Collect samples below 3 foot depth limit.

SAP Worksheet #9b – DQO Participants Sheet (Conceptual Sediment Sampling Plan Review)

(UFP-QAPP Manual Section 2.5.1)

Project Name:	**Data Gaps Investigation**	Site Name:	**Derecktor Shipyard (Off Shore)**
Projected Date(s) of Sampling:	**June 2011**	Site Location:	**NAVSTA Newport, Newport Rhode Island**
Project Manager:	**Stephen Parker**		

Date of Session: **11/22/1010**

Scoping Session Purpose: **DQO determination**

Name	Title	Affiliation	Phone #	E-Mail Address	Project Role
D. Barclift	Risk Assessor	NAVFAC LANT	215-897-4913	david.barclift@navy.mil	Advisory
K. Finkelstein	Biologist	NOAA	617-918-1499	ken.finkelstein@noaa.gov	Trustee
T. Finlayson	PM	Gannett Fleming	718-380-7750	rtfinlayson@gfnet.com	Support to USEPA
A. Gavaskar	Sediment Transport Scientist	NAVFAC LANT	757-322-4730	arun.gavaskar@navy.mil	Advisory
B. Hoskins	Ecological Risk Assessor	USEPA	617-918-6375	hoskins.bart@epa.gov	Technical
G. Jablonski	RPM	RIDEM	401-222-2797	gary.jablonski@dem.ri.gov	RIDEM RPM
W. Johnson	RPM	NAVFAC Mid-Atlantic	757-341-2008	winoma.johnson@navy.mil	Navy RPM
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S. Parker	PM	Tetra Tech	978-474-8434	stephen.parker@tetratech.com	PM

Comments/Decisions:

The Conceptual sampling plan that was distributed to the project team on 11/9/2010 was the topic of discussion for this conference call.

It was decided that separate Decision Units as presented in the Conceptual Sampling plan were too large and would not be appropriate to identify hot spots. After further discussion it was agreed by the group that Multi-increment sampling was not appropriate for sediment because the group felt that hot spots may

be present and would be necessary to identify. It was agreed that 200 foot and 100 foot grid cells would be adequate to refine the areas and define hot spots.

EPA requested adding Zinc to the COC list because of the levels it was detected at in Army Corps Data from 1987.

NOAA requested that all the stations should be analyzed for zinc and copper as well as the COCs because they could be generated by bottom paint sloughing by the ships present.

US Fish & Wildlife stated that the outer portions of Pier 2 were also of concern, and though the source may not be from a CERCLA release, additional data should be sought from this area and considered.

Navy stated that the site contaminant sources have been addressed.

Radioisotope analysis and Flume (cohesion) testing was agreed to.

It was agreed that the Navy would provide a revised sampling plan to the project team before December 6, 2010 and there would be another call following to discuss it.

Full meeting minutes and attachments from the meeting are provided in Appendix C.

Action Items

Navy to provide a revised plan 12/6/10.

Consensus Decisions

Use of a grid system using 100 and 200 foot grid spacing is adequate.

SAP Worksheet #9c – DQO Participants Sheet (Scoping Session Conceptual Site Model)
(UFP-QAPP Manual Section 2.5.1)

Project Name:	**Data Gaps Investigation**	Site Name:	**Derecktor Shipyard (Off Shore)**
Projected Date(s) of Sampling:	**June 2011**	Site Location:	**NAVSTA Newport, Newport Rhode Island**
Project Manager:	**Stephen Parker**		

Date of Session: **12/14/2010**

Scoping Session Purpose: **Discuss scoping issues that need to be addressed in order to begin development of the UFP-SAP.**

Name	Title	Affiliation	Phone #	E-Mail Address	Project Role
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K. Keckler	RPM	US EPA	617-918-1385	keckler.kymberlee@epa.gov	EPA RPM
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Comments/Decisions:

Tetra Tech presented a summary of the new conceptual sampling plan that was issued December 3, 2010. This plan utilizes a combination of 200 foot grid cells and 100 foot grid cells. One discrete core located at the center of each cell will characterize each cell. Larger cells will characterize most of the areas identified at the previous call (areas where PRGs were exceeded in 1996). Smaller cells will be

used to characterize areas surrounding and shoreward of the sediment stations that exceeded PRGs in the most recent round (2004).

Data from the proposed grid network will be used to contour contaminant concentrations. If it is appropriate, the discrete data points may be combined into decision units, but this will be determined after the data are collected and evaluated.

RIDEM requested that the Navy add samples at former sample location DSY -18. The Navy agreed to review RIDEM's request and determine if there was any need for sampling at this location.

Sample locations were discussed, requests were made to add additional sample locations to the end of Pier 2, the end of Pier 1, near former sample DSY29, the still-water basin, and near DSY 22.

It was agreed that the Navy collect samples for zinc and copper in areas not previously accessible to the sampling team in 2004 (under the piers and under the carriers).

It was agreed that all cores would be sampled using the depth scheme discussed on 11/22/10 – one sample 0-1 foot, one sample 1-2 foot, and one sample 2-4 foot.

One additional physical data point was added to the area near DSY 103.

Full meeting minutes and attachments from the meeting are provided in Appendix C.

Action Items

Navy to prepare Sampling and Analysis Plan to reflect the sample plan and the discussions held.

Navy to consider re-sampling DSY 22 and DSY 18.

Consensus Decisions

Include copper and zinc in areas previously not accessible (under ships, piers, and where it was found near pier 2).

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

10.1 INTRODUCTION

The conceptual site model has been previously published in the ecological risk assessment (SAIC 1997) and the draft Feasibility Study (Tetra Tech, 1999). It is revised below with the most recent site-specific information available gathered during the development of this Sampling and Analysis Plan.

10.2 SITE LOCATION, BACKGROUND, AND HISTORY

NAVSTA Newport is located approximately 60 miles southwest of Boston, Massachusetts, and 25 miles south of Providence, Rhode Island (Figure 10-1). It occupies approximately 1,063 acres, with portions of the facility located in the city of Newport and Towns of Middletown and Portsmouth, Rhode Island. The facility layout is long and narrow, following the western shoreline of Aquidneck Island for nearly 6 miles facing the east passage of Narragansett Bay (Figure 10-2).

The site was leased and used by a private enterprise (Robert E. Derecktor of Rhode Island, Inc.) to repair, maintain, and construct private and military ships. Figure 10-3 presents the major features of the site during operation. Repair and maintenance operations consisted of sandblasting and painting, hull inspections, and other onboard ship repairs. Two floating dry docks were moored at Pier 1, and a large ferry, known as the Greenport Ferry was moored south of Building A18 (Figure 10-3). All were removed from the site at the end of the Derecktor Lease. Construction included cutting and welding steel, sandblasting, priming and painting the structures, and assembling the ships. Supporting the ship maintenance and construction operations were an engineering department, an electrical and pipe shop, and a vehicle maintenance shop.

10.3 SITE PHYSICAL CHARACTERISTICS

The Robert E. Derecktor Shipyard Site is located in Coddington Cove (Figure 10-1). The on-shore portions of the site comprise approximately 41 acres of shoreline land and improvements, and the site includes the adjacent deep water industrial port in Coddington Cove (Figure 10-2). The shipyard boundary presented on Figure 10-2 is the portion of the property leased by Derecktor where chemical contaminants were found. Figure 10-3 presents the major features of the site during operation.

10.3.1 Shoreline Structures and Landforms

Physical features of the industrial port include two Piers, each extending approximately 1,500 feet west, into Coddington Cove; an L shaped stone breakwater at the former location of Building 42 that extends approximately 800 feet west into Coddington Cove; and a T-wharf extends approximately 800 feet west, into the cove, which formerly housed Building A18. Together, the breakwater and the Building A18 wharf form a protected small-boat basin (Stillwater area) south of Piers 1 and 2. A sheet pile wall defines the shoreline along the shipyard property and deep water port area. The eastern shoreline of Coddington Cove, along and north of the Derecktor Shipyard property, is approximately 3,200 feet long. Coddington Cove covers an area of approximately 400 acres. The cove is protected to the north by the Coddington Cove breakwater. To the southwest, the cove is surrounded by a combination of natural and altered shoreline, formed through natural erosion of landforms and Navy construction conducted over the period of their use of this area. The southern shore of the cove is characterized by a gravely and stony beach that has a very gradual grade to the off shore areas. Figures showing on-shore and off-shore site features are presented as Figure 10-3.

10.3.2 Marine Hydrographics, Sediment Characteristics and Bathymetry of Coddington Cove

A hydrographic survey was performed by the University of Rhode Island in 1995 to measure current velocity and water column profiling of conductivity, temperature, and depth to determine patterns of water circulation within the study area. This study evaluated the area during several different wind and tidal pattern cycles, but did not account for seasonal variation of wind patterns and effect of winter storms. The complete results are reported in the Derecktor Shipyard Marine Ecological Risk Assessment (URI & SAIC 1997).

The hydrographic surveys showed that the characteristic flow pattern occurs as a net counter-clockwise circulation within the interior of Coddington Cove. On average, maximum bottom velocities were found to be highest at the mouth of the cove and decreased in a counterclockwise manner following a general circulation pattern around the cove. High bottom velocities extending into the southeastern section of the cove were expected to prevent deposition of silt sized particles, while the interior sections of the region between the piers and the northeastern region were generally sluggish and expected to be depositional zones (except near shore and/or shallow areas that may be affected by wave energy or propeller wash).

This study did not account for the localized disturbance of sediments from ship activity at the piers and bulkheads. It was speculated that propeller wash from ships maneuvering to and from the piers may disturb sediments in and around these areas, and that some of the sediments could become resuspended during such activity, resulting in localized movement of contaminated sediment.

The results indicated that sediments in the Derecktor Shipyard/Coddington Cove study area were predominantly fine-grained at some stations (less than 40 percent sand content) and predominantly sandy (sand greater than 70 percent) at others. Surface sediments in Coddington Cove tended to be finer grained (contained more silt and clay) than underlying sandy sediments, probably due to the significantly decreased bottom energy and increased likelihood of fine-grained sediment deposition resulting from construction of the Coddington Cove breakwater in 1957.

A bathymetric survey of Coddington Cove, conducted by the National Ocean Survey (NOS) in 1957, indicates water depths ranging from less than 5 feet in the northeastern and southern areas of the cove, to between 33 to 40 feet adjacent to Piers 1 and 2, and gradually deepening to 50 feet at the cove entrance. Another bathymetric study performed by SAIC in 1986 in a portion of the cove south of Pier 1 was found to be in good agreement with the NOS survey (SAIC and URI, 1997). A hydrographic survey in the vicinity of Pier 1 was completed for the Naval Inactive Ships Maintenance Office in 2006 to obtain a better understanding of the bottom elevations at Pier 1 in the vicinity of two inactive aircraft carriers. The survey concluded that bottom elevations in the berth areas generally ranged from 34 to 40 feet below the NOAA Mean Lower Low Water Datum, findings that are consistent with the NOS survey. The survey further concluded that the maximum draft of the carriers currently berthed at Pier 1 ranged from 30 to 32 feet and under-keel clearance ranged between 4 and 11 feet (Sea Vision, 2006).

10.4 PREVIOUS ENVIRONMENTAL INVESTIGATIONS AND ACTIONS

Since there was a possibility of hazardous materials releases at the site, and because the Derecktor Corporation had filed for bankruptcy, the Navy performed a Preliminary Assessment (PA) of the site. The Final PA (May 1993) provided the following conclusions:

- Shipyard operations generated large quantities of hazardous wastes, including waste oil, paints, solvents, thinners, concentrated bases, and other waste solids and liquids.
- Housekeeping practices and hazardous material handling practices at the facility were poor.
- Waste materials, including spent sandblast grit and oily liquids from the dry docks, were known to be disposed of on the property.
- Sand blast grit and metals-contaminated marine sediments are present around Pier 1.
- Releases of hazardous material are suspected in the waterfront areas and around Building 6.
- Interiors of Buildings 42, 234, 6 and 40 required cleaning prior to re-use/demolition.
- Numerous unlabeled 55-gallon drums containing unknown liquids were present.
- Asbestos-containing materials were suspected in some buildings.

- Releases to the ground surface at the site would most likely cause contaminants to pass to the marine environment through groundwater flow or via storm drain systems.

Based on these findings, in 1994 the Navy added the site to the FFA list as a Study Area (SA-19). A Site Assessment Screening Evaluation (SASE) (B&R Environmental, 1997) was performed to characterize contaminants in the on-shore portions of the site – refer to Section 10.5.1. A Marine Ecological Risk Assessment (ERA) (URI & SAIC, 1997) was performed to characterize the risk posed by contaminants in the marine sediment to ecological receptors near the site. A Human Health Risk Assessment report (HHRA) (B&R Environmental, 1998) was prepared using data collected as a part of the ERA to identify the increased risks to humans from the contaminants in the marine environment – refer to Section 10.5.2 for a summary of the risk assessment conclusions.

Finally, a Feasibility Study (FS) was drafted to evaluate remedial alternatives for the marine portions of the site (B&R Environmental, 1999). The PRGs for the marine sediment were developed and finalized as a part of the draft FS. However, there were uncertainties in the volume of sediment exceeding the PRGs. Therefore the FS will be revised following the collection of the data described in this Sampling and Analysis Plan.

Between the publication of the PA report and the FS, other construction and site restoration activities occurred on shore.

- Building 234 was removed between 1993 and 1997.
- The dry docks were removed from Pier 1.
- The Greenport Ferry was removed in 1993.
- Building A-18 on the T-Wharf south of Pier 1 was demolished in 1997;
- NAVSTA Public Works Department (PWD) performed a removal at the site in 1994 that consisted of removing remaining debris, surface cleaning grossly contaminated concrete, and closing and removing storage tanks.
- In 1996, NAVSTA PWD contracted a removal of sand blast grit that was present on the ground to the north and east of Building 42. OHM Corporation removed approximately 16,600 cubic yards of this material and covered the ground with a crushed stone/gravel mix.
- In 1997, the soil berm located to the south of the site was removed and sorted. This material was found to contain excavated soil and debris from construction and expansion of Building 234.
- In 1999, a removal action was taken to address soil under Building 42 containing paint constituents

- In 1999 a removal action was taken to address PCB-containing soils to the northeast of Building 6.

The inactive ships USS Saratoga and USS Forrestal were moved to the site (Pier 1) between 1999 and 2000. The Forrestal was removed from the south side of Pier 1 in 2010. The Saratoga is expected to be removed from the north side of Pier 1 in 2011.

10.5 SUMMARY CONCEPTUAL SITE MODEL

The conceptual site model is summarized into a simplified graphic provided as Figure 10-4. This graphic provides a brief summary of the information gathered in prior documents, as described in the subsections that follow. This information is assembled in this manner to best describe the contaminants and the risks predicted from those contaminants.

10.5.1 Contaminants and Contaminant Inputs

Sediments in the Derecktor Shipyard/Coddington Cove study area are found to be predominantly fine-grained at some stations (less than 40 percent sand content) and predominantly sandy (sand greater than 70 percent) at others. Surface sediments in southern Coddington Cove tended to be finer grained (contained more silt and clay) than underlying sandy sediments, probably due to the significantly decreased bottom energy and increased likelihood of fine-grained sediment deposition resulting from construction of the Coddington Cove breakwater in 1957.

On shore investigations were conducted in 1996 to 1998 which found little residual contamination on the land portions of the site and only trace contaminants in the groundwater at the site. On shore investigations also found drainage systems on the waterfront which likely captured most industrial surface releases and directed them toward and into the shoreline sections of Coddington Cove (B&R 1997).

Marine sediment sampling conducted between 1993 and 1995 identified PCBs, PAHs and metals, particularly lead, in surface sediments near the shoreline and piers. These were found to pose risk to ecological receptors as documented in the ERA (SAIC 1997). Lower concentrations of contaminants were found in the outer portions of Coddington Cove.

Additional sediment sampling conducted in 2004 found similar contaminants to those previously cited in the ERA in the same locations, but at much lower concentrations. At some locations, COC with concentrations greater than PRGs in 1995 had concentrations less than PRGs in 2004. It was speculated

that depositional sediments at these locations were covering the contaminants present in the previously surficial layer.

The contaminants in marine sediment, particularly in the near shore areas, are presumed to be a result of contaminant discharges from ships and from shipyard operations on shore and off shore (floating dry docks and Greenport Ferry), as stated in the Ecological Risk Assessment. Because subsurface soil and groundwater contamination at the site is limited and COCs are not present in groundwater, it is believed that the primary route of contaminant transport from the shipyard operations into the marine sediment was via overland runoff of spilled materials discharging directly to Coddington Cove through the storm drainage system, and direct release of contaminated materials into the cove from the shoreline, floating dry docks and Greenport Ferry.

Since the completion of the risk assessments and the calculation of PRGs, inactive ships have been moored at Pier 1 for a period of 10 years. Regulatory parties and trustees have stated concerns that metals (zinc and copper) may have leached from the hulls of these ships, and that the flow dynamics within the cove may have been changed by the presence of the large hulls. They have also stated concern that moving these ships via tugboat could have scoured sediments and moved them within the cove.

In late 2007, it was found that pipe hangars under Pier 1 failed and sections of steam pipes covered with asbestos containing insulation fell into the water under the pier. 90 linear feet of pipe were lost and another 600 feet of pipe were in danger of falling in. Divers found pipes with the insulation jacket attached, but insulation was not recoverable. USEPA has stated concern about the presence of asbestos in the marine sediment.

10.5.2 Human Health and Ecological Risk

Risk assessments were conducted to identify potential risks to humans and ecological receptors from exposure to the contaminants in marine sediment.

The human health risk assessment identified potential elevated risks to persons (recreational, subsistence fishermen) from ingestion of shellfish (hard clams, mussels and lobster) collected from the site but no risk from direct exposure to the contaminated marine sediment.

The marine Ecological Risk Assessment consisted of an assessment of several exposure and effects endpoints within a line-of-evidence framework. Eight lines of evidence were evaluated individually for each station (site sub-area), based on the analysis of exposure and effects data and then the eight were

evaluated in combination to categorize the overall risk for each station. The ecological risk assessment concluded that there was elevated risk at some stations, particularly those nearest the most active areas of the former shipyard.

10.5.3 Development of Cleanup Goals (PRGs)

Based on the human health and ecological risk assessments conducted, contaminants of potential concern (COPCs) were identified for marine sediment. Baseline PRGs (BPRGs) were developed for these COPCs using a hazard quotient of 1.0, and with consideration of reference concentrations measured at background sample stations. From the list of COPCs, certain constituents were named as "limiting" COCs. Limiting COCs are COCs that dominated the unacceptable risk posed to the receptors. For these limiting COCs, Recommended PRGs (RPRGs) were calculated (SAIC 1998). The development of RPRGs addressed both anthropogenic contaminants and some uncertainties of the exposure assumptions (i.e. feeding areas for avian predators).

Table 10-1 (located at the end of Section 10) presents the COCs, BPRGs and RPRGs. The RPRGs were finalized as cleanup criteria for marine sediment at Site 19. The limiting COCs, their RPRGs, and their associated risk endpoints are summarized below (see SAIC (1998) for details).

COC	RPRG	Risk Endpoint
Lead	168 mg/kg dry weight	Toxicity to aquatic organisms from exposure to suspended sediment
Benzo(a)pyrene	539 µg/kg dry weight	Adverse human health effects from ingestion of shellfish
Total HMW PAHs	13,903 µg/kg dry weight	Toxicity to aquatic organisms from exposure to bedded sediment
Total PCBs	1,060 µg/kg dry weight	Toxicity to aquatic organisms from exposure to suspended sediment

Following the development of the PRGs, a feasibility study (FS) was conducted and an FS report was drafted, however, during the review of the FS report, it became evident that the data available for the marine sediment was not adequate to support the selection of a remedy for this medium.

10.5.4 Summary

The findings of the combined investigations conclude that contaminants were discharged from the on shore and off shore shipyard operations areas to the waterfront areas of Coddington Cove and around Pier 1. The primary routes of contaminant transport from the shipyard operations into the marine sediment were likely overland runoff of spilled materials discharging to Coddington Cove through the storm drainage system, and direct release of contaminated materials into the cove from the shoreline, floating dry docks and other activities. The highest COC concentrations measured during the risk assessments (1992-1994) are centered on the waterfront and pier areas of the cove. Additional sampling conducted in 2004 identified a similar mixture of contaminants in the same areas. The concentrations of contaminants measured in 2004, however, were consistently lower at all stations sampled in both time frames, indicating a decreased risk, possibly from deposition of clean sediment.

Review of a Draft FS has revealed that the limited chemical data available from the risk assessment studies do not provide adequate spatial resolution to support the selection of a remedy for the marine sediment. In addition, several uncertainties have been raised during the review of the Draft FS: Regulatory parties and trustees have stated concerns that dockage of inactive navy ships between 1999 and 2010 may have contributed other constituents and that the flow dynamics within the cove may have been changed by the presence of the large ship hulls. . Ship traffic and inactive ship dockage may also have changed the condition of the exposed sediment. Asbestos from steam lines under Pier 1 also is a new potential concern. Overall, the data available do not address these uncertainties.

TABLE 10-1
DEVELOPMENT OF PRGs
FORMER ROBERT E. DERECKTOR SHIPYARD
NAVAL STATION NEWPORT, NEWPORT, RHODE ISLAND
FROM SAIC 1998

COC	POTENTIAL PRGS								RECOMMENDED PRGs	
	Ecological Risk Endpoint						Human Health Risk Endpoint			
	Aquatic Bedded[2]		Aquatic Resuspended[3]		Avian Predator[4]		Subsistence Fisherman[5]		ALL ENDPOINTS	
	BPRG (HQ=1)	RPRG	BPRG (HQ=1)	RPRG	BPRG (HQ=1)	RPRG	BPRG (HQ=1)	RPRG	BPRG	RPRG
Copper	NA	NA	73.74 mg/kg	None	184 mg/kg	None	None	None	73.74 mg/kg	None
Lead[1]	NA	NA	83.94 mg/kg	168 mg/kg	622,000 mg/kg	None	NA	NA	83.94 mg/kg	168 mg/kg
TBT	The TBT concentration of 228 µg/kg is a No-Observed Effects Concentration (NOEC) for ecological receptors								NA	NA
Zinc	NA	NA	NA	NA	118 mg/kg	None	NA	NA	118 mg/kg	None
Benzo(a)pyrene[1]	NA	NA	NA	NA	NA	NA	53.92 µg/kg	539 µg/kg	53.92 µg/kg	539 µg/kg
HMW PAHs[1]	6,951 µg/kg	13,903 µg/kg	NA	NA	NA	NA	NA	NA	6,951 µg/kg	13,903 µg/kg
Total PCBs[1]	1,638 µg/kg	1,638 µg/kg	530 µg/kg	1,060 µg/kg	92.82 µg/kg	None	NA	NA	92.82 µg/kg	1,060 µg/kg

See Appendix B of FS for details of PRG development and selection of recommended PRGs.

1. COCs noted are "Limiting COCs" - COCs with the maximum contribution to risk for each station and pathway.
2. Toxicity to aquatic organisms (amphipods) exposed to bedded sediment
3. Toxicity to aquatic organisms (sea urchin larvae) exposed to resuspended sediment
4. Modeled effects to avian predator species ingesting shellfish (bivalves or lobster) exclusively from site
5. Predicted health effects to subsistence fisherman ingesting shellfish (bivalves or lobster) exclusively from site

NA - Not applicable: contaminant is not a limiting COC for the endpoint noted.
None – Recommended PRG not developed for this receptor (SAIC, 1998)

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

The primary data users of this investigation will be Tetra Tech and the Navy, in consultation with the other members of the Project Team. The Project Team includes Navy, USEPA, RIDEM, NOAA and US Fish and Wildlife representatives.

11.1 PROBLEM STATEMENT

Contaminants were discharged from on shore and off shore shipyard operations to site marine sediment, and unacceptable risks are likely to affect ecological and human receptors. In order to develop and select an appropriate remedy for site contaminants under CERCLA, a better understanding is needed of surface and subsurface sediment conditions and the propensity for contaminant movement with sediment within the cove.

Additional investigation is necessary to gather sufficient data to make an informed remedial decision. Data are needed:

- to better determine the extent of limiting COCs exceeding finalized cleanup criteria (RPRGs),
- to determine if a PRG needs to be calculated for TBT,
- to provide copper and zinc sediment data to the regulatory parties for their information,
- to determine if asbestos is present at concentrations that may impact possible response actions and
- to evaluate the mechanisms of sediment deposition and scouring under normal and extreme conditions, and to determine the rate of sediment accumulation, in order to refine the conceptual site model and to properly develop remedial alternatives in the FS.

The project objective is documented in meeting notes summarized in Worksheets #9a through #9c.

11.2 INPUTS TO PROBLEM RESOLUTION

The inputs needed to resolve the problem identified in Section 11.1 include field observations and measurements and chemical data, as described below (RPRGs have already been established as described in the Marine ERA (SAIC, 1998). Field tasks to be performed to collect these data inputs are summarized in Worksheet #14.

Field Observations and Measurements

The following field observations and measurements are needed for overall support of the data gaps investigation:

- Measurement of the lateral coordinates of sediment sample stations using GPS with sub-meter accuracy and expressed in US State Plane 1983, Rhode Island, NAD 1983, Feet.
- Physical descriptions of sediment during collection of sediment cores.
- Refined sediment topography within the area beginning approximately 200 feet north of Pier 2, and extending south to the CCRF area surface discharge outfall (small peninsula to the south of the site) including the area between Piers 1 and 2 and extending from the eastern shore to the west end of the piers. Such data can be provided using a multibeam sonar system, operated by qualified ocean surveyors.

The following field tests and measurements are needed to identify the propensity for sediments to settle out of the water column or for the bedded sediment to be scoured and resuspended:

- Data for current flow and direction at the sediment surface, and wave height information during normal conditions. Such data will be provided from automatic reading ADC Profilers that will be deployed throughout the study area for a period of 15 days.
- Sediment stability data that indicate the current velocity required to scour sediment from the bedded condition. Sediment cohesion testing will be conducted by others to provide this data.

Note: See Worksheet #14 for details concerning geophysical and sediment stability data required to support this project.

Fixed Laboratory Chemical Data and Project Action Limits

The following laboratory tests and measurements are needed to identify presence of target constituents in sediment at planned sample positions at the site.

Fixed laboratory chemical data for these constituents are needed to compare concentrations with project action limits (PALs), as follows:

1. The following “limiting” COC concentrations must be compared with their RPRGs (SAIC 1998) as summarized as follows:

COC	RPRG	Risk Endpoint
Lead	68 mg/kg dry weight	Toxicity to aquatic organisms from exposure to suspended sediment
Benzo(a)pyrene	539 µg/kg dry weight	Adverse human health effects from ingestion of shellfish
Total HMW PAHs	13,903 µg/kg dry weight	Toxicity to aquatic organisms from exposure to bedded sediment
Total PCBs	1,060 µg/kg dry weight	Toxicity to aquatic organisms from exposure to suspended sediment

2. In accordance with EPA request, concentrations of the following secondary constituent of interest must be compared to the No Observed Effects Concentration (NOEC) established through the development of ecological risk assessment:
   - Tributyltin (TBT), 228 µg/kg dry weight (No Observed Effects Concentration).

3. Concentrations of the following constituents, which contributed to risk but were not selected as COCs, will be compared to their "Baseline" PRGs (SAIC 1998) for informational purposes at the request of the regulatory agencies:
   - Zinc (under former carriers and piers), 118 mg/kg dry weight (Modeled effects to avian predators eating shellfish from the site)
   - Copper (under former carriers and piers), 74 mg/kg dry weight (toxicity to aquatic organisms exposed to suspended sediment)

4. In certain target areas, data for the following other specific analyte is needed for comparison with a reference criterion at the request of the regulatory agencies:
   - Asbestos (under Pier 1), 1% (common definition of asbestos-containing material)

To support the evaluation of sediment stability conditions [grain size and total Organic Carbon (TOC)]:

- Surface sediment grain size and TOC (no action limit)
- Subsurface sediment grain size (no action limit)

To assist in the interpretation of the ADCP data, total suspended solids (TSS) data from surface water are needed: TSS data is used to "ground-truth" measurements by providing quantity of particulates in the water column.

The list of target chemical analytes and PALs is presented in Worksheet #15. The sampling methods are presented in Worksheet #18, and the analytical methods are presented in Worksheet #19.

Fixed laboratory analytical methods must be selected such that the laboratory can achieve limits of quantitation (LOQs) less than or equal to the PALs, to the extent technically feasible using conventional methods. Worksheet #15 presents the selected methods and the laboratory LOQs, limits of detection (LODs), and detection limits (DLs) for each analyte. The laboratory will measure analyte concentrations down to the laboratory DL, and will report positive detections between the LOQ and the DL qualified as estimated "J". The "J" alerts the data user to the increased uncertainty at concentrations between the DL and LOQ. Non-detected results will be qualified as "U" and reported with an associated value of the LOD. Use of J-flagged data to achieve project goals is acceptable; however, greater scrutiny will be applied to J-qualified data.

Non-detected results with associated LOD values greater than the PAL will be treated as values that are less than the PAL for the purpose of making the decisions identified in Section 11.4. The limitations on data usability due to unmet sensitivity goals will be evaluated as described in Worksheet #37 and will be discussed in the project report. The data usability assessment will consider uncertainty associated with LOQ and/or LOD values that are greater than the PAL and will evaluate whether the inability to detect or quantify an analyte at levels equal to or less than the PAL has an adverse effect on decision making.

Inputs to Determination of Sediment Accumulation Rate

Fixed laboratory radioisotope data for the activity (rate of decay) of lead-210 (Pb-210) in sediment cores are needed to determine the rate of sediment accumulation in sediment bed layers to support refinement of the conceptual site model and development of remedial alternatives. The model for determining the sediment accumulation rate is detailed in Appendix F, Section 3.0.

The activity of polonium-210 (Po-210) will be measured by alpha spectroscopy to approximate the activity of Pb-210. Po-210 is a granddaughter radionuclide of Pb-210 that, in a closed system, such as buried sediment, is assumed to reach secular equilibrium (the same activity) with Pb-210 after approximately two years. This means that Po-210 is produced and then decays at roughly the same rate at which the Pb-

210 decays. Po-210 will be measured because it provides a more accurate estimate of Pb-210 based on alpha-particle emission than direct measurement of Pb-210 (Flett, R., 2009).

To support the sedimentation rate determination, the saturated bulk density of each sediment sample collected for Po-210 analysis will be needed. This value will be calculated from the sediment sample wet and dry weights, which will be measured by the laboratory as part of the sample preparation procedures; and the sediment sample volume, which will be calculated from the core inner diameter and the length of the core section collected by the field sampler for Po-210 analysis.

After evaluation of the sedimentation rate inputs, the geologist may determine that grain size data from the radiochemical samples are needed to support the determination of the sedimentation rate. If so, these data will be obtained from the leftover, unused portions of the dried sediment samples, which will be returned to Tetra Tech by the laboratory.

Quality Control Samples, Data Validation, and Reporting

Quality control (QC) sample analyses are described in Worksheet #14, Section 14.1.

Because the sediment analytical data will be used to assess risk, the chemical data will be subjected to USEPA Region I Tier II validation. The geotechnical/physical data (including grain size and asbestos) and radioisotope (Po-210) data will be verified but not validated. Data verification and validation are described in Worksheets #34 through #36, and data usability assessment is described in Worksheet #37. Data reporting is described in Worksheet #14, and data archiving is described in Worksheet #29.

11.3 STUDY BOUNDARIES

Horizontal and vertical boundaries of the site were defined in conjunction with the CSM that was developed for the marine ERA, and with input from members of the project team during the DQO sessions that are described in Worksheets #9a through #9c.

The population of interest includes both surface and subsurface sediment containing contaminants associated with the Derecktor construction operations, at concentrations that exceed the PALs. Sample stations were selected using a grid approach by the project team to better delineate (both horizontally and vertically) the extent of contamination at areas where contamination was previously detected, and in areas where samples were not previously collected.

The horizontal boundaries of the study area are as follows, the eastern boundary is the shoreline from a point 750 feet north of Pier 2 down to the former location of Building 254, the western boundary is the far end of Pier 1, the northern boundary is 750 feet north of Pier 2, and the southern boundary is the former foundation of Building 234 (Figure 10-3).

The vertical boundary for this investigation is the surface of the sediment to 4 feet below the sediment surface, although physical data will be collected about the water column above the sediment at preselected locations discussed in Worksheet #14. The vertical sample intervals 0-1 foot, 1-2 feet and 2-4 feet were selected to characterize vertical extent of contamination and support the selection of possible remedies for deeper sediments, if they are merited by the data. The vertical sample intervals for radioisotope analysis will be determined in the field as described in Worksheet #17, Section 17.2. They are also selected based on the expected depths of scouring that could occur from ship movement. A 75% recovery rate on the core penetrations will be required to assure representativeness of the deeper intervals. Potential for compaction during collection will also be addressed.

Samples will be collected as described in Worksheets #14 and #17.

11.4 ANALYTIC APPROACH

Data from the grid network discussed during planning meetings will be used to contour contaminant concentrations. If it is appropriate, the discrete data points may be combined into decision units but this will be determined after the data are collected and evaluated.

If all newly-acquired target analyte concentrations in the site sediment are less than the associated PALs (discussed in Section 11.2), then the team will recommend no further investigation or remedial action. Otherwise, the Project Team will determine the next course of action based on the following rules (see notes following the rules for descriptions of how the determinations referenced in the rules will be made):

- If the measured concentrations of any limiting COC exceeds the RPRG in any sediment sample, then the data will be contoured and incorporated into the FS to evaluate different remedial actions. The selected remedial action is documented in the Record of Decision.

- If the measured TBT concentration is greater than the calculated NOEC value in any sediment sample, convene the project team to determine if a PRG needs to be calculated for this analyte.

- The measured copper or zinc concentrations will be reported to the regulatory parties regardless as to whether they exceed PALs, but no specific action is determined based on such exceedances.

- If the measured asbestos concentration is >1% in any sediment sample, then include asbestos as a contaminant to be evaluated in the FS; otherwise, recommend no further action with regard to asbestos.

The FS report will evaluate the data by examining spatial contamination patterns of limiting COCs for the presence of "hot spots" and concentration gradients (contaminant contouring). If "hot spots" or localized areas of relatively high concentrations are identified, the Project Team will determine appropriate actions to reduce risk to acceptable levels or discuss steps for further investigation. If no hot spots are defined, concentration gradients of limiting COCs will provide information regarding the potential location(s) of sediment where site contamination has come to reside and will be considered by the Project Team to determine whether additional characterization is necessary within or outside of the study area. It is the expectation that additional investigations will not be needed to further define extent of contamination and that the target data set will provide sufficient delineation of sediment with limiting COCs exceeding PALs (RPRGs). If the Project Team agrees that contaminants have been sufficiently delineated, the Project Team will meet to discuss the development of remedial alternatives for an FS.

11.5 PERFORMANCE CRITERIA

The Project Team will determine whether the amount and type of data collected are sufficient to fill the data gaps and complete the site FS. This will involve an evaluation of contaminant concentrations and an evaluation of uncertainty for contaminants that have PALs below the LODs to ensure that contaminants are likely to have been detected, if present. Data quality, evaluated in accordance with criteria and processes described in Worksheets #34 through 37 will be used to determine whether data quality is sufficient to meet project objectives. If any data are rejected the rejected data will be identified as data gaps and the Project Team will determine whether the presence of these data gaps are severe enough to require additional data collection.

The sample locations were selected based on the need to refine the horizontal and vertical extent of contamination in the site sediment. The sediment analytical data will be used to map the spatial boundaries of sediment containing contaminant concentrations exceeding PALs. Comparisons of measured chemical concentrations to PALs will be made. Particular scrutiny will be applied to analytical results that are less than the LOQ when PALs are below the LOQ. The data usability evaluation process is described in more detail in Worksheet #37.

11.6 SAMPLING DESIGN AND RATIONALE

The sampling design and rationale are described on Worksheet #17 along with details of the sampling and analysis plan. Worksheet #18 lists the sample locations.

SAP Worksheet #12 -- Measurement Performance Criteria Table

(UFP-QAPP Section 2.6.2)

Measurement Performance Criteria Table – Sediment Field QC Samples[1]

QC Sample	Analytical Group[2]	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria (MPC)	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Equipment Blank	HMW PAHs, PCBs, TBT, Lead, Lead/ Copper/Zinc	One per Sample Delivery Group (SDG), or every 20 samples[3]	Accuracy/Bias/ Contamination	No target analytes ≥½ LOQ (>LOQ for common laboratory contaminants), unless target analytes in field samples are >10x those in rinsate blank.	S & A
Field Duplicates	HMW PAHs, PCBs, TBT, TOC	1 per 10 samples	Precision	Relative percent difference (RPD) must be ≤50%. If samples results are <2x LOQ, professional judgment is used.	S & A
	Lead, Lead/ Copper/Zinc, Po-210	1 per 10 samples	Precision	Relative percent difference (RPD) must be ≤50%. If samples results are <5x LOQ, absolute difference must be <4x LOQ.[4]	S & A
Cooler Temperature Blanks	HMW PAHs, PCBs, TBT, TOC	One per cooler	Representativeness	Temperature must be ≤ 6 degrees Celsius (°C).	S

Notes:
1. Laboratory QC samples are addressed in Worksheet #28.
2. The specific target compounds are presented in Worksheet #15.
3. Applicable for non-disposable equipment, if any.
4. MPCs are not applicable for Po-210 since data will not be validated, but field duplicates will be collected for informational purposes. The average of the two results will be reported.

SAP Worksheet #12 -- Measurement Performance Criteria Table (Continued)

(UFP-QAPP Section 2.6.2)

Measurement Performance Criteria Table – Aqueous Field QC Samples[1]

QC Sample	Analytical Group[2]	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria (MPC)	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Field Duplicates	TSS	1 per 10 samples	Precision	Relative percent difference (RPD) must be ≤30%. If samples results are <5x LOQ, absolute difference must be <2x LOQ.	S & A
Cooler Temperature Blanks	HMW PAHs, PCBs, TBT, TSS	One per cooler	Representativeness	Temperature must be ≤ 6 °C.	S

Notes:
1. Laboratory QC samples are addressed in Worksheet #28.
2. The specific target compounds are presented in Worksheet #15.

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

(UFP-QAPP Manual Section 2.7)

Not applicable., Data collected during previous investigations were only used to aid in the selection of sample locations for this SAP. Previously collected data will not be used in the development of the FS for the DSY.

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

The following project tasks are summarized below:

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

The standard operating procedures (SOPs) and field documentation forms referred to in this worksheet are included in Appendix A and Appendix B, respectively. Notes from the planning meetings are presented in Appendix C. The laboratory analytical specifications are presented in Appendix D and the laboratory SOPs are presented in Appendix E. Detailed field sampling procedures are presented in Appendix F.

14.1 FIELD TASKS

The project field tasks are summarized in this section.

Mobilization/Demobilization – Mobilization includes procurement of field equipment and supplies; initiation of a dig-safe utility request to NAVSTA; a site walkover; mobilization of field staff, equipment, and supplies to the Site; and site set-up. The Navy RPM will be notified of Tetra Tech mobilizations a minimum of 1 week before start of the field activities.

A field team orientation meeting will be conducted prior to starting the fieldwork to familiarize the team personnel with the Site's health and safety requirements, the objectives and scope of the field activities, and chain-of-command. This meeting will be attended by the field staff, FOL, PM, project chemist, and health and safety officer.

A site walkover will be conducted prior to mobilization. The field team will identify/mark sample locations and will identify related field support areas and requirements. Survey benchmarks will be set and marked. This site walk over will be conducted in accordance with SOP GH-1.1, Site Reconnaissance.

Demobilization includes removing field equipment and supplies from the Site, cleaning and returning rented equipment, managing investigation-derived waste (IDW), performing general site cleanup, organizing and finalizing field paperwork, and entering field records/data into the Site database.

Utility Clearance – Prior to mobilizing sampling equipment, the sampling team will meet with NAVSTA staff and if necessary, available Base utility drawings will be reviewed and a DIGSAFE number will be

obtained. Sample stations will be marked out to the extent possible for off shore work. The site utility clearance will be performed in accordance with SOP HS-1.0, Utility Locating and Excavation Clearance.

Sediment Sampling - Marine sediment sampling at the Site will be conducted in accordance with SOP SA-1.2 and parts of SOP SA-2.5 (this SOP is for DPT, it is relevant because the equipment used to collect sediment cores is similar to DPT) by a subcontractor assisted and overseen by Tetra Tech. Sediment cores for chemical analysis will be advanced to a maximum depth of 4 feet utilizing a core barrel fitted with a vibratory hammer (vibracore) at the target locations to acquire the target depth. Sediment cores for physical analysis will be advanced using a core barrel fitted to a weight only (gravity core) to keep the integrity of the sediment matrix.

Details regarding sediment collection techniques and procedures are provided in Worksheet #17 and Appendix F.

Quality Control Tasks - Field QC samples will be collected as part of the investigation, including field duplicates and equipment rinsate blanks (for non-disposable equipment, if any). Worksheet #20 presents the field QC sample summary.

Also, samples will be assigned for the laboratory to perform MS/MSD analysis (for organics except TOC), MS/laboratory duplicate analysis (for metals), laboratory duplicate (for TSS), or MS/laboratory quadruplicate analysis (for TOC). Additional volumes of these samples will be provided as necessary. Frequency of assignment is specified in Worksheet #20, and minimum sample volume requirements are specified in Worksheet #19.

Calibration Procedures - Field instrument calibration requirements are presented in Worksheet #22. All field instruments will be calibrated in accordance with the manufactures instructions.

Decontamination - The decontamination procedures presented in SOP SA-7.1 will be followed. Non-disposable equipment that comes in contact with the sample medium will be decontaminated to prevent cross-contamination between sampling points. Personnel decontamination is discussed in the health and safety plan (HASP).

Investigation-Derived Waste

IDW includes decontamination fluid, grossly contaminated used personal protective equipment (PPE) and used sampling equipment, IDW will be managed in accordance with SOP SA-7.1, as follows:

- Decontamination rinse water will be collected and transferred for staging in 55-gallon drums or bulk containers. Drums will be moved to a central staging area within a storage box and stored

on pallets until removal. Following waste characterization, the drums will be transported and disposed by a licensed contractor at an off-site facility licensed to accept the waste. IDW characterization, transport, and disposal will be performed after all IDW collection has been completed and properly containerized.

- Disposable PPE and disposable sampling equipment/materials (spoons, etc.) will be disposed of as general refuse at an approved disposal location.

Minor quantities of excess sediment left over after sampling will be returned to the location where it was collected from, this material will not be considered IDW.

Field Documentation - SOP SA-6.3 will be followed for all field documentation requirements.

Sample Handling - Samples will be handled and shipped in accordance with SOP SA-6.1, and in accordance with all chain of custody procedures.

Sonar Survey - A multi beam sonar survey will be conducted within the sampling area to provide information on slight variations (mounding and scouring) in the surface of the sediment in the study area. This information may be used to assist interpretation of thickness of sediment contamination and locations where deposition is occurring.

Geophysical Data Collection – The hydrographic survey conducted by URI in 1996 will be augmented by deploying Acoustic Doppler Current (ADC) profilers to collect data during a 15 day period. Deployments at ten stations near and around piers will assist in determination of strength of fluctuation and short term currents at the sediment surface. Surface water samples will be collected as a part of this effort to be analyzed for total suspended solids (TSS). This information will be used to assist in the interpretation of ADC data. At the same ten locations sediment will be collected for grain size analysis (0-1 foot), and sediment stability analysis as described below.

Sediment Stability Testing – Sediment stability testing will be performed by ERDC Coastal and Hydraulics Laboratory, Vicksburg MS. The method to be employed will be a cohesive sediment erosion flume. These data will provide information as to the current velocity required to scour sediments from different depths within the sediment strata.

14.2 ANALYTICAL TASKS

Chemical analysis of sediment samples will be performed by subcontracted laboratories in accordance with the methods identified in Worksheet #19. Katahdin will perform the HMW PAHs (including benzo(a)pyrene), PCBs, TOC, TSS, and metals analyses. Katahdin will subcontract with Test America for

the grain size analysis and with EMSL for the asbestos analysis. ARI will perform the TBT analysis. Radioisotope analysis (Po-210) will be performed by GEL. All analyses will be performed in accordance with the requirements of the analytical specifications for laboratory services developed for this work by Tetra Tech (Appendix D).

Katahdin, Test America, EMSL, ARI will follow the laboratory-specific SOPs (Worksheets #19 and #23) developed based on the methods listed in Worksheet #19. Copies of the Laboratory SOPs for chemical and physical analyses are included in Appendix E. GEL will follow laboratory-specific SOPs (Worksheets #19 and #23) developed based on various technical references cited in the SOPs. The GEL SOPs are proprietary and are not appended but are available to government parties upon request.

All sediment analytical results will be reported by the laboratory on a dry-weight basis. Results of percent moisture will be reported in each chemical analytical data package and electronic data files. Percent moisture information will also be captured in the data gaps report.

Percent moisture will not be reported for the radioisotope (Po-210) data because adjustment for percent moisture is not used to report on a dry-weight basis. The sediment samples are dried and homogenized by the laboratory prior to collection of an appropriate aliquot for preparation and analysis. The reported results are in dry weight without adjustment for percent moisture. However, GEL will report the weight of the entire sample in the drying container before and after drying and the tare (weighing container) weight, to enable Tetra Tech to calculate sediment density in support of the determination of sedimentation rates. These data will be captured in the data gaps report.

The analytical data packages provided by Katahdin, ARI, and GEL will be in a contract laboratory program-like format and will be fully validatable and contain raw data, summary forms for all sample and laboratory method blank data, and summary forms containing all method specific quality control (results, recoveries, relative percent differences, relative standard deviations, and/or percent differences etc.). The laboratories will provide data in an electronic data deliverable (EDD) compliant with Tetra Tech EDD specifications.

14.3 DATA MANAGEMENT AND REVIEW

Data management and review tasks are described below and in other worksheets in this SAP, as noted below.

Project documentation and records

- Field sample collection and field measurement records are described in Worksheets #27 and #29.

- Laboratory data package deliverables are described in the analytical specification (Appendix D).
- Data assessment documents and records are listed in Worksheet #29.

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

Data Handling and Management - After the field investigation is completed, the field sampling log sheets will be organized by date and media and filed in the project files. The field logbooks for this project will be used only for this Site, and will also be categorized and maintained in the project files after the completion of the field program. Project personnel completing concurrent field activities may maintain multiple field logbooks. When possible, logbooks will be segregated by sampling activity. The field logbooks will be titled based on date and activity. The data handling procedures to be followed by the laboratories will meet the requirements of the technical specification. The electronic data results will be automatically downloaded into the Tetra Tech database in accordance with proprietary Tetra Tech processes.

Data Tracking and Control - The Tetra Tech PM (or designee) is responsible for the overall tracking and control of data generated for the project. Electronic laboratory data will be captured in the Tetra Tech project database which will eventually be uploaded to Naval Installation Restoration Information Solution (NIRIS) by Tetra Tech.

- **Data Tracking.** Data is tracked from its generation to its archiving in the Tetra Tech project-specific files. The Project Chemist (or designee) is responsible for tracking the samples collected and shipped to the contracted laboratory. Upon receipt of the data packages from the analytical laboratory, the Project Chemist will oversee the data validation effort, which includes verifying that the data packages are complete and that results for all samples have been delivered by the analytical laboratory. The data validation outputs will be reviewed by the Data Validation Manager for completeness and correctness.

- **Data Storage, Archiving, and Retrieval.** The data packages received from the subcontract laboratory are tracked in the data validation log book. After the data are validated, the data packages are entered into the Tetra Tech CLEAN file system and archived in secure files. The field records including field logbooks, sample logs, chain-of-custody records, and field calibration logs will be submitted by the FOL to be entered into the CLEAN file system prior to archiving in secure project files. The project files are audited for accuracy and completeness. At the completion of the Navy contract, the records will be stored by Tetra Tech.

- **Data Security.** The Tetra Tech project files are restricted to designated personnel only. Records can only be borrowed temporarily from the project file using a sign-out system. The Tetra Tech

Data Manager maintains the electronic data files. Access to the data files is restricted to qualified personnel only. File and data backup procedures are routinely performed.

Data Review

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

14.4 PROJECT REPORT

At the conclusion of the sampling program, Tetra Tech will prepare a data report which will include a summary of laboratory analysis results, a comparison to the respective PRGs, and discussion of the results. The report will describe the areas where analytes are present exceeding BPRGs and RPRGs, and the areas where concentrations of the other target analytes are present relative to the project action levels. In addition, the physical data collected (multibeam sonar map, sediment cohesion test results, current velocities measured via ADCP, wave heights measured via ADCP radioisotope measurements) and other screening results will be summarized to provide a qualitative description of the behavior of sediment and the propensity for scouring or deposition over time.

The report will be prepared in draft format (paper/electronic format) for Navy, USEPA, and RIDEM review and then finalized (paper/electronic format) based upon the agreed-upon resolution of reviewer comments. The final version of the report will be distributed to the project team members listed in Worksheet #3.

SAP Worksheet #15 -- Reference Limits and Evaluation Table

(UFP-QAPP Manual Section 2.8.1)

Matrix: Sediment
Analytical Group: HMW PAHs, analyzed by SW-846, Method 8270D (Full Scan)[1]

Analyte	CAS Number	PAL[2] (µg/kg)	PAL Reference[2]	Project LOQ Goal (µg/kg)	Katahdin Limits		
					LOQ (µg/kg)	LOD (µg/kg)	DL (µg/kg)
HMW PAHs							
Benzo(a)anthracene	56-55-3	--	--	495	330	248	86
Benzo(a)pyrene	50-32-8	539	RPRG	180	330	248	93
Benzo(b)fluoranthene	205-99-2	--	--	495	330	248	134
Benzo(g,h,i)perylene	191-24-2	--	--	495	330	248	104
Benzo(k)fluoranthene	207-08-9	--	--	495	330	248	83
Chrysene	218-01-9	--	--	495	330	248	95
Dibenzo(a,h)anthracene	53-70-3	--	--	495	330	248	128
Fluoranthene	206-44-0	--	--	495	330	248	106
Indeno(1,2,3-cd)pyrene	193-39-5	--	--	495	330	248	122
Pyrene	129-00-0	--	--	495	330	248	101
Total High Molecular Weight PAHs	Not applicable	13903	RPRG	4635	3300	2480	--

Notes:
1. The laboratory first will analyze the samples in full scan mode. If the value associated with any J-flagged sample result or U-flagged (non-detected) sample result for any HMW PAH is above the project LOQ goal, the laboratory will reanalyze the sample for that PAH in SIM mode. (See next table below.)
2. The PAL is the RPRG as determined by the PRG Development Document (SAIC, 1998).

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

Matrix: Sediment
Analytical Group: HMW PAHs, analyzed by SW-846, Method 8270D SIM[1]

Analyte	CAS Number	PAL[(2)] (µg/kg)	PAL Reference[(2)]	Project LOQ Goal (µg/kg)	Katahdin Limits		
					LOQ (µg/kg)	LOD (µg/kg)	DL (µg/kg)
HMW PAHs							
Benzo(a)anthracene	56-55-3	--	--	495	20	10	1.9
Benzo(a)pyrene	50-32-8	539	RPRG	180	20	10	3.3
Benzo(b)fluoranthene	205-99-2	--	--	495	20	10	3.1
Benzo(g,h,i)perylene	191-24-2	--	--	495	20	10	2.0
Benzo(k)fluoranthene	207-08-9	--	--	495	20	10	3.1
Chrysene	218-01-9	--	--	495	20	10	1.7
Dibenzo(a,h)anthracene	53-70-3	--	--	495	20	10	1.8
Fluoranthene	206-44-0	--	--	495	20	10	1.8
Indeno(1,2,3-cd)pyrene	193-39-5	--	--	495	20	10	1.9
Pyrene	129-00-0	--	--	495	20	10	2.1
Total High Molecular Weight PAHs	Not applicable	13903	RPRG	4635	200	100	--

Notes:

1. The laboratory first will analyze the samples in full scan mode. If the value associated with any J-flagged sample result or U-flagged (non-detected) sample result for any HMW PAH is above the project LOQ goal, the laboratory will reanalyze the sample for that PAH in SIM mode. (See previous table above.)
2. The PAL is the RPRG as determined by the PRG Development Document (SAIC, 1998).

SAP Worksheet #15 -- Reference Limits and Evaluation Table (Continued)

(UFP-QAPP Manual Section 2.8.1)

Matrix: Sediment
Analytical Group: PCBs, analyzed by SW-846, Method 8082A

Analyte	CAS Number	PAL[1] (µg/kg)	PAL Reference[1]	Project LOQ Goal (µg/kg)	Katahdin Limits		
					LOQ (µg/kg)	LOD (µg/kg)	DL (µg/kg)
PCBs							
Aroclor-1016	12674-11-2	--	--	50	17	8.5	6.0
Aroclor-1221	11104-28-2	--	--	50	17	8.5	7.9
Aroclor-1232	11141-16-5	--	--	50	17	10	9.3
Aroclor-1242	53469-21-9	--	--	50	17	8.5	5.8
Aroclor-1248	12672-29-6	--	--	50	17	8.5	6.1
Aroclor-1254	11097-69-1	--	--	50	17	8.5	4.7
Aroclor-1260	11096-82-5	--	--	50	17	8.5	6.0
Total PCBs	1336-36-3	1060	RPRG	350	17	10	--

Notes:
1. The PAL is the RPRG as determined by the PRG Development Document (SAIC, 1998).

Matrix: Sediment
Analytical Group: TOC, analyzed by Lloyd Kahn

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	LOQ Goal (mg/kg)	Katahdin Limits		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
TOC	7440-44-0	--	--	--	400	200	99

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

Matrix: Sediment
Analyte: Tributyltin (as Tin), analyzed by SW846, Method 8270D SIM

Analyte	CAS Number	PAL[1] (µg Tin/kg)	PAL Reference[1]	Project LOQ Goal (µgSn/kg)	ARI Limits		
					LOQ (µgSn/kg)	LOD (µgSn/kg)	DL (µgSn/kg)
Tributyltin (as Tin)	36643-28-4	228	NOEC	20	1.6	0.74	0.74

Notes:
1. The PAL is the NOEC as determined by the Marine ERA (SAIC, 1997).

Matrix: Sediment
Analyte: Asbestos, analyzed by ASTM Draft Sieve Method, Determination of Asbestos in Soil; Polarized Light Microscopy (PLM) Quantitative with Transmission Electron Microscopy (TEM) Qualitative

Analyte	CAS Number	PAL[1] (%)	PAL Reference[1]	Project Detection Limit Goal (%)	EMSL LOQ[2] (%)	EMSL DL[2] (%)
Asbestos	1332-21-4.	1	NESHAPs[1]	1	1	0.25

Notes:
1. The PAL is a regulatory and industry standard for determination of the presence of asbestos in soil, building materials, etc.
2. The quantitative method provides an estimate of the percentage concentration of asbestos in sediment down to the DL. The concentration is a weighted average of the asbestos found in the fine, medium, and coarse fractions. Asbestos detected below 1% will be reported as “< 1%”. If asbestos is not detected, the laboratory will report the result as “None Detected” (ND). ND results will be confirmed qualitatively by TEM.

SAP Worksheet #15 -- Reference Limits and Evaluation Table (Continued)

(UFP-QAPP Manual Section 2.8.1)

Matrix: Sediment
Analytical Group: Metals, analyzed by SW846, Method 6010C

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	LOQ Goal (mg/kg)	Katahdin Limits		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Metals							
Copper	7440-50-8	74[2]	BPRG	25	2.5	1	0.073
Lead	7439-92-1	168[1]	RPRG	56	0.5	0.4	0.10
Zinc	7440-66-6	118[2]	BPRG	39	2.5	1.2	0.06

Notes:
1. The PAL is the RPRG as determined by the PRG Development Document (SAIC, 1998).
2. The PAL is the BPRG as determined by the PRG Development Document (SAIC, 1998).

Matrix: Sediment
Analytical Group: Po-210[1], analyzed by GEL SOP GL-RAD-I-009

Analyte	CAS Number	PAL (pCi/g)	PAL Reference	LOQ Goal (pCi/g)	GEL Limits		
					LOQ (pCi/g)	LOD (pCi/g)	MDA (pCi/g)
Po-210	14255-04-0	--	--	--	--	--	1

MDA = Minimum detectable activity
pCi/g = picocuries per gram

1. The activity of Po-210 will be used as an approximation of the activity of Pb-210.

Matrix: Surface Water
Analytical Group: TSS, analyzed by SM2540D

Analyte	CAS Number	PAL (mg/L)	PAL Reference	LOQ Goal (mg/L)	Katahdin Limits		
					LOQ (mg/L)	LOD (mg/L)	DL (mg/L)
TSS	C-009	--	--	--	4	3	2.8

SAP Worksheet #16 -- Project Schedule / Timeline Table

(UFP-QAPP Manual Section 2.8.2)

Activities	Organization	Dates		Deliverable	Draft Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Site Walkover	Tetra Tech	June 2011	Same day	None	NA
Multibeam Sonar Profile	Tetra Tech	May 2011	3 weeks after initiation	Map of Sediment Surface	June 3, 2011
Sediment Sampling (coring)	Tetra Tech	June/July 2011	4-5 weeks after initiation	Draft Data Gaps Report	November 11, 2011
Sediment Cohesion Field Tests	Tetra Tech	June/July 2011	8 weeks after initiation		
ADCP Data Recording (Field data)	Tetra Tech	July, 2011	6 weeks after initiation		

SAP Worksheet #17 -- Sampling Design and Rationale

(UFP-QAPP Manual Section 3.1.1)

Section 17.1 below presents the overall sampling design and rationale for this project, and Section 17.2 provides the detailed plan for collecting samples and submitting them for analysis.

17.1 SAMPLING DESIGN AND RATIONALE

The sampling design for this project was developed to support the evaluation of remedial alternatives in a revised FS, and to support the selection of one of those alternatives for action. A sample grid approach was selected because it is beneficial for identifying extent of contamination and concentration gradients both vertically and horizontally. Gaps have been identified in previously collected sediment data because some areas were previously inaccessible (under ships and piers), while other areas simply did not have adequate sample density to properly assess the best remedy. Data collected during this DGI will be used to contour contaminant concentrations. If it is appropriate, the discrete data points may be combined into decision units, but this will be determined after the data are collected and evaluated.

Prior to sample collection, a multibeam sonar survey will be conducted within an area beginning approximately 200 feet north of Pier 2, and extending south to the CCRF area surface drainage discharge outfall (small peninsula to the south of the site) including the area between Piers 1 and 2 and extending from the eastern shore to the west ends of Piers 1 and 2. This survey will be compared to the survey conducted in 2007 and anomalies noted. Data will be used to identify potential depositional areas, scour areas, and overall changes in depth over time. If mounding or scouring more than 1 foot vertically is noted in areas not planned for sampling, some sediment core samples (locations described below) may be adjusted to target these areas with agreement from the project team.

A grid approach was utilized to choose sediment core sample locations and assign sample designations. As was mentioned in Worksheet #11, samples were planned 200 feet apart where RPRGs are not expected to be exceeded and 100 feet apart where RPRGs are somewhat likely to be exceeded. In accordance with the planning discussions, one sample will represent each 200 foot or 100 foot area. For the sake of tracking sample locations and interpretation of results, a 50-foot grid was extended across the Site. The 50-foot grid is used to orient and locate samples within each 200-foot or 100-foot area. The sample grid and selected core locations are depicted on Figure 17-1.

Target sample stations presented on Figure 17-1 are represented by symbols covering slightly less than an area of 40 feet by 40 feet. All samples will be collected as close to the center of the assigned 50-foot grid cell as possible, but may have to be relocated within that cell to either avoid an obstruction or focus on a specific target area such as the under-deck portions of the piers. As of the date of this SAP publication, the EX-Saratoga is present on the north Side of Pier 1. If this vessel is still present during sample collection, the planned stations will be moved north to the closest position available on the north side of the ship's hull (see Figure 17-1). Based on site history and known configuration, chemical conditions in this alternate area are anticipated to be similar to the targeted sampling area. Actual sample stations where required sample depths and recoveries are achieved will be recorded with a DGPS that is accurate to within one meter.

There is limited access under the piers between the pilings. During site inspections, a small boat can be maneuvered between the pilings although there is limited overhead space available. Therefore, work under the piers may be slow, and work may only be able to be conducted during low tide. Coring will be limited to smaller equipment such as a hand held drill hammer, push cores and threaded rods. Other approaches will be entertained to capture the target sample depth intervals described in this SAP.

All sediment samples collected for chemical analysis will be analyzed for lead, benzo(a)pyrene, HMW PAHs, total PCBs and TBT. TOC analysis will be conducted for the surface (0-1 ft) interval of every core. Only selected sample locations will require asbestos, copper and zinc, and/or grain size analysis. The locations for each analysis are presented on Figure 17-1. Worksheet #18 presents the analyses required for all samples.

Sediment will be collected using a "vibracore" device which is a vibratory drill hammer fitted with core tubes and mounted on a floating barge. For chemical analyses, a sediment core will be collected from each targeted sample station. From this core three samples will be collected; one sample from the 0-1 foot interval, one sample from the 1-2 foot interval and one sample from the 2-4 foot interval. For radioisotope analysis, one sediment core will be collected from each targeted geophysical (GP01 through GP10) sample station. From this core, samples will be collected from two-inch intervals as determined by the Project Geologist. Additional cores may be required for adequate depth and volume. Detailed field sampling procedures are presented in Appendix F. Sample stations will be assigned identifiers according to the grid coordinates. If recovery is poor, repeated attempts will be made to acquire adequate depth (thickness) of sediment so that samples will represent these target intervals (Appendix F).

The hydrographic survey conducted by URI in 1996 will be augmented by deploying Acoustic Doppler Current Profilers and allowing them to collect data for a 15 day period. Deployments at the ten geophysical stations near and around the piers will assist in determination of strength of fluctuating and short term currents at

the sediment surface. Stations were selected based on physical features that may create transitions at the site and coverage of different areas of interest and are in the following areas:

- North side of Pier 2
- South side of Pier 2
- Inshore area between Piers 1 and 2
- North side of Pier 1
- South side of Pier 1
- DSY104 area
- DSY 28 area, near outfall, between Pier 1 and small stone breakwater
- South of Stillwater Area
- Between Stations DSY22 and DSY30
- Outer cove

Surface water will be collected in conjunction with the ADCPs. These samples will be collected with a Kemmerer water sampler. Samples will be analyzed for TSS and this data will be used to assist in the evaluation of the ADCP data. Samples will be collected approximately 2-3 ft above the ADCP during deployment, during the middle of the recording cycle (day 6, 7, or 8), and lastly at the end of the recording cycle.

Additional cores will be collected for sediment stability testing at the same ten locations. The method to be employed will utilize a “flume” type device ex situ. This physical test will provide information as to the current velocity required to strip sediments from their bedded condition and move them into the water column from different depths within the sediment strata. Other cores will be collected at these stations for grain size and radioisotope analysis.

17.2 SAMPLING AND ANALYSIS PLAN

The following summarizes the details of the sediment collection process for chemical analysis. The field tasks are described in more detail in Worksheet #14.

A single sediment sampling event is anticipated to be performed in September of 2011. The field team will navigate to each sample location using a boat mounted GPS unit pre-loaded with coordinates for each of the core locations. The GPS will also be able to display the sample grid to allow for navigation within the target grid cell. Sediment cores will be advanced using a barge-mounted vibracore device. A continuous core will be collected to a target depth of 4 feet below the sediment surface at each core location. The cores will be brought up to the barge, and opened for retrieval of the sediment. Sediment from each target depth interval will be placed into separate pre-cleaned stainless steel bowls, homogenized and transferred to sample containers for shipment to the analytical laboratories.

Multiple cores will be taken if necessary to achieve a recovery of 75% of the penetration of the core tube. This recovery rate is adequate to minimize impacts of compaction of the sediment during coring. One sediment core will be collected from each of the target locations. Up to four attempts within the associated grid cell will be made to achieve the 75% recovery target. From each core, samples will be collected to represent the 0-1 foot interval, the 1-2 foot interval and the 2-4 foot interval at that location. If the targeted recovery is not achieved in the four attempts, individual cores will be taken for each target interval. Details regarding this process are presented in Appendix F. Lower recovery rates can impact data interpretation because uncertainty in the sample depth intervals increases as recovery decreases.

It is anticipated that 119 sediment cores will be collected for chemical analysis, one at each location. It is anticipated that multiple cores will be collected at 10 stations each for geophysical evaluation. The planned sediment sampling locations and geophysical data collection locations are presented on Figure 17-1, and in Worksheet #18. Every sample that is collected for chemical analysis will be submitted to the analytical laboratory for analysis of HMW PAHs, PCBs, TBT and lead; depending on location, select samples will be additionally analyzed for TOC, asbestos, copper and zinc. Worksheet #18 presents a summary of samples to be collected and analysis required at each location.

Cores that are collected for geophysical evaluation will undergo radioisotope analysis (one core each of 10 stations), cohesion (flume) testing (one core each station) and grain size analysis (one core each station). Up to nine sections of the core will be analyzed separately from each core collected for radioisotope analysis. Cores that are collected for geophysical evaluation will be collected to a target minimum depth of 1 foot, but actual depth will be determined in the field by the Project Geologist, based on penetration, recovery, bedding layers present, and other location-specific sediment characteristics.

After analysis by GEL for Po-210, core section samples will be returned to Tetra Tech for possible additional analysis of grain size. Such analysis is not addressed in this SAP, and will be determined as a data need after initial evaluation of the radioisotope data by the project team.

Every sample station will be labeled with the coordinates of the grid as shown on Figure 17-1, and the depth interval, stated in inches: The sample collected at grid C9, taken from 12 to 24 inches below the surface will be labeled as DSY-SD-C09-1224. Using this approach, if samples need to be moved for any reason whatsoever, the grid coordinates can be used to provide the location.

Further details on sample acquisition, handling, management and shipping are provided in Appendix F.

Field duplicate samples will be collected, and rinsate blanks will be collected if any non-disposable equipment is used. The number of sediment and surface water samples and field QC samples is summarized in Worksheet #20.

The sampling methods are listed in Worksheet #18 and described in Worksheet #14.

The specific target analytes are presented in Worksheet #15. The analytical methods are listed in Worksheet #19.

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

(UFP-QAPP Manual Section 3.1.1)

Sampling Location/ ID Number[1]	Matrix	Depth (ft bgs)	Analytical Group	Number of Samples[3]	Sampling SOP Reference[4]
DSY-SD-C09	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2
DSY-SD-C13	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-C17	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-C21	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-C25	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-C29	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-G01	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2
DSY-SD-G05	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2
DSY-SD-G09	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2
DSY-SD-G13	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2
DSY-SD-G17	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2
DSY-SD-G21	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2
DSY-SD-G25	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2
DSY-SD-G29	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2
DSY-SD-K05	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2
DSY-SD-K09	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-K13	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-K17	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-K21	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-J24	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2

Sampling Location/ ID Number[1]	Matrix	Depth (ft bgs)	Analytical Group	Number of Samples[3]	Sampling SOP Reference[4]
DSY-SD-J26	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-J28	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-J30	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-L24	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-L26	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-L28	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-L30	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-N24	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-N26	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-N28	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-N30	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-O09	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-O13	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-O17	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-O21	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-Q25	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-Q29	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-T25	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-T29	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-S09	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-S13	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-S17	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-S21	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2

Sampling Location/ ID Number[1]	Matrix	Depth (ft bgs)	Analytical Group	Number of Samples[3]	Sampling SOP Reference[4]
DSY-SD-X09	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2
DSY-SD-X13	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2
DSY-SD-X17	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2
DSY-SD-X21	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn,	3	SA-1.2
DSY-SD-AA01	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn, Asbestos	3	SA-1.2
DSY-SD-AA05	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn, Asbestos	3	SA-1.2
DSY-SD-AA09	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn, Asbestos	3	SA-1.2
DSY-SD-AA13	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn, Asbestos	3	SA-1.2
DSY-SD-AA17	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn, Asbestos	3	SA-1.2
DSY-SD-AA21	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn, Asbestos	3	SA-1.2
DSY-SD-AD01	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-AD05	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2
DSY-SD-AD09	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2
DSY-SD-AD13	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn,	3	SA-1.2
DSY-SD-AD17	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2
DSY-SD-AD21	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2
DSY-SD-W24	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-W26	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-W28	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-W30	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-Y24	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-Y26	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-Y28	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2

Sampling Location/ ID Number[1]	Matrix	Depth (ft bgs)	Analytical Group	Number of Samples[3]	Sampling SOP Reference[4]
DSY-SD-Y30	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-AA24	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn, Asbestos,	3	SA-1.2
DSY-SD-AA26	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn, Asbestos	3	SA-1.2
DSY-SD-AA28	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn, Asbestos,	3	SA-1.2
DSY-SD-AA30	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn, Asbestos	3	SA-1.2
DSY-SD-AC24	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2
DSY-SD-AC26	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-AC28	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-AC30	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-AE24	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-AE26	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-AE28	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2
DSY-SD-AE30	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-AG24	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-AG26	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-AG28	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-AG30	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-AI24	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-AI26	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-AI28	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-AI30	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-AK24	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-AK26	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2

Sampling Location/ ID Number[1]	Matrix	Depth (ft bgs)	Analytical Group	Number of Samples[3]	Sampling SOP Reference[4]
DSY-SD-AK28	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-AK30	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-AO30	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-AP20	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-AT16	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-AT20	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-AT24	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-AT30	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-AX20	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-AX06	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-AY30	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-BB02	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-BB06	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-BB10	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-BB26	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2
DSY-SD-BC28	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2
DSY-SD-BC30	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2
DSY-SD-BD26	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2
DSY-SD-BE28	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2
DSY-SD-BE30	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2
DSY-SD-BF06	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DYS-SD-BF22	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2
DSY-SD-BG26	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2

Sampling Location/ ID Number[1]	Matrix	Depth (ft bgs)	Analytical Group	Number of Samples[3]	Sampling SOP Reference[4]
DSY-SD-BG28	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2
DSY-SD-BI26	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2
DSY-SD-BJ22	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2
DSY-SD-18	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb	3	SA-1.2
DSY-SD-Y03	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn,	3	SA-1.2
DSY-SD-AB11	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2
DSY-SD-AB15	Sediment	0-4[2]	HMW PAHs, PCBs, TBT, Pb, Cu, Zn	3	SA-1.2
DSY-GP01	Sediment	0-1	Grain Size	1	SA-1.2
		TBD[5]	Po-210[7]	9	
DSY-GP02	Sediment	0-1	Grain Size	1	SA-1.2
		TBD[5]	Po-210[7]	9	
DSY-GP03	Sediment	0-1	Grain Size	1	SA-1.2
		TBD[5]	Po-210[7]	9	
DSY-GP04	Sediment	0-1	Grain Size	1	SA-1.2
		TBD[5]	Po-210[7]	9	
DSY-GP05	Sediment	0-1	Grain Size	1	SA-1.2
		TBD[5]	Po-210[7]	9	
DSY-GP06	Sediment	0-1	Grain Size	1	SA-1.2
		TBD[5]	Po-210[7]	9	
DSY-GP07	Sediment	0-1	Grain Size	1	SA-1.2
		TBD[5]	Po-210[7]	9	
DSY-GP08	Sediment	0-1	Grain Size	1	SA-1.2
		TBD[5]	Po-210[7]	9	

Sampling Location/ ID Number[1]	Matrix	Depth (ft bgs)	Analytical Group	Number of Samples[3]	Sampling SOP Reference[4]
DSY-GP09	Sediment	0-1	Grain Size	1	SA-1.2
		TBD[5]	Po-210[7]	9	
DSY-GP10	Sediment	0-1	Grain Size	1	SA-1.2
		TBD[5]	Po-210[7]	9	
DSY-GP01	Surface Water	TBD[6]	Total Suspended Solids	3	SA=1.2
DSY-GP02	Surface Water	TBD[6]	Total Suspended Solids	3	SA=1.2
DSY-GP03	Surface Water	TBD[6]	Total Suspended Solids	3	SA=1.2
DSY-GP04	Surface Water	TBD[6]	Total Suspended Solids	3	SA=1.2
DSY-GP05	Surface Water	TBD[6]	Total Suspended Solids	3	SA=1.2
DSY-GP06	Surface Water	TBD[6]	Total Suspended Solids	3	SA=1.2
DSY-GP07	Surface Water	TBD[6]	Total Suspended Solids	3	SA=1.2
DSY-GP08	Surface Water	TBD[6]	Total Suspended Solids	3	SA=1.2
DSY-GP09	Surface Water	TBD[6]	Total Suspended Solids	3	SA=1.2
DSY-GP10	Surface Water	TBD[6]	Total Suspended Solids	3	SA=1.2

Notes:
1. Sample nomenclature is detailed in Worksheet #27.
2. Three samples collected from each sediment core, 0-1 foot, 1-2 foot, and 2-4 foot. All samples in the 0-1 foot interval will be analyzed for TOC in addition to the parameters described.
3. Number does not include field duplicates. Locations of field duplicates will be selected during sampling based on conditions in the field. Refer to Worksheet #20 for frequency and number of field duplicates.
4. See Worksheet #21 for the complete reference. The SOP listed provides sediment sampling procedures to be followed.
5. A core will be collected beginning at the sediment-water interface and ending at a depth to be determined in the field by the project geologist Worksheet 17, Section 17.2 and Appendix F. Nine 2-inch sections of these cores will be collected at equally-spaced intervals along the length of the core.
6. Surface water will be sampled approximately 2-3 ft above the ADCP instrument. One sample will be collected at each station during deployment, middle of the recording cycle and lastly at the end of the recording cycle.
7. Analytical groups in the core sections analyzed for Po-210 may include grain size as determined by the project geologist.

SAP Worksheet #19 -- Analytical SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group[1]	Analytical and Preparation Method/SOP Reference[2]	Containers (number, size, and type)	Sample volume[3] (units)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time[4] (preparation/ analysis)
Sediment	HMW PAHs	SW-846 3540C or 3550C, 8270D, 8270D SIM/ CA-204, CA-213; CA-512 or CA-526	4-ounce (oz) wide-mouth jar	30 gram (g)	Cool to ≤ 6 °C	14 days to extraction, 40 days to analysis
	PCBs	SW-846 3540C, 3545A or 3550C, 8082/CA-329, CA-500, CA-524, CA-537		30 g	Cool to ≤ 6 °C	30 days to extraction; 40 days from extraction to analysis
	TBT	Krone[(5)]; SW-846 3546, 8270D SIM/ 3304S, 802S	4-oz wide-mouth jar with Teflon-lined screw lid	30 g	Cool to ≤ 6 °C	14 days to extraction, 40 days to analysis (1 year to extraction if frozen ≤ 18 °C)
	Lead; or Lead, Copper, and Zinc	SW-846 3050B, 6010C/CA-605, CA-608	4-oz wide-mouth jar	2 g	None[(8)]	6 months to analysis
	TOC	Lloyd Kahn/CA-741	4-oz wide-mouth jar	2 g	Cool to ≤ 6 ° C	14 days to analysis
	Grain Size[(6)]	ASTM D422-63/BR-GT-006	8-oz wide mouth jar	100 g	None	None
	Asbestos[(6)]	ASTM Draft Sieve Method, Determination of Asbestos in Soil; PLM Quantitative with TEM Qualitative/ EMSL SOP	One 250ml plastic container	6 to 8 oz	None	None
	Po-210[(6,7)]	GL-RAD-I-009, GL-RAD-D-003, GL-RAD-A-016, GL-RAD-A-021	8-oz wide-mouth plastic jar	20 g wet sample (1 g dry sample)	None[(8)]	6 months
Aqueous Field QC Blanks Associated with Sediment Samples	HMW PAHs	SW-846 3510C or 3520C,8270D, 8270D SIM/ CA-204, CA-213, CA-502	Two 1-liter (L) amber glass bottles	1000 milliliter (ml)	Cool to ≤ 6 °C	7 days to extraction 40 days to analysis
	PCBs	SW-846 3510C or 3520C, 8082A/ CA-329, CA-515	Two 1-L amber glass bottles	1000 ml	Cool to ≤ 6 °C	30 days to extraction; 40 days from extraction to analysis
	TBT	Krone[(5)]; SW-846 3510C, 8270D SIM/ 3311S, 802S	Two 500 mL amber glass bottles	150 ml	Cool to ≤ 6 °C	7 days to extraction 40 days to analysis
	Lead; or Lead, Copper, and Zinc	SW-846 3010A, 6010C/CA-604, CA-608	One 250-ml polyethylene bottle	250 ml	Nitric Acid to pH<2.	6 months to analysis
Surface Water	TSS	SM2540D/ CA-720	500 ml	250 ml	Cool to ≤ 6 °C	7 days to analysis

Notes:

1. Refer to Worksheet #15 for specific target analytes.
2. Refer to the Analytical SOP References table (Worksheet #23).
3. Minimum sample volume requirement.
4. Maximum holding time is calculated from the time the sample is collected to the time the sample is extracted, digested or analyzed.
5. (Krone, et al., 1989)
6. Geotechnical/physical and radioisotope analytical groups are presented on this worksheet and on Worksheet #20 for use by field personnel. Quality control information is available in the laboratory SOPs in Appendix E but is not presented in any of the remaining worksheets as data validation will not be performed for these analyses.
7. Po-210 analysis will include measurement of the dry weight of the entire contents of the sediment sample container.
8. There is no temperature preservation requirement for these metals, but the sediment samples will be chilled to prevent potential biological activity.

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[2]	No. of Assigned Laboratory QC Samples[3, 4]	No. of Source Blanks	No. of Equip. Blanks[5]	No. of Trip Blanks	Total No. of Samples to Laboratory[4]
Sediment	HMW PAHs	357	36	18	0	18	0	411
	PCBs	357	36	18	0	18	0	411
	TBT	357	36	18	0	18	0	411
	Lead	222	22	12	0	12	0	256
	Lead, Copper, and Zinc	135	14	7	0	7	0	156
	Grain Size[(6)]	10	0	0	0	0	0	10
	TOC	119	12	6	0	0	0	131
	Asbestos[(6)]	30	0	0	0	0	0	30
	Po-210[(6,7)]	90	10	0	0	0	0	100
Surface Water	TSS	30	4	2	0	0	0	34

Notes:
1. Each sampling depth at a given location is counted as a separate sampling location.
2. Collect 1 field duplicate per 10 field samples. Ship blind to the laboratories.
3. Assign 1 Laboratory QC sample per 20 samples for MS/MSD (organics); MS/laboratory duplicate (metals), laboratory duplicate (TSS), or MS/laboratory quadruplicate for TOC on the chain-of-custody form.
4. Number of Assigned Laboratory QC Samples is not included in Total Number of Samples to Laboratory.
5. Collect 1 rinsate blank per 20 field samples for any non-disposable sampling equipment. If all sampling equipment (acetate liners, spoons, bowls, etc.) is disposable, no rinsate blank will be collected. Rinsate blank is deionized (DI) water run through sample collection equipment after decontamination.

6. Geotechnical/physical and radioisotope analytical groups are presented on this worksheet and on Worksheet #19 for use by field personnel. Quality control information is available in the laboratory SOPs in Appendix E (except for the radioisotope SOPs, which are on file with Tetra Tech) but is not presented in any of the remaining worksheets as data validation will not be performed for these analyses.
7. The leftover, unused portions of the dried Po-210 samples will be returned to Tetra Tech. Some or all of the Po-210 samples may be analyzed for grain size if determined necessary by the geologist as described in Worksheet #17

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
HS-1.0	HS-1.0 - Utility Locating and Excavation Clearance; Revision 2, December 2003	Tetra Tech	Magnetometer	N	SOP is included in Appendix A.
GH-1.1	GH-1.1 – Site Recon	Tetra Tech	Field Logbook, GPS, Stakes, Site Map	N	SOP is included in Appendix A.
SA-1.2	SA-1.3 – Surface Water and Sediment Sampling; Revision 5, March 2008	Tetra Tech	Sampling supplies	Y	SOP is included in Appendix A.
SA-2.5	SA-2.5 – Direct Push Technology	Tetra Tech	Barge Mounted Vibracore Device	Y	SOP is included in Appendix A.
SA-6.1	SA-6.1 - Non-Radiological Sample Handling ; Revision 3, February 2004	Tetra Tech	Sample Bottleware, Packaging Material, Shipping Materials	Y	SOP is included in Appendix A.
SA-6.3	SA-6.3 - Field Documentation; Revision 3, March 2009	Tetra Tech	Field Logbook, Field Sample Forms, Boring Logs	Y	SOP is included in Appendix A.
SA-7.1	SA-7.1 - Decontamination of Field Equipment; Revision 6, January 2009	Tetra Tech	Decontamination Equipment (scrub brushes, phosphate free detergent, de-ionized water)	Y	SOP is included in Appendix A.

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

(UFP-QAPP Manual Section 3.1.2.4)

Field Equipment	Activity[1]	Frequency	Acceptance Criteria	Corrective Action	Resp. Person	SOP Reference	Comments
PID for Safety Monitoring	Visual Inspection Calibration/ Verification	Daily Beginning and end of day	Manufacturer's Guidance	Operator Correction or Replacement	SSO or FOL	Manufacturer's Guidance	None.
Acoustical Doppler Current Profiler	Verification	Beginning and end of recording interval	Manufacturer's instructions	Operator correction of data or replacement of unit	Rental Vendor	Manufacturer's Guidance	None
Multibeam sonar	Verification	Daily, beginning and end of day	Manufacturer's instructions, and field surveyors corrections	Operator Correction	Subcontracted Marine Surveyor	Manufacturer's Guidance	Depth measurement devices are subject to calibration. Horizontal control is maintained through GPS which is checked continuously through triangulation to a base station.

Notes:
1. Rental equipment and instruments will be used in the field. The rental firms will be responsible for the proper care, maintenance, and repair of these items, and for tracking and documenting equipment and instrument maintenance and repairs. The FOL will ensure that the equipment is operational and suitable for its intended use.
2. In addition laboratory instrument calibration, method blank, and method specific QC is performed. The frequency, acceptance criteria, and corrective actions are presented in the SOP. Field duplicates also will be analyzed as presented in Worksheet #12.

SAP Worksheet #23 -- Analytical SOP References Table

(UFP-QAPP Manual Section 3.2.1)

Lab SOP Number[1]	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
CA-204	Analysis of SVOAs by Capillary Column GC/MS: SW-846 Method 8270, 04/10, Revision 12.	Definitive	Soil and Water/SVOCs	GC/MS	Katahdin	N
CA-213	Analysis of Semivolatile Organic Compounds By: SW 846 Method 8270 – Modified For Selected Ion Monitoring (SIM), 04/10, Revision 8.	Definitive	Sediment and Water/PAHs	Gas Chromatography (GC)/Mass Spectroscopy (MS)	Katahdin	N
CA-329	Analysis Of PCBs As Total Aroclors By Gas Chromatography/Electron Capture Detector (GC/ECD): SW-846 Method 8082, 04/10, Revision 11.	Definitive	Sediment and Water/PCBs	GC/Electron Capture Detector (ECD)	Katahdin	N
CA-500	Preparation Of Sediment/Soil Samples By Sonication Using Method 3550 For Subsequent Pesticides/PCBs Analysis, 08/10, Revision 7.	Definitive	Sediment /Pesticide/PCBs	Not applicable (extraction)	Katahdin	N
CA-502	Preparation Of Aqueous Samples For Extractable Semivolatile Analysis, 10/09, Revision 6.	Definitive	Water/ PAHs	Not applicable (extraction)	Katahdin	N
CA-512	Preparation Of Sediment/Soil Samples By Sonication Using Method 3550 For Subsequent Extractable Semi-Volatiles Analysis, 08/10, Revision 8.	Definitive	Sediment / PAHs	Not applicable (extraction)	Katahdin	N
CA-515	Preparation of Aqueous Samples for Pesticides/PCBs Analysis, 08/10, Revision 7.	Definitive	Water/PCBs	Not applicable (extraction)	Katahdin	N
CA-524	Preparation Of Sediment/Soil Samples By Soxhlet Extraction Using Method 3540 For Pesticide/PCB Analysis, 08/10, Revision 7.	Definitive	Sediment /PCBs	Not applicable (extraction)	Katahdin	N

Lab SOP Number[1]	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
CA-526	Preparation Of Sediment/Soil Samples By Soxhlet Extraction Using Method 3540 For Subsequent Extractable Semivolatile Analysis, 08/10, Revision 7.	Definitive	Sediment / PAHs Extraction	Not applicable (extraction)	Katahdin	N
CA-537	Preparation of Sediment/Soil and Tissue Samples by Accelerated Solvent Extraction Using Method 3545 for Subsequent Extractable Pesticide and PCB Analysis, 12/10, Revision 3.	Definitive	Sediment/PCBs	Not applicable (extraction)	Katahdin	N
CA-604	Acid Digestion of Aqueous Samples by EPA Method 3010 for ICP and ICP-MS Analysis of Total or Dissolved Metals, 04/10, Revision 5.	Definitive	Water/Metals Digestion	Not applicable (digestion)	Katahdin	N
CA-605	Acid Digestion of Solid Samples by USEPA Method 3050 for Metals by ICP-AES and GFAA, 09/10, Revision 5.	Definitive	Sediment/Metals Digestion	Not applicable (digestion)	Katahdin	N
CA-608	Trace Metals Analysis By ICP-AES Using EPA Method 6010, 06/10, Revision 11.	Definitive	Sediment and Water/Metals	Inductively Coupled Plasma (ICP) - Atomic Emission Spectroscopy (AES)	Katahdin	N
CA-720	Total Suspended Solids (Non-Filterable Residue) by EPA Method 160.2 and Standard Method 2540 D, 06/10, Revision 06.	Definitive	Water/TSS	Balance	Katahdin	N
CA-741	Determination of Total Organic Carbon in Solids using the EPA Region II Lloyd Kahn Method, 06/10, Revision 4.	Definitive	Sediment/TOC	TOC Analyzer	Katahdin	N
EMSL SOP	ASTM Draft Sieve Method, Determination of Asbestos in Soil, 06/09, Revision 1.	Definitive	Sediment/Asbestos	Sieves, Stereomicroscope, PLM, TEM	EMSL	Y[2]

Lab SOP Number[1]	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
BR-GT-006	Particle Size Analysis (ASTM D 2217 and D422-63), 03/10, Revision 6.	Definitive	Sediment/Grain Size	Sieves	Test America	N
3304S	Extraction of Soil/Sediment Samples using Sonication or Microwave (MARS) (EPA Methods 3550B and 3546), 07/10, Revision 001	Definitive	Sediment/TBT	Not Applicable (Extraction)	ARI	N
3311S	Aqueous Extractions using Separatory Funnel or Continuous Liquid-Liquid Extraction (EPA Methods 3510C and 3520C), 3/10, Revision 000	Definitive	Water/TBT	Not Applicable (Extraction)	ARI	N
802S	Analysis of Butyltin Species Using GC-MS Selective Ion Monitoring (SIM)	Definitive	Sediment and Water/TBT	GC/MS	ARI	N
GL-RAD-I-009	Standard Operating Procedure for Alpha Spectroscopy System, 7/11, Revision 12	Definitive	Sediment/Po-210	Alpha Spectrometer	GEL	N
GL-RAD-D-003	Standard Operating Procedure for Data Review, Validation, and Data Package Assembly, 7/11, Revision 29	Definitive	Sediment/Po-210	Not Applicable (Calculations and QC Limits)	GEL	N
GL-RAD-A-016	Standard Operating Procedure for the Determination of Radiometric Polonium, 5/11, Revision 13	Definitive	Sediment/Po-210	Not Applicable (Preparation)	GEL	N
GL-RAD-A-021	Standard Operating Procedure Soil Sample Preparation for the Determination of Radionuclides	Definitive	Sediment/Po-210	Not Applicable (Preparation)	GEL	N

Notes:
1. The preparation and analytical SOPs except for Po-210 are included in Appendix E.
2. The procedures will be modified to dry the sediment samples prior to sieving.

SAP Worksheet #24 -- Analytical Instrument Calibration Table

(UFP-QAPP Manual Section 3.2.2)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
GC/MS (Full Scan and SIM) PAHs	Decafluorotriphenyl-phosphine (DFTPP) Tune.	Beginning of each analytical run or every 12 hours	DFTPP within method specifications for tuning criteria.	Re-tune instrument, clean MS source as needed.	Analyst, Department Manager	CA-204 and CA-213
	Initial Calibration (ICAL) - 5-7 (minimum 5 points required) calibration standards, initial calibration for all analytes.	Instrument receipt, major instrument change, when continuing calibration verification (CCV) does not meet criteria.	Project specific criteria: Average Response Factor (RF) for all PAHs must be ≥ 0.05. Percent Relative Standard Deviation (%RSD) for RFs for all PAHs must be ≤ 30% or one option below: Option 1) Linear least squares regression: correlation coefficient (r) must be ≥ 0.99 Option 2) Non-linear regression: coefficient of determination (r^2) must be ≥ 0.99 (6 points for second order).	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst, Department Manager	
	Initial Calibration Verification (ICV) (Second Source)	Once after each ICAL.	Percent recovery (%R) must be within 80-120% for all project compounds.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Analyst, Department Manager	
	Establish Retention Time (RT) Window Position	Once per ICAL for each analyte and surrogate.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	Not applicable.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
GCMS – PAHs (cont.)	Evaluation of Relative Retention Times (RRTs)	With each sample.	RRT of each target compound must be within ±0.06 RRT units.	Correct problem, then rerun ICAL.	Analyst, Department Manager	CA-213
	CCV	Daily before sample analysis and every 12 hours	Project specific criteria: Full Scan and SIM: For all PAHs RF must be ≥ 0.05 Full Scan: All PAHs and surrogates must be ≤ 20% Difference of Drift (%D). SIM: All PAHs and surrogates must be ≤ 25%D	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	Analyst, Department Manager	
GC/ECD PCBs	ICAL - A minimum 5-point calibration is required.	Instrument receipt, major instrument change, when CCV does not meet criteria.	6 point calibration of Aroclors 1016/ 1260, 1242, 1248, and 1254 – One of the options below must be met: Option 1: RSD for each analyte ≤ 20%; Option 2: linear least squares regression: r ≥ 0.995; Option 3: non-linear regression: r^2 ≥ 0.99 (6 points shall be used for second order) Mid-point calibration of Aroclors 1221 and 1232; if targets are detected, 6-point calibration is performed.	Repeat ICAL and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst, Department Manager	CA-329
	ICV	Aroclors 1016 and 1260: Once after each ICAL.	The %D must be ≦ 20%D for Aroclors 1016 and 1260. (D = Difference or Drift)	Correct problem, rerun ICV. If that fails, repeat ICAL.	Analyst, Department Manager	
	CCV	Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence.	The %D must be ≦ 20%D for all project analytes.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
TOC Combustion Analyzer	ICAL – 5 points plus a blank.	Initially, when the daily CCV does not pass, but, no longer than every 3 months.	Correlation coefficient (r) must be greater than or equal to 0.995	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Analyst, Department Manager	CA-741
	CCV	Every 10 samples and at the end of the run	%R must be within 80-120%.	If the CCV fails high, report samples that are <LOQ. Recalibrate and/or reanalyze samples back to last acceptable CCV recovery.	Analyst, Department Manager	
	LCS (Second Source)	With every batch of 20 or fewer samples	%R must be within 80-120%.	Correct problem, then reanalyze.	Analyst, Department Manager	
Balance - TSS	Balance Calibration Verification	Each day of use, prior to use.	Measured balance weights must be within the following limits: Weight(g) True Value — Acceptance Criteria (g) 0.20 — ± 0.0002 10.0000 — ± 0.0005 50.0000 — ± 0.0005 100.0000 — ± 0.0005	Investigate problem. Do not use balance until verification has passed.	Analyst, Supervisor	CA-720
ICP-AES - Metals	ICAL	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	CA-608
	ICV (Second Source)	Once after each ICAL, prior to beginning a sample run.	The %R must be within 90-110% of true value for all analytes.	Do not use results for failing elements unless the ICV > 110% and the sample results are non-detect. Investigate and correct the problem.	Analyst, Department Manager	
	Calibration Blank	Before beginning a sample sequence, after every 10 samples and at end of the analysis sequence.	No analytes detected > LOD. For negative blanks, absolute value < LOD.	Correct problem. Re-prep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
	CCV	After every 10 samples and at the end of each run sequence.	The %R must be within 90-110% of true value for all analytes.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Department Manager	
ICP-AES - Metals (continued)	Low-level Calibration Check Standard (if using one-point ICAL)	Daily after one-point ICAL.	The %R must be within 80-120% of true value for all analytes.	Do not use results for failing elements, unless low-level standard recovery.> upper limit and sample results are non-detect. Investigate and correct the problem.	Analyst, Department Manager	CA-608
	Interference Check Sample (ICS)	Beginning of analytical run	ICS-A: Absolute value of concentration for all non-spiked analytes (except verified trace impurities) must be < LOD. ICS-AB: %R must be within 80-120%.	Terminate analysis; locate and correct problem; reanalyze ICS; reanalyze all samples.	Analyst, Laboratory Department Manager	
GCMS – TBT	Tuning: Inject standard solution containing Decafluorotriphenyl-phosphine. Solution includes p,p'-DDT, pentachlorophenol and bezidine to check for injection port inertness and GC column performance.	Beginning of each analytical run or every 12 hours	DFTPP must be within specifications for tuning criteria (ARI SOP 802S Appendix 20.3) DDT degradation should be < 20%. Pentachlorophenol, and benzidine responses should not exceed a tailing factor of 2 as per Section 11.3.1.3 of Method 8270D.	Re-tune instrument, clean MS source as needed. If DDT degradation exceeds 20%, follow Section 16.1.2 of SOP 802S. Breakdown does not affect the tins and analyst may continue with analysis, but must note the exceedence. If pentachlorophenol or benzidine tailing factors exceeds a factor of 2, follow the corrective actions listed in Section 16.1.2 of SOP 802S.	Analyst, Department Manager	802S

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
	ICAL - A minimum of five calibration standards are used.	Instrument receipt, major instrument change, when continuing calibration verification (CCV) does not meet criteria.	ARI SOP 802S Section 10.3: %RSD for RFs for all butyltin species must be ≤ 20% or one option below: 1) Linear least squares regression: correlation coefficient (r) must be ≥ 0.99 2) Non-linear regression: coefficient of determination (r^2) must be ≥ 0.99 (6 points for second order).	Recalibrate and/or perform the necessary equipment maintenance, noted in SOP 802S section 16.1.2. Check the calibration standards. Reanalyze the affected extracts if required.	Analyst, Department Manager	
GCMS – TBT (continued)	Establish RT Window Position	Once per ICAL for each analyte and surrogate.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	Not applicable.	GCMS Analyst	802S
	Evaluation of RRTs	Each extract analyzed	RRT of each target compound must be within ±0.06 RRT units.	Project-specific (not written in SOP) corrective action: If outliers are a laboratory QC sample (method blank, LCS), correct problem and recalibrate. If outliers are in real samples, reanalyze affected extracts at dilution to determine if the outliers are due to system performance or column overload. If diluted extract results meet RRT requirements, submit both sets of data. If diluted extracts still show RRTs outside requirements, recalibrate.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
	CCV [(2)]	Daily before sample analysis and every 12 hours. (Midpoint of the ICAL may be used as the CCV for that 12 hour bracket , but it must meet these requirements)	Internal standard areas must be within -50 to +100% of the midpoint of the ICAL. Absolute retention times must be within 0.5 minutes (30 seconds) of the midpoint of the ICAL. The TBT and surrogate RRF (relative response factor) must be ≤ 20%D from the average RRF of the ICAL.	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If CCV fails, repeat ICAL. Reanalyze all samples since last acceptable CCV.	Analyst, Department Manager	

Notes:
1. Refer to the Analytical SOP References table (Worksheet #23).
2. No ICV will be analyzed for TBT because no second source standard is commercially available.

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	CA	Responsible Person[2]	SOP Reference
GC/MS	Check pressure and gas supply daily. Manual tune if DFTPP not in criteria, change septa as needed, change liner as needed, cut column as needed. Other maintenance specified in lab Equipment Maintenance SOP	PAHs	Ion source, injector liner, column, column flow	Prior to ICAL and/or as necessary.	Acceptable ICAL or CCV	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	CA-213
GC/ECD	Check pressure and gas supply daily. Change septa and/or liner as needed, replace or cut column as needed. Other maintenance specified in lab Equipment Maintenance SOP.	PCBs	Injector liner, septa, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable ICAL or CCV	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	CA-329
TOC Combustion Analyzer	Check level of dilution water, drain vessel water, humidifier water, autosampler rinse water and phosphoric acid vessel and fill as needed. Replace oxygen cylinder.	TOC	Tubing, sample boat, syringe, humidifier, rinse electrode, phosphoric acid vessel, oxygen pressure	Prior to ICAL and as necessary.	Acceptable ICAL or CCV	Correct the problem and repeat ICAL or CCV	Analyst, Department Manager	CA-741
Balance (TSS)	Clean and calibrate balances annually (minimum). Check balance calibration each day of use. Clean balance pan prior to each use.	Weights	Cleanliness	Prior to sample weighing	Acceptable calibration verification	Correct the problem and repeat calibration verification	Analyst, Department Manager	CA-720

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	CA	Responsible Person[2]	SOP Reference
ICP-AES	Clean torch assembly and spray chamber when discolored or when degradation in data quality is observed. Clean nebulizer, check argon, and replace peristaltic pump tubing as needed. Other maintenance specified in lab Equipment Maintenance SOP.	Metals	Torch, nebulizer chamber, pump, pump tubing.	Prior to ICAL and as necessary.	Acceptable ICAL or CCV	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	CA-608
GCMS	Check pressure and gas supply daily. DFTPP not in criteria, change septa as needed, change liner as needed, cut column as needed. Other maintenance specified in Section 16 of ARI SOP 802S.	TBT	Ion source, injector liner, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable ICAL or CCV	Correct the problem and repeat ICAL or CCV. Recalibrate or stop for service on repeat failure	GCMS Analyst, Department Manager	802S

SAP Worksheet #26a – Sample Handling System

(UFP-QAPP Manual Appendix A)

Sample Handling System – Katahdin, Test America, EMSL

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): FOL, Tetra Tech
Sample Packaging (Personnel/Organization): FOL, Tetra Tech
Coordination of Shipment (Personnel/Organization): FOL, Tetra Tech.
Type of Shipment/Carrier: Hand carrier or overnight courier service (Federal Express)
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt: Sample Custodians / Katahdin, Test America, EMSL
Sample Custody and Storage : Sample Custodians / Katahdin, Test America, EMSL
Sample Preparation : Extraction Lab, Metals Preparation Lab / Katahdin; Soil Prep Lab / EMSL; Geotechnical lab / Test America,
Sample Determinative Analysis : Gas Chromatography Lab, Gas Chromatography/Mass Spectrometry Lab, Metals Lab / Katahdin; PLM Lab / EMSL; Geotechnical lab / Test America
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): 3 months from sample digestion/extraction/preparation
Biological Sample Storage (No. of days from sample collection): N/A
SAMPLE DISPOSAL
Personnel/Organization: Sample Custodians/ Katahdin, Test America, EMSL

SAP Worksheet #26b -- Sample Handling System

(UFP-QAPP Manual Appendix A)

Sample Handling System - ARI

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): FOL, Tetra Tech
Sample Packaging (Personnel/Organization): FOL, Tetra Tech
Coordination of Shipment (Personnel/Organization): FOL, Tetra Tech
Type of Shipment/Carrier: Overnight courier service (FedEx)
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): ARI sample custodians
Sample Custody and Storage (Personnel/Organization): ARI sample custodians
Sample Preparation (Personnel/Organization): ARI extraction/preparation laboratory analysts
Sample Determinative Analysis (Personnel/Organization): ARI GC/MS laboratory analysts
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): 60 days (frozen samples)
Sample Extract/Digestate Storage (No. of days from extraction/digestion): 60 days (refrigerated extracts)
Biological Sample Storage (No. of days from sample collection): Not applicable
SAMPLE DISPOSAL
Personnel/Organization: ARI sample custodians

SAP Worksheet #26c -- Sample Handling System

(UFP-QAPP Manual Appendix A)

Sample Handling System - GEL

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): FOL, Tetra Tech
Sample Packaging (Personnel/Organization): FOL, Tetra Tech
Coordination of Shipment (Personnel/Organization): FOL, Tetra Tech
Type of Shipment/Carrier: Overnight courier service (FedEx)
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): GEL sample custodians
Sample Custody and Storage (Personnel/Organization): GEL sample custodians
Sample Preparation (Personnel/Organization): GEL preparation laboratory analysts
Sample Determinative Analysis (Personnel/Organization): GEL alpha spectroscopy laboratory analysts
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): 60 days. After 60 days (or sooner, if requested by Tetra Tech), laboratory will return leftover, unused portions of dried sediment samples to Tetra Tech, at Tetra Tech's cost.
Sample Extract/Digestate Storage (No. of days from extraction/digestion): 60 days
Biological Sample Storage (No. of days from sample collection): Not applicable
SAMPLE DISPOSAL
Personnel/Organization: GEL sample custodians

SAP Worksheet #27 – Sample Custody Requirements Table

(UFP-QAPP Manual Section 3.3.3)

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

AAA	-	AA	-	A(A)##	-	####
(Site ID)	-	(Medium)	-	(Location)	-	(Depth)

Site identifier: **DSY**

Medium identifier: **SD** for sediment

SW for surface Water

Sample location identifier: Each sample station will be assigned a unique location number based on the **grid cell address**.

Depth: This portion of the sample tracking number will represent the **depth (in inches for sediment, in feet for surface water**) of the sample collected, "#####".

For example, a sediment sample from grid cell D2 collected from the 1 to 2 foot interval and sent for laboratory analysis will be identified as: **DSY-SD-D02-1224**.

Quality Control Samples: Field QC designations will conform to the following formats:

- Field Duplicates: Blind field duplicate samples will be designated such that the location designation will be replaced with "DUP" followed by a sequential value (the nth duplicate sample collected during that sampling event) and the date (MMDDYY). The sample log sheet will note which sample location the duplicate was collected from. For example, the first field duplicate sample collected July 1, 2011 would be labeled DSY-SD-DUP01-070111.

- Rinsate Blanks: Rinsate blank sample identifiers will consist of the Site identifier, the associated sample medium for which the equipment is used, the label "-RB", a sequential value (the nth trip blank collected for that medium during that sampling event), and the date (MMDDYY), for example: DSY-SD-RB01-070111.

- Laboratory QC samples: These assigned samples (matrix spike and matrix spike duplicate samples or laboratory duplicates) have no separate sample identifier codes, but are noted on the

chain-of-custody form and sample log sheet.

Sample Collection Documentation

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

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

Field Sample Handling and Chain-of Custody Procedures

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

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

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

The samples will be shipped to the laboratory in coolers packed with ice and bubble wrap or equivalent packing material, to cushion the samples to prevent breakage and to maintain the required temperature for the samples. A container filled with water and labeled "temperature blank" will be included in each cooler. The temperature of this blank will be measured by the laboratory upon sample receipt to verify acceptable sample preservation temperature. The coolers will be taped and sealed with a signed custody seal to ensure the chain-of-custody is maintained. Samples will be shipped to the laboratory by an

overnight courier to ensure that maximum sample holding times are not exceeded. Before the commencement of field work, the FOL will coordinate with the analytical laboratories to ensure that the samples to be analyzed by a laboratory through a subcontract with Katahdin are shipped to the proper destination. The maximum allowable sample holding times are presented in Worksheet #19, along with sample containers, chemical preservatives, and temperature condition requirements to maintain sample integrity.

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

- Project name and number
- Sample matrix
- Sample collector's name
- Dates/times of sample collection
- Sample identification numbers
- Number and type of containers for each sample aliquot
- Type of preservation
- Quality control (QC) sample designation
- Analysis
- Special handling instructions
- Destination of samples
- Name, date, time, and signature of each individual releasing the shipping container

Laboratory Custody Procedures

Katahdin's sample receipt and chain-of-custody procedures are detailed in SOP SD-902 (Appendix E-1), and ARI's are detailed in SOP 1019S (Appendix E-2). GEL's procedures are detailed in SOP GL-SR-E-001 (on file at Tetra Tech). The laboratory sample custodian will inspect the integrity of the cooler custody seals and measure the temperature of the samples received using the "Temperature Blank" container included in each cooler. The samples will be checked against the chain-of-custody form for holding times, sample identification, and integrity. The samples will be logged into the laboratory management system. Custody of the samples will be maintained and recorded in the laboratory, from receipt to analysis, and this record will be included with the data package deliverables.

Katahdin's SOP 903 (Appendix E-1) and ARI's SOP 1019S (Appendix E-2) describe the laboratory procedures for disposal of the environmental samples. The waste management procedures of Test America and EMSL are described in the analytical SOPs for grain size and asbestos (Appendix E-1). GEL's waste management procedures are described in its SOP GL-LB-G-001.

SAP Worksheet #28a -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Note: Katahdin's statistically-derived QC limits referenced in the worksheets below refer to Katahdin's limits at the time of analysis. Katahdin's current limits are presented in Appendix E-1. The following types of QC samples are not applicable to aqueous field QC blanks: MS, MSD, post-digestion spike, laboratory duplicate, and ICP serial dilution.

Matrix	Sediment/Aqueous Field QC Blanks
Analytical Group	PAHs
Analytical Method/ SOP Reference	SW-846 8270D/8270D SIM / CA-204, CA-213

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per preparation batch of twenty or fewer samples of similar matrix.	No target compounds > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the PSL, whichever is greater.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.
Surrogate	Full Scan - 6 per sample:2-Fluorophenol Phenol-d6 Nitrobenzene-d5 2-Fluorobiphenyl 2,4,6-Tribromophenol Terphenyl-d14 SIM – 3 per sample: 2-Methylnaphthalene-d10 Fluorene-d10 Pyrene-d10	%R must be within Katahdin's statistically-derived QC limits.	For QC and field samples, correct problem then reprepare and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Contact Client if samples cannot be reprepared within hold time.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
LCS	One per preparation batch of twenty or fewer samples of similar matrix.	%R must be within Katahdin's statistically-derived QC limits, allowing for the number of	Correct problem, then reprepare 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 E-	Analyst, Laboratory Department Manager, and Data Validator	Accuracy / Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Sediment/Aqueous Field QC Blanks
Analytical Group	PAHs
Analytical Method/ SOP Reference	SW-846 8270D/8270D SIM / CA-204, CA-213

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
		marginal exceedances presented in DoD QSM Table G-1.	1). Contact Client if samples cannot be reprepared within hold time.			
MS/MSD	One per sample delivery group (SDG) or every 20 samples.	%R should be within Katahdin's statistically-derived QC limits. Water Precision: RPD should be ≤ 30%. Soil Precision: RPD should be ≤ 50%.	Corrective actions will not be taken for samples when recoveries are outside limits if likely due to matrix, otherwise contact client.	Analyst, Laboratory Department Manager, and Data Validator	Precision/Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
IS	Six per sample – 1,4-Dichlorobenzene-d4 Naphthalene-d8 Acenaphthene-d10 Phenanthrene-d10 Chrysene-d12 Perylene-d12	Retention times for internal standards must be ± 30 seconds and the responses within -50% to +100% of the ICAL midpoint.	Inspect mass spectrometer or gas chromatograph for malfunctions. Mandatory reanalysis of samples analyzed while system was malfunctioning.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
Results between DL and LOQ	NA	Apply "J" qualifier to results between DL and LOQ.	NA	Analyst, Laboratory Department Manager, and Data Validator	Accuracy	Same as QC Acceptance Limits.

SAP Worksheet #28b -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	Sediment/Aqueous Field QC Blanks
Analytical Group	PCBs
Analytical Method/ SOP Reference	SW846 8082A / CA-329

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparation batch of 20 or fewer samples of similar matrix.	Contaminants in the method blank must be < ½ LOQ,	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Re-prepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result. Contact Client if samples cannot be re-prepared within hold time.	Analyst, Laboratory Department Manager and Data Validator	Bias/ contamination	Same as Method/SOP QC Acceptance Limits.
Surrogates	PCBs: one per sample: Decachloro-biphenyl	%Rs must meet the laboratory statistically-derived control limits.	For QC and field samples, correct problem then re-prepare and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Contact Client if samples cannot be re-prepared within hold time.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
LCS	One per preparation batch of 20 or fewer samples of similar matrix.	%R must be within Katahdin's statistically-derived QC limits.	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Contact Client if samples cannot be re-prepared within hold time.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per sample delivery group (SDG) or every 20 samples.	%R should be within Katahdin statistically derived limits. Precision: RPD should be ≤ 30%.	Corrective actions will not be taken for samples when recoveries are outside limits if likely due to matrix, otherwise contact client.	Analyst, Laboratory Department Manager, and Data Validator	Precision/ Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Sediment/Aqueous Field QC Blanks
Analytical Group	PCBs
Analytical Method/ SOP Reference	SW846 8082A / CA-329

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Second Column Confirmation	All positive results must be confirmed.	Results between primary and second column must be RPD ≤ 40%. The higher of the two results will be reported unless matrix interference is apparent.	None. Apply qualifier if RPD >40% and discuss in the case narrative.	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as Method/SOP QC Acceptance Limits.
Results between DL and LOQ	NA	Apply "J" qualifier to results between DL and LOQ.	NA	Analyst, Laboratory Department Manager, and Data Validator	Accuracy	Same as QC Acceptance Limits.

SAP Worksheet #28c -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	Sediment
Analytical Group	TOC
Analytical Method/ SOP Reference	Lloyd Kahn/ CA-741

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per preparation batch of 20 or fewer samples.	No TOC ≥ ½ LOQ.	Investigate source of contamination. Re-analyze all affected samples.	Analyst, Supervisor, QA Manager	Accuracy/bias-Contamination	Same as Method/SOP QC Acceptance Limits.
Laboratory Quadruplicate	One per preparation batch of 20 or fewer samples.	RPD should be ≤ 30% if concentration > 5x LOQ.	Narrate any results that are outside control limits	Analyst, Supervisor, QA Manager	Precision	Same as Method/SOP QC Acceptance Limits.
Matrix Spike	One per preparation batch of 20 or fewer samples.	%R must be within 75-125%.	No corrective action will be taken for samples where recoveries are outside limits and LCS criteria are met. Narrate outages	Analyst, Supervisor, QA Manager	Accuracy/bias	Same as Method/SOP QC Acceptance Limits.
LCS	One per preparation batch of 20 or fewer samples.	%R must be within 80-120%.	Re-analyze associated samples If sample is within holding time, re-analyze affected sample batch. If the LCS recovery is high but the sample results are <LOQ, narrate. Otherwise, re-prep blank and affected sample batch.	Analyst, Supervisor, QA Manager	Accuracy/bias	Same as Method/SOP QC Acceptance Limits.

SAP Worksheet #28d -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	Sediment
Analytical Group	TSS
Analytical Method/ SOP Reference	SM2540D / CA-720

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per prep batch of 20 or fewer samples	Measured TSS <LOQ	1) Investigate source of contamination 2) Evaluate sample results. Report results <LOQ. Report results >10x blank contamination with narration. 3) Reprepare a blank and all remaining samples.	Analyst, Supervisor, QA Manager	Accuracy/bias-Contamination	Same as Method/SOP QC Acceptance Limits.
Laboratory Duplicate	One duplicate every ten samples	RPD ≤20	1) Investigate problem and reanalyze sample in duplicate 2) If RPD still >20, report original result with notation.	Analyst, Supervisor, QA Manager	Precision	Same as Method/SOP QC Acceptance Limits.
LCS	One per prep batch of 20 or fewer samples	%R must be within 90-110%	1) Investigate source of problem. 2) If the LCS recovery is high report sample results <LOQ with narration. 3) Reprepare a blank and all remaining samples.	Analyst, Supervisor, QA Manager	Accuracy/bias	Same as Method/SOP QC Acceptance Limits.

SAP Worksheet #28e – Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	Sediment/Aqueous Field QC Blanks
Analytical Group	Lead/ Lead, Copper, and Zinc
Analytical Method/ SOP Reference	SW-846 6010C / CA-608

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per digestion batch of 20 or fewer samples of similar matrix.	No target metals> ½ LOQ (> LOQ for common laboratory contaminants) and > 1/10 the amount measured in any sample or 1/10 the PSL, whichever is greater. For negative blanks, absolute value must be < LOD.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Re-prepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.
LCS	One per digestion batch of 20 or fewer samples of similar matrix (varies by lot).	Water and Soil: %R must be within 80-120%, allowing for the marginal exceedances presented in DoD QSM Table G-1.	Re-digest and reanalyze all associated samples for affected analyte.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
Matrix Spike (not applicable for rinsate blanks)	One per sample delivery group (SDG) or every 20 samples.	%R should be within 80-120%if sample < 4x spike added.	Flag results for affected analytes for all associated samples with ”N”.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	%R should be within 75-125% if sample < 4x spike added.
Post-digestion Spike (not applicable for rinsate blanks)	Project-specific frequency: When MS recovery fails or analyte concentration in all samples < 50x LOD	%R should be within 75-125%.	Run associated samples by method of standard addition or flag results.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Sediment/Aqueous Field QC Blanks
Analytical Group	Lead/ Lead, Copper, and Zinc
Analytical Method/ SOP Reference	SW-846 6010C / CA-608

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Laboratory Duplicate	One per sample delivery group (SDG) or every 20 samples.	Project-specific criteria: If values are ≥ 5x LOQ, RPD should be ≤ 20%. If values are < 5x LOQ, Absolute Difference should be ≤ LOQ.	Flag results for affected analytes for all associated samples.	Analyst, Laboratory Department Manager, and Data Validator	Precision	Sediment: If values are ≥ 5x LOQ, RPD should be ≤ 35%; if values are < 5x LOQ, Absolute Difference should be ≤ 2x LOQ.
ICP Serial Dilution	One per preparation batch of twenty or fewer samples of similar matrix.	If original sample result is at least 50x LOQ, 5-fold dilution must agree within ± 10% of the original result.	Flag results for affected analytes for all associated samples with “E”.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Results between DL and LOQ	NA	Apply “J” qualifier to results between DL and LOQ.	NA	Analyst, Laboratory Department Manager, and Data Validator	Accuracy	Same as QC Acceptance Limits.

SAP Worksheet #28f -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Note: ARI's QC recovery acceptance limits referenced in the worksheet below are the laboratory's statistically-derived limits at the time of draft SAP preparation. ARI's limits at the time of analysis will apply for reporting and data validation purposes.

Matrix	Sediment/Aqueous Field QC Blanks
Analytical Group	TBT
Analytical Method/ SOP Reference	SW-846 8270D SIM / 802S

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One blank per preparation batch of twenty or fewer samples of similar matrix.	No TBT greater than ½ the reporting limit, 1/10th of the amount measured in any sample, or 1/10th the PAL, whichever is greater.	Re-extract and reanalyze the method blank and all affected samples if sample volume available. Report both sets of data if second set extracted outside holding times. Flag all positive results with a "B".	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.
Internal Standard	Tetrapentyl Tin will be added to every sample injection. (d14-p-Terphenyl will also be used but will not be evaluated for corrective action unless samples show interference with the tetrapentyl tin)	Internal standard areas must be within -50 to +100% of the midpoint of the ICAL.	If the internal standards fail to meet their acceptance criteria, check calculations, the internal standard compound spiking solutions, and instrument operation and make appropriate corrections (ARI SOP 802S Section 16.4) If above steps do not correct problem, issue may be due to matrix effect. Reanalyze sample at dilution unless samples from the site has already been proved to have a matrix effect. Submit both sets of data.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Sediment/Aqueous Field QC Blanks
Analytical Group	TBT
Analytical Method/ SOP Reference	SW-846 8270D SIM / 802S

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Surrogate:	Tripentyl Tin, spiked into every laboratory QC sample (Method Blank, LCS), sample and matrix QC (MS, MSD) extracted. (Tripropyl Tin also in the spiking solution)	%R must be within 35-130% in soil method blank and LCS %R must be within 48-110% in water method blank and LCS %R must be within 25-140% in soil samples %R must be within 35-124% in water samples Tripropyl Tin will also be analyzed as an indicator of blow down issues, but corrective action will be waived for outliers)	For QC and field samples, correct problem then re-extract and reanalyze failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available and no obvious matrix effect has been proved for the site. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Inform Client if samples cannot be re-extracted within recommended hold time.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
LCS	One per preparation batch of twenty or fewer samples of similar matrix.	TBT %R in soil must be within 40-144% TBT %R in water must be within 60-125%	Follow corrective actions in Section 16.7 of the SOP, including reanalyzing the LCS extract reviewing calculations and bench sheets. If LCS is still out of control, re-extract and reanalyze the method blank, LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available, after consideration of all batch QC data. Submit both sets of data if re-extracted outside hold time.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy / Bias	Same as Method/SOP QC Acceptance Limits.
MS/MSD (not applicable to field	One per sample delivery group		No corrective actions are required for matrix QC, as outliers are an indicator of the effect of	Analyst, Laboratory	Precision/Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Sediment/Aqueous Field QC Blanks
Analytical Group	TBT
Analytical Method/ SOP Reference	SW-846 8270D SIM / 802S

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
QC blanks)	(SDG) or every 20 samples of similar matrix.	TBT %R in soil should be within 40-144% RPD should be within 40%	the matrix on the analysis. Outliers will be noted.	Department Manager, and Data Validator		
Results between DL and LOQ	NA	Apply "J" qualifier to results between DL and LOQ.	NA	Analyst, Laboratory Department Manager, and Data Validator	Accuracy	Same as QC Acceptance Limits.

SAP Worksheet #29 -- Project Documents and Records Table

(UFP-QAPP Manual Section 3.5.1)

Document	Where Maintained
Field Documents Field Logbook Field Sample Forms Chain-of-Custody Records Air Bills Sampling Instrument Calibration Logs Sampling Notes Photographs FTMR Forms This SAP HASP	Field documents will be maintained in the project file located in the Tetra Tech Wilmington, Massachusetts office.
Laboratory Documents and Records - in the form of analytical data package: Sample receipt/login form Sample storage records Sample preparation logs Equipment calibration logs Sample analysis run logs Reported results for standards, QC checks, and QC samples Data completeness checklists Telephone logs Extraction/clean-up records Raw data	Laboratory documents will be included in the hardcopy and electronic deliverables from the laboratory. Laboratory data deliverables will be maintained in the Tetra Tech Wilmington, Massachusetts project file and in long-term data package storage at a third-party professional document storage firm. Electronic data results will be maintained in a database on a password protected Structured Query Language (SQL) server.
Assessment Findings Field Sampling Audit Checklist (if conducted) Analytical Audit Checklist (if conducted) Data Validation Memoranda (include tabulated data summary forms)	All assessment documents will be maintained in the Tetra Tech Wilmington, Massachusetts project file.
Reports Data Report (Data Gaps Investigation)	All versions of the Project Report and support documents (e.g., Data Validation Reports) will be stored in hard copy in the Tetra Tech Wilmington, Massachusetts project file and electronically in the server library.

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[1] Turnaround Time	Laboratory / Organization (name and address, contact person and telephone number)	Backup Laboratory / Organization (name and address, contact person and telephone number)
Sediment and Water (Field QC Blanks)	PAHs	See Worksheet #18	SW-846 8270D/8270D SIM	21 days	Katahdin Analytical Services, Inc. 600 Technology Way Scarborough, Maine 04074 Contact: Ms. Kelly Perkins (207) 874-2400	Not applicable
	PCBs		SW-846 8082A			
	Lead		SW-846 6010C			
	Lead, Copper, and Zinc					
Sediment	TOC	See Worksheet #18	Lloyd Kahn			
Surface Water	TSS	See Worksheet #18	SM2540D			
Sediment	Grain Size	See Worksheet #18	ASTM D422-63		Test America[2] 30 Community Drive, Suite 11 South Burlington, VT 05403 Primary Contact: Mr. Joseph Carabillo Backup Contact: Mr. Steve Timmons (802)923-1038	Not applicable

Matrix	Analytical Group	Sample Locations/ ID Number	Analytical Method	Data Package[1] Turnaround Time	Laboratory / Organization (name and address, contact person and telephone number)	Backup Laboratory / Organization (name and address, contact person and telephone number)
	Asbestos		ASTM Draft Sieve Method, Determination of Asbestos in Soil; PLM Quantitative with TEM Qualitative		EMSL Analytical, Inc. [2] 200 Route 130 North Cinnaminson, NJ 08077 Contact: Stephen Siegel, CIH Asbestos/Silica Lab Manager Phone: 800-220-3675x2555 Direct Dial: 856-303-2555 Cell 856-296-5329 Email:ssiegel@emsl.com	Not applicable
Sediment and Water (Field QC Blanks)	TBT	See Worksheet #18	SW-846 8270D SIM		Analytical Resources, Inc. 4611 S. 134th Place, Suite 100 Tukwila, WA 98168 Primary Contact: Susan D. Dunnihoo 206-695-6207 sue@arilabs.com Backup Contact: Cheronne Oreiro 206-695-6214 cheronneo@arilabs.com	Not applicable
Sediment	Po-210	See Worksheet #18	GEL SOP GL-RAD-I-009	21 days	GEL Laboratories, LLC P.O. Box 30712 Charleston, SC 29417 Contact: Ann Skradski 843-556-8171 team.skradski@gel.com	Not applicable

1. Data package deliverables are detailed in the Analytical Technical Specifications included in Appendix D. Data packages will be provided as both hardcopy and portable document format (PDF). The laboratory will provide a Naval Installation Restoration Information Solutions (NIRIS) compatible electronic data deliverable (EDD). Data packages will be Contract Laboratory Program (CLP)-equivalent (i.e., they will contain CLP-equivalent summary forms and raw data). Data will be stored by the analytical laboratory for 7 years.
2. Katahdin will subcontract with Test America for grain size analysis and EMSL for asbestos analysis.

SAP Worksheet #31 -- Planned Project Assessments Table

(UFP-QAPP Manual Section 4.1.1)

Assessment Type	**Frequency**	**Internal or External**	**Organization Performing Assessment**	**Person(s) Responsible for Performing Assessment** (title and organizational affiliation)	**Person(s) Responsible for Responding to Assessment Findings** (title and organizational affiliation)	**Person(s) Responsible for Identifying and Implementing Corrective Actions (CA)** (title and organizational affiliation)	**Person(s) Responsible for Monitoring Effectiveness of CA** (title and organizational affiliation)
Laboratory System Audit	Every 2 years	External	DoD ELAP Accrediting Body	DoD ELAP Accrediting Body Auditor	QAM, Katahdin and ARI	QAM, Katahdin and ARI	DoD ELAP Accrediting Body Auditor

1. Katahdin, ARI, and GEL successfully completed the DoD's Environmental Laboratory Accreditation Program (ELAP) audit for all analytical methods. A copy of the DoD ELAP accreditations for both analytical laboratories are included in Appendix E.

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

(UFP-QAPP Manual Section 4.1.2)

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Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (name, title, organization)	Timeframe for Response
Laboratory System Audit	Written audit report	QAM, Katahdin QAM, ARI QAM, GEL	Not specified by DoD ELAP	Letter	DoD ELAP Accrediting Body	Specified by DoD ELAP

SAP Worksheet #33 -- QA Management Reports Table

(UFP QAPP Manual Section 4.2)

Type of Report	**Frequency** (Daily, weekly monthly, quarterly, annually, etc.)	**Projected Delivery Date(s)**	**Person(s) Responsible for Report Preparation** (title and organizational affiliation)	**Report Recipient(s)** (title and organizational affiliation)
Data Validation Report	Per sample delivery group (SDG)	Within 3 weeks of receipt of laboratory data package	Project Chemist, Tetra Tech	PM, Tetra Tech Tetra Tech project file
Project monthly progress report[1]	Monthly for duration of the project	Monthly	PM, Tetra Tech	Navy, project file
Laboratory QA report	When significant plan deviations result from unanticipated circumstances	Immediately upon resolution of problem (same day)	PM, Katahdin PM, ARI PM, GEL	Tetra Tech project file

1. The monthly progress report is an update for the Navy RPM and contract office. The report includes information such as activities completed, an updated schedule, identification of outstanding issues, and plans for the next period, and a financial narrative.

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

(UFP-QAPP Manual Section 5.2.1)

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Chain-of-Custody Forms	The Tetra Tech FOL or designee will review and sign the chain-of-custody form to verify that all samples listed are included in the shipment to the laboratory and the sample information is accurate. The forms will be signed by the sampler and a copy will be retained for the project file, the Tetra Tech PM, and the Tetra Tech Data Validators.	Internal	Sampler and FOL, Tetra Tech
	The Laboratory Sample Custodian will review the sample shipment for completeness, integrity, and sign accepting the shipment. The Tetra Tech Data Validators will check that the chain-of-custody form was signed and dated by the Tetra Tech FOL or designee relinquishing the samples and also by the Laboratory Sample Custodian receiving the samples for analyses.	Internal/ External	1 - Laboratory Sample Custodian, Katahdin, ARI, and GEL 2 - Data Validators, Tetra Tech
SAP Sample Tables/ Chain-of-Custody Forms	Verify that all proposed samples listed in the SAP tables have been collected.	Internal	FOL or designee, Tetra Tech
Sample Log Sheets	Verify that information recorded in the log sheets is accurate and complete.	Internal	FOL or designee, Tetra Tech
Sample coordinates	Verify that actual sample locations are correct and in accordance with the SAP proposed locations. Document any discrepancies in the final report.	Internal	Tetra Tech, FOL or designee
SAP/ Field Logs/ Analytical Data Packages	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 sampling plan was implemented and carried out as written and that any deviations are documented.	Internal	PM or designee, Tetra Tech
SAP/ Laboratory SOPs/ Raw Data/ Applicable Control Limits Tables	Ensure that all laboratory SOPs were followed. Verify that the correct analytical methods/SOPs were applied. 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 QAM will contact the Tetra Tech PM via telephone or e-mail for guidance prior to report preparation.	Internal	QAM, Katahdin, ARI, and GEL
SAP/ Chain-of-Custody Forms	Check that field QC samples listed in Worksheet #20 were collected as required.	Internal	FOL or designee, Tetra Tech

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Analytical Data Packages	All analytical data packages will be verified internally for completeness by the laboratory performing the work. The Laboratory QAM will sign the case narrative for each data package.	Internal	QAM, Katahdin, ARI, and GEL
Electronic Data Deliverables (EDDs)/ Analytical Data Packages	Each EDD produced by each lab will be verified against the chain-of-custody and hard copy data package 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 DL and the LOQ will be qualified as estimated. Extraneous laboratory qualifiers will be removed from the validation qualifier.	External	Data Validators, Tetra Tech (external to the laboratory)
	Each data package will be verified for completeness by the Tetra Tech Data Validator. Missing information will be requested by the Tetra Tech Data Validator from the Laboratory PM.	External	Data Validators, Tetra Tech

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

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

Step IIa / IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIa	Chain-of-Custody Forms	Custody - Ensure that the custody and integrity of the samples was maintained from collection to analysis and the custody records are complete and any deviations are recorded. Review that the samples were shipped and store at the required temperature and sample pH for chemically-preserved samples meet the requirements listed in Worksheet #19. Ensure that the analyses were performed within the holding times listed in Worksheet #19.	Project Chemist or Data Validators, Tetra Tech
IIa/IIb	SAP/ Laboratory Data Packages/ EDDs	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.	Project Chemist or Data Validators, Tetra Tech
		Check the field sampling precision by calculating the RPD for field duplicate samples. Check the laboratory precision by reviewing the RPD or percent difference values from laboratory duplicate analyses; MS/MSDs; and LCS/laboratory control sample duplicate (LCSD), if available. Ensure compliance with the methods and project MPCs accuracy goals listed in Worksheet #12.	
		Check that the laboratory recorded the temperature at sample receipt and the pH of the chemically preserved samples to ensure sample integrity from sample collection to analysis.	
		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. The Tetra Tech Data Validator will verify that elements of the data package required for validation are present, and if not, the laboratory will be contacted and the missing information will be requested. Validation will be performed as per Worksheet #36. Check that all data have been transferred correctly and completely to the final Structured Query Language (SQL) database.	

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

Step IIa / IIb	Validation Input	Description	Responsible for Validation (name, organization)
		QA/QC - Ensure that all QC samples specified in the SAP were collected and analyzed and that the associated results were within prescribed SAP acceptance limits. Ensure that QC samples and standards prescribed in analytical SOPs were analyzed and within the prescribed control limits. If any significant QC deviations occur, the Laboratory QAM shall have contacted the Tetra Tech PM.	
		Deviations - Summarize deviations from methods, procedures, or contracts in the Data Validation Report. Determine the impact of any deviation from sampling or analytical methods and SOPs requirements and matrix interferences effect on the analytical results. Qualify data results based on method or QC deviation and explain all the data qualifications. Print a copy of the project database qualified data depicting data qualifiers and data qualifiers codes that summarize the reason for data qualifications. Determine if the data met the MPCs and determine the impact of any deviations on the technical usability of the data.	

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	Sediment and Water (Rinsate Blanks)	PAHs and TBT	Tier II[1] data validation. Project-specific criteria for PAHs by SW-846 8270D and 8270D SIM and for TBT by SW-846 8270D SIM are listed in Worksheets #12, #15, #19, #24, and #28. Region I EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, December 1996 (EPA, 1996) will be applied using these criteria.	Tetra Tech, Project Chemist (L. Guzman) and staff chemists
		PCBs	Tier II[1] data validation. Criteria for PCBs by SW-846 8082A are listed in Worksheets #12, #15, #19, #24, and #28. Region I EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, February 2004 (EPA, 2004) will be applied using these criteria.	
		Lead; and Lead, Copper, and Zinc	Tier II[1] data validation. Project-specific criteria for metals by SW-846 6010C are listed in Worksheets #12, #15, #19, #24, and #28. Region I EPA-NE Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses, Part IV, November 2008 (EPA, 2008) will be applied using these criteria.	
	Sediment	TOC	Tier II[1] data validation. Project-specific criteria for TOC by Lloyd Kahn are listed in Worksheets #12, #15, #19, #24, and #28. Region I EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, December 1996 (EPA, 1996) will be applied using these criteria.	
	Surface Water	TSS	Tier II[1] data validation. Project-specific criteria for TSS by SM2540D are listed in Worksheets #12, #15, #19, #24, and #28. Region I EPA-NE Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses, Part IV, November 2008 (EPA, 2008) will be applied using these criteria.	

1. As defined in the Region 1 EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part I, Attachment B, "Region 1 Tiered Organic and Inorganic Data Validation Guidelines", July 1, 1993, Draft (EPA, 1996).

SAP Worksheet #37 -- Usability Assessment

(UFP-QAPP Manual Section 5.2.3)

Data Usability Assessment
The usability of the data directly affects whether project objectives can be achieved. The following characteristics will be evaluated at a minimum. The results of these evaluations will be included in the project report. The characteristics will be evaluated for multiple concentration levels if the evaluator determines that this is necessary. To the extent required by the type of data being reviewed, the assessors will consult with other technically competent individuals to render sound technical assessments of these data characteristics: **Completeness** o The FOL acting on behalf of the Project Team will determine whether deviations from the scheduled sample collection or analyses occurred. If they have occurred and the Tetra Tech PM determines that the deviations compromise the ability to meet project objectives she will consult with the Navy RPM and other project team members, as necessary (determined by the Navy RPM), to develop appropriate corrective actions. **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 Worksheets #12 and #28. This will also include a comparison of field and laboratory precision with the expectation that laboratory duplicate results will be no less precise than field duplicate results. If the goals are not met, or data have been flagged as estimated (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 LCS, LCSD, MS, MSD, and surrogate compounds to accuracy goals identified in Worksheet #28. This assessment will include an evaluation of field and laboratory contamination; instrument calibration variability; and analyte recoveries for surrogates, matrix spike, matrix spike duplicate, and laboratory control samples. If the goals are not met, limitations on the use of the data will be described in the project report. Bias of the qualified results and a description of the impact of identified non-compliances on a specific data package or on the overall project data will be described in the project report. **Representativeness** o A project scientist identified by the Tetra Tech PM and acting on behalf of the Project Team will determine whether the data are adequately representative of intended populations, both spatially and temporally. This will be accomplished by verifying that samples were collected and analyzed in accordance with this SAP, by reviewing spatial and temporal data variations, and by comparing these characteristics to expectations. The usability report will describe the representativeness of the data for each matrix and analytical fraction. This will not require quantitative comparisons unless professional judgment of the project scientist indicates that a quantitative analysis is required. **Comparability** o The Project Chemist acting on behalf of the Project Team will determine whether the data generated under this project are sufficiently comparable to historical property data generated by different methods and for samples collected using different procedures and under different property conditions. This will be accomplished by comparing overall precision and bias among data sets for each matrix and analytical fraction. This will not require quantitative comparisons unless the Project Chemist indicates that such quantitative analysis is required.

Sensitivity

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

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

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

For statistical evaluations, non-detected values will be represented by a concentration equal to one-half the LOD.

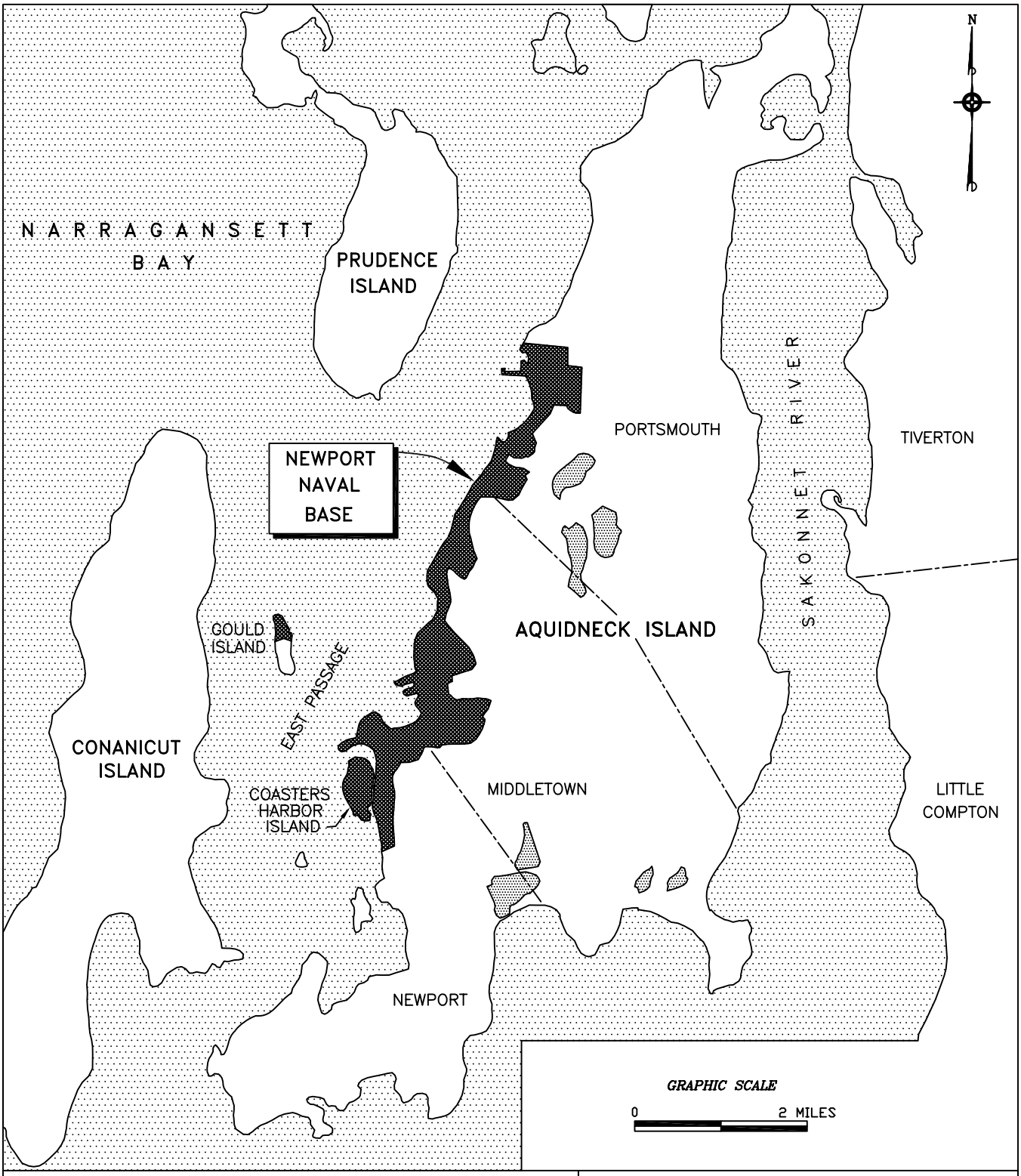
Identify the personnel responsible for performing the usability assessment:

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

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

The data will be presented in tabular format, including data qualifications such as estimation (J, UJ) or rejection (R). The project report will identify and describe the data usability limitations and suggest re-sampling or other corrective actions, if necessary. Graphical presentations of the data such as concentration tag maps will be generated as part of the overall data evaluation process.

FIGURES

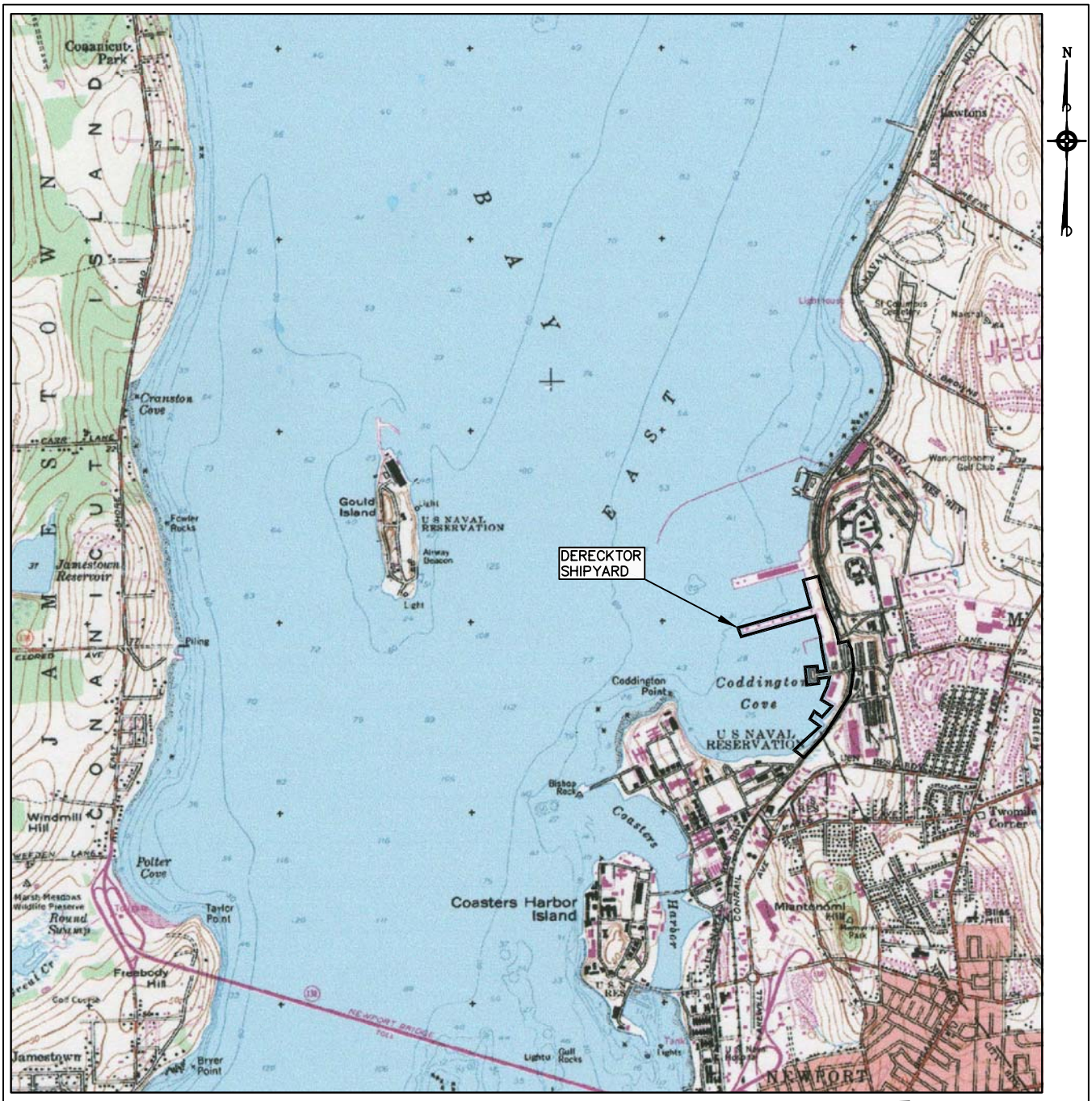


SITE LOCUS	FIGURE 10–1
NAVAL EDUCATION AND TRAINING CENTER	
NEWPORT, RHODE ISLAND	

DRAWN BY:	D.W. MACDOUGALL	REV.:	0
CHECKED BY:	M. HORTON	DATE:	MARCH 8, 2011
SCALE:	AS NOTED	ACAD NAME:	I:\02747\PP.DR\NAVSTA_LOCUS.DWG

TETRA TECH NUS, INC.

55 Jonspin Road Wilmington, MA 01887
(978)658–7899



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

GRAPHIC SCALE
0 0.5 MILE 1 MILE

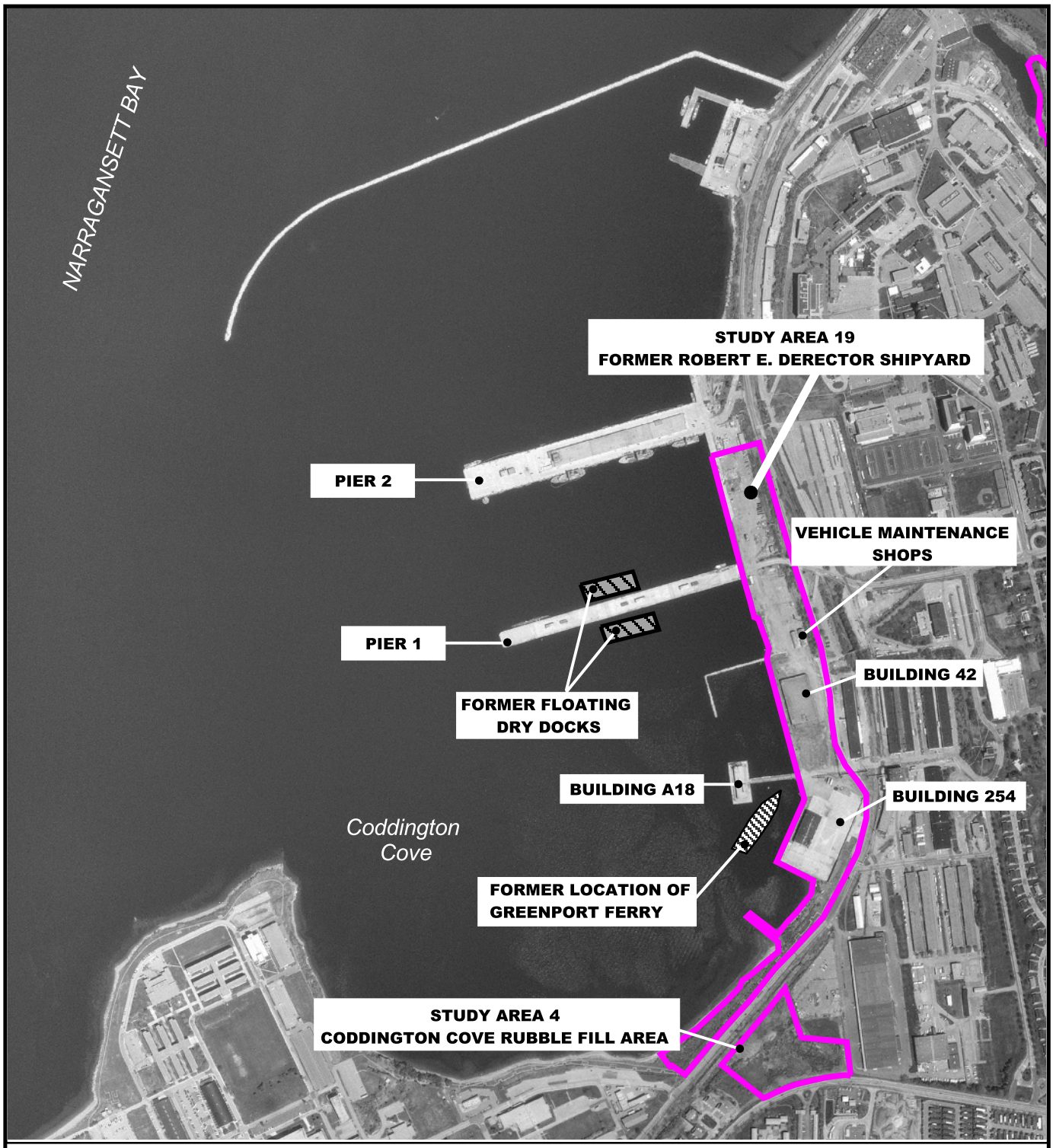
QUADRANGLE LOCATION

SITE LOCUS	FIGURE 10–2
SAMPLING AND ANALYSIS PLAN	
FORMER DERECKTOR MARINE SEDIMENT	
NAVSTA NEWPORT, RHODE ISLAND	

DRAWN BY:	D.W. MACDOUGALL	REV.:	0
CHECKED BY:	M. HORTON	DATE:	MARCH 8, 2011
SCALE:	AS NOTED	ACAD NAME:	I:\02747\PP.DR\DER_LOCUS.DWG

TETRA TECH NUS, INC.

55 Jonspin Road Wilmington, MA 01887
(978)474–8400



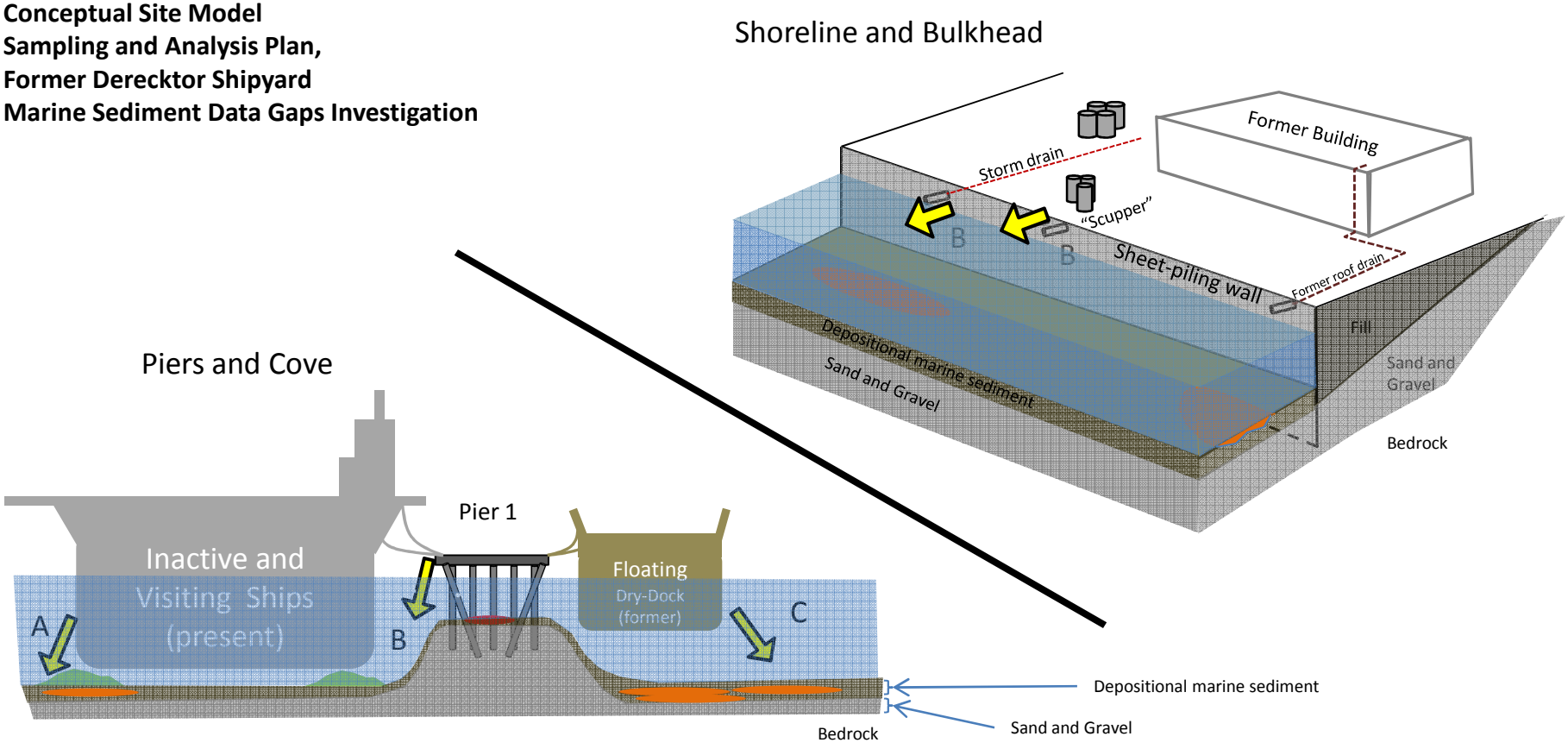
SOURCE:
1997 AERIAL PHOTOGRAPH, RHODE ISLAND DEPARTMENT OF ADMINISTRATION, DIVISION OF PLANNING

300 0 300 600 900 1200 Feet

N

POINTS OF INTEREST	FIGURE 10-3
FORMER ROBERT E. DERECKTOR SHIPYARD NAVSTA NEWPORT - NEWPORT, RHODE ISLAND	TETRA TECH NUS, INC. 55 JONSPIN ROAD WILMINGTON, MA 01887 (978) 658-7899
DRAWN BY: D. MACDOUGALL — DATE: MARCH 9, 2011	
CHECKED BY: M. HORTON — FILE: I:\02747\PP.DR\DER_POI.PDF	

Figure 10-4
Conceptual Site Model
Sampling and Analysis Plan,
Former Derecktor Shipyard
Marine Sediment Data Gaps Investigation



Determine vertical, horizontal extent of target analytes in:

- New sedimentation, trapped by the presence of the vessel, may contain contaminants
- Sediment contaminants possibly trapped by reduced flow between pilings
- Historic sediment contaminants, bedded and possibly covered by recent sedimentation

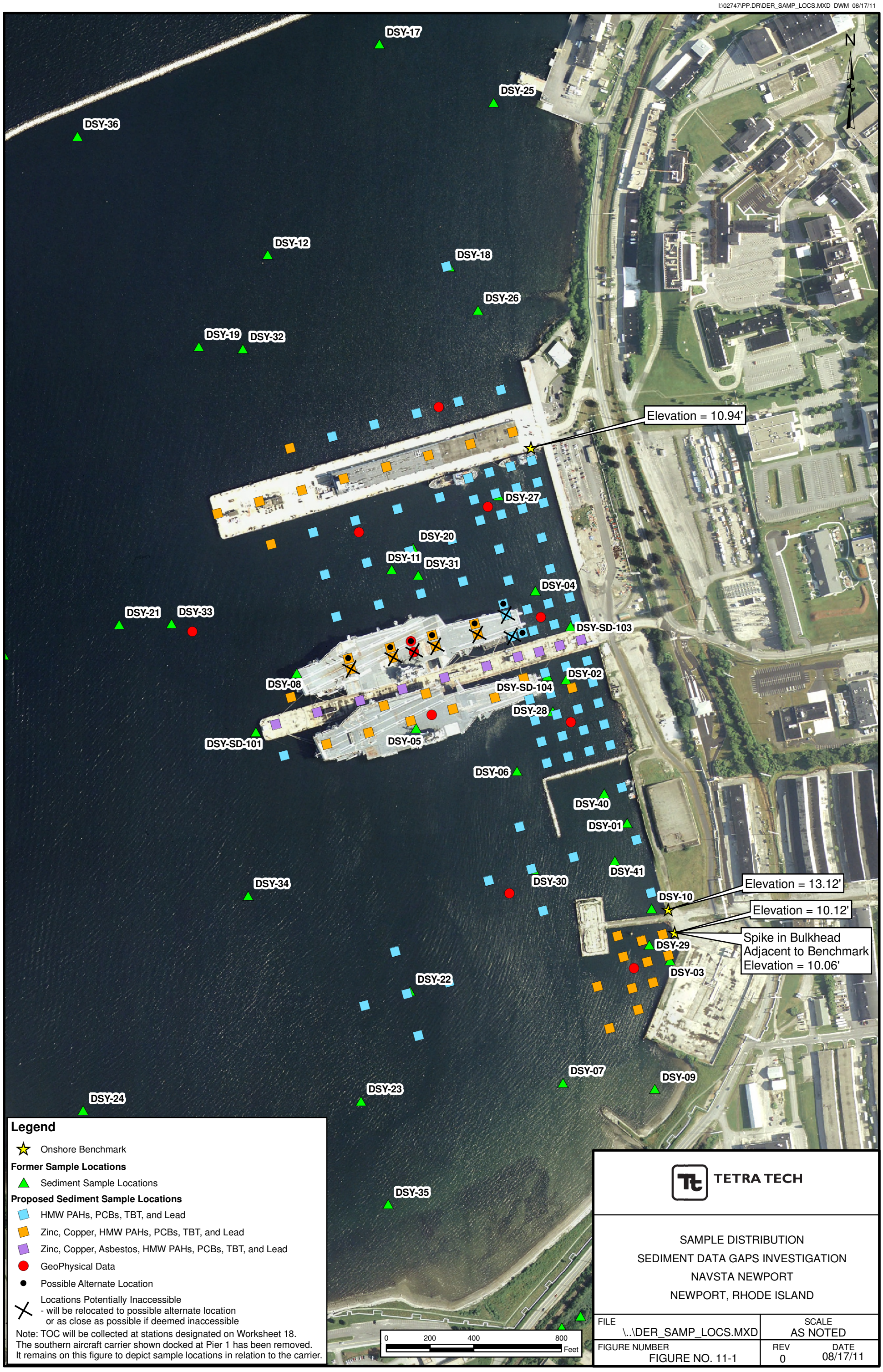
Former Releases Suspected

A – Metals from bottom paints

B – Spillage and discards from the piers and bulkheads on shore

C – Presumed sandblast grit, paints, PAHs from dry docks and maintenance

I:\02747\PP.DR\DER_SAMP_LOCS.MXD DWM 08/17/11



Legend

- Onshore Benchmark

Former Sample Locations

- Sediment Sample Locations

Proposed Sediment Sample Locations

- HMW PAHs, PCBs, TBT, and Lead
- Zinc, Copper, HMW PAHs, PCBs, TBT, and Lead
- Zinc, Copper, Asbestos, HMW PAHs, PCBs, TBT, and Lead
- GeoPhysical Data
- Possible Alternate Location
- Locations Potentially Inaccessible
  - will be relocated to possible alternate location
  or as close as possible if deemed inaccessible

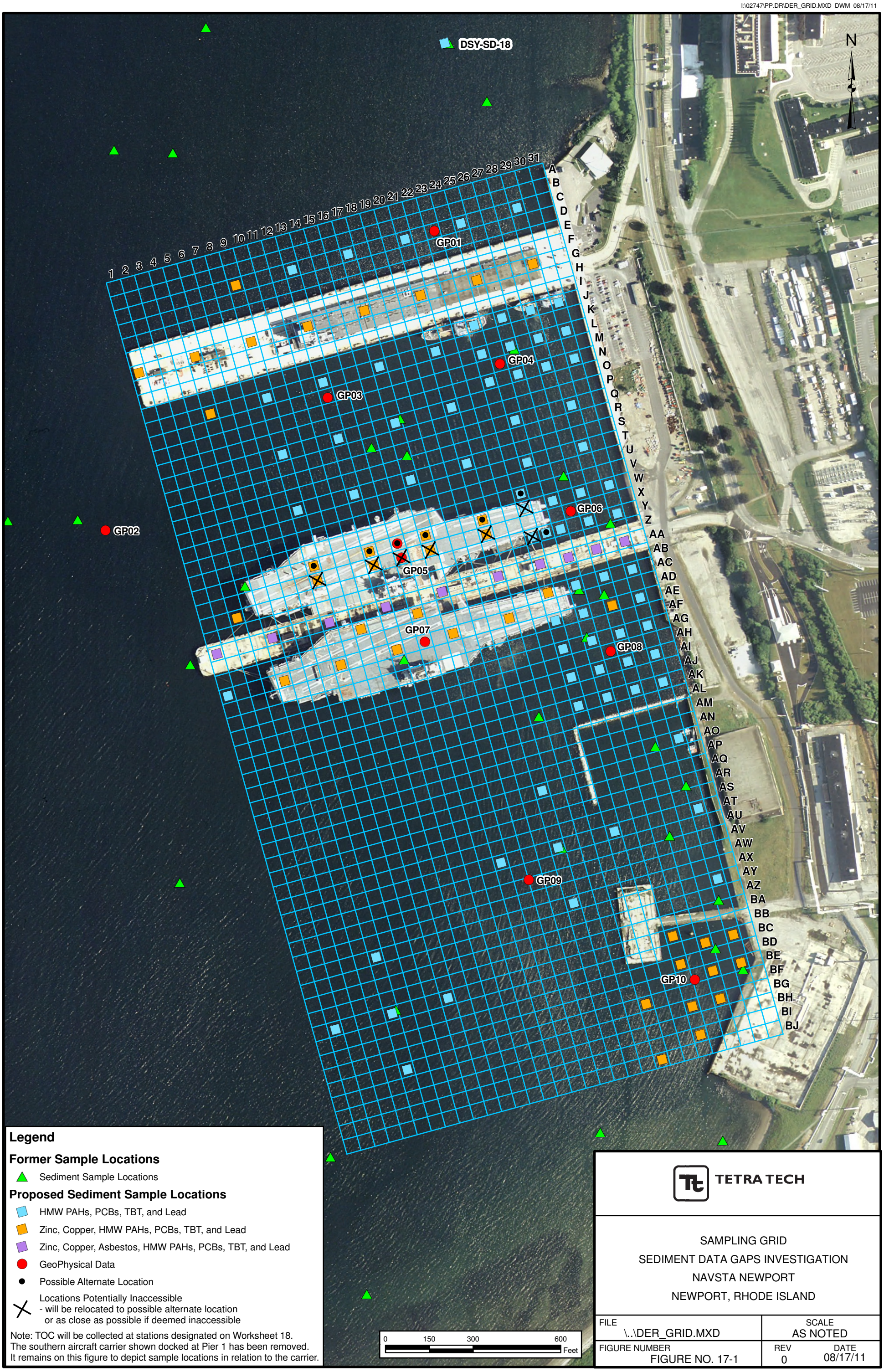
Note: TOC will be collected at stations designated on Worksheet 18.
The southern aircraft carrier shown docked at Pier 1 has been removed.
It remains on this figure to depict sample locations in relation to the carrier.

TETRA TECH

SAMPLE DISTRIBUTION
SEDIMENT DATA GAPS INVESTIGATION
NAVSTA NEWPORT
NEWPORT, RHODE ISLAND

FILE	SCALE
\..\DER_SAMP_LOCS.MXD	AS NOTED
FIGURE NUMBER: FIGURE NO. 11-1	REV: 0 / DATE: 08/17/11

I:\02747\PP.DR\DER_GRID.MXD DWM 08/17/11



Legend

Former Sample Locations

- Sediment Sample Locations

Proposed Sediment Sample Locations

- HMW PAHs, PCBs, TBT, and Lead
- Zinc, Copper, HMW PAHs, PCBs, TBT, and Lead
- Zinc, Copper, Asbestos, HMW PAHs, PCBs, TBT, and Lead
- GeoPhysical Data
- Possible Alternate Location
- Locations Potentially Inaccessible
  - will be relocated to possible alternate location
  or as close as possible if deemed inaccessible

Note: TOC will be collected at stations designated on Worksheet 18.
The southern aircraft carrier shown docked at Pier 1 has been removed.
It remains on this figure to depict sample locations in relation to the carrier.

TETRA TECH

SAMPLING GRID
SEDIMENT DATA GAPS INVESTIGATION
NAVSTA NEWPORT
NEWPORT, RHODE ISLAND

FILE	SCALE
\..\DER_GRID.MXD	AS NOTED
FIGURE NUMBER: FIGURE NO. 17-1	REV: 0 / DATE: 08/17/11

REFERENCES

REFERENCES

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APPENDIX A

Tetra Tech SOPs

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A SITE RECONNAISSANCE CHECKLIST 6

1.0 PURPOSE

The purpose of a site reconnaissance is to collect both general and technical information which will support the scoping, scheduling, implementing project activities, and writing reports for an environmental investigation. This procedure is not intended as a guide for Phase I investigations or for Environmental Baseline Survey activities.

2.0 SCOPE

This procedure is applicable to the performance of a site reconnaissance for initial site characterization. The steps necessary to develop and carry out a site reconnaissance are presented here. These steps include a list of equipment and items which may be needed, areas of special interest during field observations, and methods by which the field observation team can ensure that necessary and appropriate observations have been made.

3.0 GLOSSARY

Site reconnaissance. An onsite inspection program used to identify site-specific conditions that control scheduling, manpower, and affect costs. A site reconnaissance usually consists of visual observations and, often, the use of field monitoring instruments to identify potential health and safety threats and potential sampling locations for site evaluation during subsequent field investigations.

4.0 RESPONSIBILITIES

Field Operations Leader (FOL) is responsible for ensuring that the survey is carried out in sufficient detail. To accomplish this, the FOL must assign the proper personnel and equipment to characterize the site adequately, in accordance with the requirements defined in this procedure and best engineering practices. Other disciplines which may be applicable include (but are not limited to): Geology/Hydrogeology; Health and Safety; Ecological Specialists; and/or Engineering. In addition, the FOL is responsible for supervising equipment preparation, including necessary calibrations, and supervising field data collection and documentation in accordance with the methods described in all referenced standard operation procedures.

Project Manager is responsible for the following:

- Supervising the retrieval and examination of available, applicable information regarding the site.
- Obtaining appropriate program approvals and ensuring the preparation of a site Health and Safety plan for the site reconnaissance.
- Coordinating the field activities with the client and regulatory agencies, as applicable.

Field Personnel are primarily responsible for observing and documenting, either through written documentation or photographic evidence, the site reconnaissance. Field personnel will take direction from the FOL.

5.0 PROCEDURES

5.1 Equipment Items/Needed

Below is a list of items that may be useful when conducting a site reconnaissance. All, or a portion of these items may be required, depending upon the objective of the site reconnaissance.

- Health and Safety equipment and information as required by the Site Safety Officer.
- Maps (U.S.G.S. quadrangle, geologic maps, street and highway maps, and client facility maps).
- Geologic tools (compass, tape measure, hand level, camera, etc.).
- Physical monitoring equipment, if applicable (PID, Immunoassay Test Kits, etc.)
- Regional publications (U.S.G.S reports, water well surveys, U.S.D.A. soil conservation surveys, etc.).
- Site-specific publications by previous investigators (EPA aerial photographic analyses, remedial investigation reports, data on waste disposal practices, boring logs, etc.).
- Marking items (ink markers, surveyor's flagging, spray paint, pin flags, wooden stakes).
- Field notebooks.
- Local telephone book with yellow pages (for obtaining utilities, site trailer, living accommodations, etc.).

Sufficient time will be required in order to obtain some of the aforementioned material. In general, most publications can be obtained in time to be used in the site reconnaissance if ordered approximately 2 weeks before the actual site visit takes place.

5.2 Observations

A site reconnaissance usually requires one to two days, however, additional time may be needed depending upon the objective, site size, etc. The following observations, when applicable, should be documented either on a site map, field notebook, or photographed.

- General Site Access. It should be noted whether site roads provide access to all proposed work locations, or if it will be necessary to prepare access roads with either a backhoe, dozer, chain saws, etc., in order to get drill rigs, excavators, or other work vehicles to specific locations. If temporary driveways must be constructed from existing public roads, regulatory permits may be required. Military facilities may have specific security requirements which require detailed clearance procedures.
- Location of the Command Post or Site Trailer and Sanitary Facilities. The ideal location for the site trailer and sanitary facilities is a level area, within an uncontaminated zone, and centralized in order to provide easy access to work areas on the site. However, certain utility companies may require that the site trailer be placed within a specified radius (usually 100 feet), of the nearest utility pole. Contact the necessary utility companies and inquire about the requirements regarding service before conducting the site reconnaissance. Information that may be required by the utility companies is: type of electric service needed (inquire with trailer vendor for this information); and utility pole number of interest (pole numbers are usually stamped on a brass plate on the pole).

- Potable Water Sources. Local fire departments may allow access to fire hydrants. Private water delivery companies may also be available in the area.

- Sources of Possible Contamination. Drums, tanks, sludge areas, areas of stressed vegetation, fill areas, and leachate seeps may indicate where sources of contamination exist. Filler pipes protruding from the ground surface may indicate the presence of underground storage tanks. Areas where the original ground surface has been reworked may be contaminated fill areas that have since been buried and covered with natural material. Previous environmental investigations may also identify source areas.

- Location of Decon Areas and Storage/Disposal Areas for Equipment and Wastes Generated by Field Activities.

- Locations of Surface Water Bodies. The locations of surface water bodies, both man-made and natural, and their relation to topographic highs may give an indication of the groundwater flow direction in the area (groundwater flow typically follows topography with the topographic highs serving as groundwater recharge areas, and the surface waters at topographic lows serve as groundwater discharge areas). Visible signs of contamination, the existence of aquatic life, flow rates, and approximate levels should also be observed and noted. Check if the surface water bodies could potentially be impacted by field activities. If so, appropriate sedimentation and erosion controls will be required.

- Existing Wells. Existing monitoring wells, or domestic wells within the site and off site, should be noted on a map, and access checked to see if the wells can be used for data collection.

- Outcrops. Outcrops can be useful in providing hydrogeologic data (lithologic description, strike and dip information, fracture and joint system analysis, identification of moist zones, etc.) Outcrops may occur naturally or be a part of a man-made feature such as a road-cut.

- Lineaments. A lineament is a straight lengthy feature on the earth's surface which is expressed topographically as a line of depression. Stream beds, vegetation patterns or soil characteristics may be aligned or controlled by this feature. Lineaments are due in some cases to the presence of intense jointing or faults beneath the ground surface. Groundwater in the bedrock may follow lineaments. Lineaments should be noted on site maps and described in the field notebooks.

- Bench or Property Markers. Benchmarks or property markers should be marked with paint or surveyor's flagging if encountered during a site reconnaissance. Surveyors may need to use these markers as a reference point when surveying. Benchmarks are typically a brass plate secured in concrete in the ground with numbering on the top. Property markers can range from a stake driven into the ground to a rock protruding from the ground surface. Facility contacts may also be aware of local benchmarks used during the course of other environmental or public work projects.

- Metal Cultural Effects. Overhead power lines, railroad tracks, junk automobiles, fences, etc. will greatly affect certain geophysical surveys. These features should be noted while conducting a site reconnaissance.

6.0 RECORDS

The data collected during a site reconnaissance may have to be compiled into a trip report when returning from the field. This trip report can then be distributed to the project team. A site reconnaissance checklist is located in Attachment A which can be copied and used while conducting the site reconnaissance.

ATTACHMENT A

SITE RECONNAISSANCE CHECKLIST

SITE SKETCH

Include the following as appropriate:

- Site Name
- Site location
- Site Boundaries
- Entrance locations
- Access Roads and Security Requirements
- Disposal locations
- Storage areas
- Office areas
- Well locations
- Treatment facility locations
- Surface drainage, outcrops, general topography descriptions
- Cultural interferences

CHEMICAL STORAGE FACILITIES DESCRIPTION

- Storage tanks - numbers, volumes, condition, contents, etc.
- Drums - number, conditions, labeling, etc.
- Lagoons and surface pits - number, size, use of liner, contents, etc.

TREATMENT SYSTEMS

Note the presence of any treatment systems. These can be difficult to evaluate visually. One should appraise general appearance, maintenance and visual integrity; ask operators for any monitoring records; note presence of odors; and visually characterize any effluents or residues. Describe type of wastes and volumes treated.

- Incinerators
- Flocculation/filtration
- Chemical/physical treatment
- Biological treatment
- Volume reduction
- Waste recycling
- Compositing
- Other

ATTACHMENT A
SITE RECONNAISSANCE CHECKLIST
PAGE TWO

DISPOSAL FACILITIES

Note the presence and use of any of the following operations. Include a description of the size, use of liners, soil type, and the presence of leachate. Provide a description of management practices. Interview site workers if possible. Describe waste types.

- Landfills
- Land forms
- Open dump
- Surface impoundment
- Underground injection
- Incineration

Also, records for disposal of concentrated/containerized waste should be reviewed.

HAZARDOUS SUBSTANCE CHARACTERISTICS

Ask facility contacts for manifests, inventories, or monitoring reports. Note markings on containers.

- Chemical identities
- Quantities
- Hazard characteristics (toxic, explosive, flammable, etc.)
- Container markings
- Monitoring data, other analytical data
- Physical state (liquid, solid, gas, sludge)

CHEMICAL PROCESS INFORMATION

- Manufacturing processes and chemicals
- Off-specification or by-product disposal processes
- Housekeeping practices
- Locations of Plant Operations

ATTACHMENT A
SITE RECONNAISSANCE CHECKLIST
PAGE THREE

HYDROGEOLOGIC ASSESSMENT

Look for situations that promote hazardous substance migration, i.e., porous soils, fractured bedrock formations, shallow water table and karst features.

- Soil type
- Surface water features
- Surface drainage pattern
- Outcrop studies
- Water wells (use, water depth, and construction details)
- Erosion potential
- Flooding potential
- Climatology

IDENTIFICATION OF SENSITIVE RECEPTORS

- Number and locations of private homes
- Public buildings including tenant usage
- Areas of dead or dying vegetation or animals
- Presence of sensitive ecosystems (wetlands, tidal marshes, etc.)
- Other public use areas (roads, parks, etc.)
- Natural areas

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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

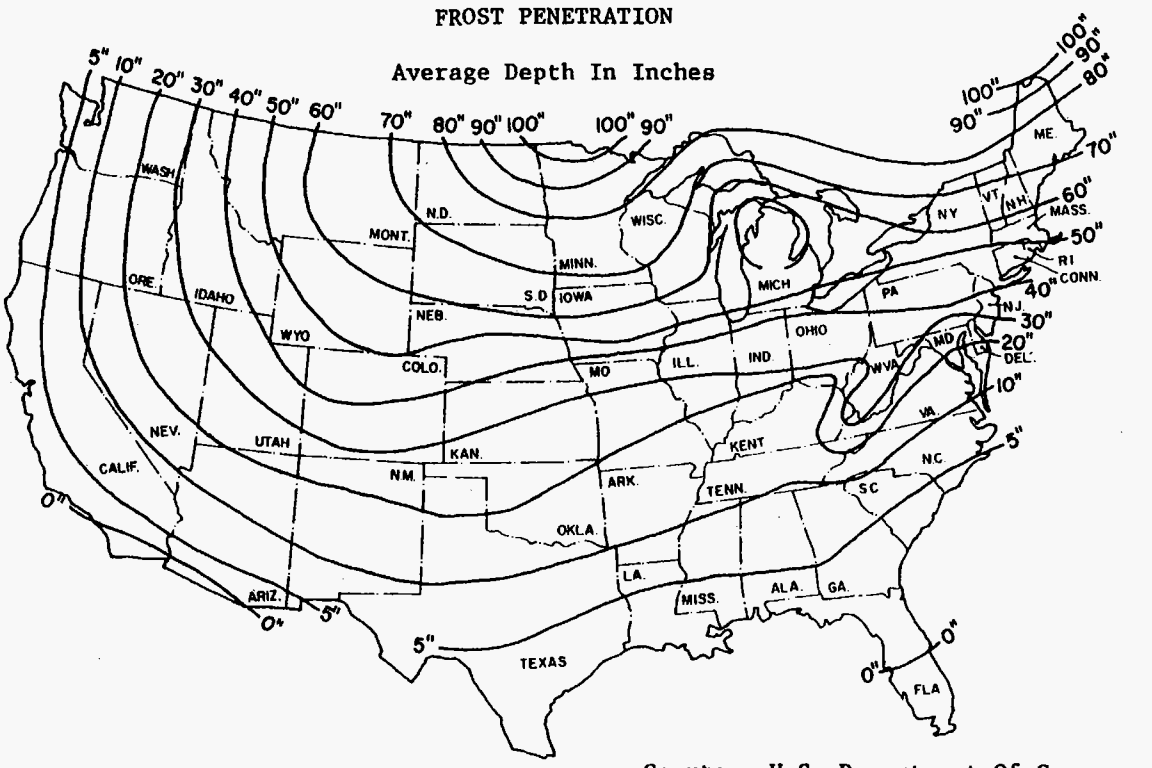
British Columbia
BC One Call
1-800-474-6886

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.

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ATTACHMENTS

A SURFACE WATER SAMPLE LOG SHEET 16
B SOIL & SEDIMENT SAMPLE LOG SHEET 17
C GUIDANCE ON SAMPLE DESIGN AND SAMPLE COLLECTION 18

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes procedures and equipment commonly used for collecting environmental samples of surface water and aquatic sediment for either onsite examination and chemical testing or for offsite laboratory analysis.

2.0 SCOPE

The information presented in this document is applicable to all environmental sampling of surface waters (Section 5.3) and aquatic sediments (Section 5.5), except where the analyte(s) may interact with the sampling equipment. The collection of concentrated sludges or hazardous waste samples from disposal or process lagoons often requires methods, precautions, and equipment different from those described herein.

3.0 GLOSSARY

Analyte – Chemical or radiochemical material whose concentration, activity, or mass is measured.

Composite Sample – A sample representing a physical average of grab samples.

Environmental Sample – A quantity of material collected in support of an environmental investigation that does not require special handling or transport considerations as detailed in SOP SA-6.1.

Grab Sample – A portion of material collected to represent material or conditions present at a single unit of space and time.

Hazardous Waste Sample – A sample containing (or suspected to contain) concentrations of contaminants that are high enough to require special handling and/or transport considerations per SOP SA-6.1.

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

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - The Project Manager is responsible for determining the sampling objectives, initial sampling locations, and field procedures used in the collection of soil samples. The Project Manager also has the overall responsibility for seeing that all surface water and sediment sampling activities are properly conducted by appropriately trained personnel in accordance with applicable planning documents.

Field Operations Leader - This individual is primarily responsible for the execution of the planning document containing the Sampling and Analysis Plan (SAP). This is accomplished through management of a field sampling team for the proper acquisition of samples. He or she is responsible for the supervision of onsite analyses; ensuring proper instrument calibration, care, and maintenance; sample collection and handling; the completion and accuracy of all field documentation; and making sure that

custody of all samples obtained is maintained according to proper procedures. When appropriate and as directed by the FOL, such responsibilities may be performed by other qualified personnel (e.g., field technicians) where credentials and time permit. The FOL is responsible for finalizing the locations for collection of surface water and sediment samples. The FOL is ultimately responsible for adherence to Occupational Safety and Health Administration (OSHA) regulations during these operations through self acquisition or through the management of a field team of samplers.

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

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

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

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.
- Familiarity with appropriate procedures for sample documentation, handling, packaging, and shipping.

5.0 HEALTH AND SAFETY

Precautions to preserve the health and safety of field personnel implementing this SOP are distributed throughout. The following general hazards may also exist during field activities, and the means of avoiding them must be used to preserve the health and safety of field personnel:

Bridge/Boat Sampling – Potential hazards associated with this activity include:

- Traffic – one of the primary concerns as samplers move across a bridge because free space of travel is not often provided. Control measures should include:
  - When sampling from a bridge, if the samplers do not have at least 6 feet of free travel space or physical barriers separating them and the traffic patterns, the HASP will include a Traffic Control Plan.
  - The use of warning signs and high-visibility vests are required to warn oncoming traffic and to increase the visibility of sample personnel.
- Slips, trips, and falls from elevated surfaces are a primary concern. Fall protection shall be worn when or if samplers must lean over a rail to obtain sample material. A Fall Protection Competent

Person (in accordance with Occupational safety and Health Administration [OSHA] fall protection standards) must be assigned to ensure that fall protection is appropriately and effectively employed

- Water hazards/drowning – if someone enters the water from an elevated surface (such as a bridge or dock) and when sampling from a boat. To minimize this potential, personnel shall wear United states Coast Guard (USCG)-approved floatation devices, and the sampling crew must also have on hand a Type IV Throwable Personal Floatation Device with at least 90 feet of 3/8-inch rope. See Section 5.5.2 of this SOP.

- Within the HASP, provisions will also be provided concerning the requirement of a Safe Vessel Certification or the necessity to conduct a boat inspection prior to use. In addition, the HASP shall also specify requirements as to whether the operator must be certified as a commercial boat operator and whether members of the sampling team must have a state-specific safe boating certification.

Entering Water to Collect Samples – Several hazards are associated with this activity and can be mitigated as follows:

- Personnel must wear a USCG-approved Floatation Device (selected and identified in the HASP). The SSO shall ensure that the device selected is in acceptable condition and suitable for the individual using it. This includes consideration of the weight of the individual.

- Lifelines shall be employed from a point on the shore. This activity will always be conducted with a Buddy. See Section 6.5.2.

- Personnel shall carry a probe to monitor the bottom ahead of them for drop offs or other associated hazards.

- The person in the water shall exercise caution concerning the path traveled so that the lifeline does not become entangled in underwater obstructions such as logs, branches, stumps, etc., thereby restricting its effectiveness in extracting the person from the water.

- Personnel shall not enter waters on foot in situations where natural hazards including alligators, snakes, as well as sharks, gars, and other predators within inland waterways may exist.

- In all cases, working along and/or entering the water during high currents or flood conditions shall be prohibited.

- Personnel shall not enter bodies of water where known debris exists that could result in injuries from cuts and lacerations.

Sampling in marshes or tidal areas in some instances can be accomplished using an all-terrain vehicle (ATV). This is not the primary recommended approach because the vehicle may become disabled, or weather conditions or tidal changes could result in environmental damage as well as loss of the vehicle. The primary approach is recommended to be on foot where minimal disturbance would occur. The same precautions specified above with regard to sediment disturbance apply as well as the previously described safety concerns associated with natural hazards. The natural hazards include alligators, bees (nests in dead falls and tree trunks), snakes, etc. In addition, moving through and over this terrain is difficult and could result in muscle strain and slips, trips, and falls. Common sense dictates that the sampler selects the most open accessible route over moderate terrain. Move slowly and deliberately through challenging terrain to minimize falls. Mud boots or other supportive PPE should be considered and specified in the HASP to permit samplers to move over soft terrain with the least amount of effort. In these situations, it is also recommended, as the terrain allows, that supplies be loaded and transported in a sled over the soft ground.

Working in these areas, also recognize the following hazards and means of protection against them:

Insects are also a primary concern. These include mosquitoes, ticks, spiders, bees, ants, etc. The HASP will identify those particular to your area. Typical preventative measures include:

- Use insect repellant. Approval of various repellants should be approved by the Project Chemist or Project Manager.
- Wearing light-colored clothing to control heat load due to excessive temperatures. In addition, it makes it easier to detect crawling insects on your clothing.
- Taping pants to boots to deny access. Again, this is recommended to control access to the skin by crawling insects. Consultation with the Project Health and Safety Officer SSO/Health and Safety Manager is recommended under extreme heat loads because this will create conditions of heat stress.
- Performing a body check to remove insects. The quicker you remove ticks, the less likely they will become attached and transfer bacteria to your bloodstream. Have your Buddy check areas inaccessible to yourself. This includes areas such as the upper back and between shoulder blades where it is difficult for you to examine and even more difficult for you to remove.

Safety Reminder

If you are allergic to bee or ant stings, it is especially critical that you carry your doctor-recommended antidote with you in these remote sampling locations due to the extended time required to extract incapacitated individuals as well as the effort required to extract them. In these scenarios, instruct your Buddy in the proper administration of the antidote. In all cases, if you have received a sting, administer the antidote regardless of the immediate reaction, evacuate, and seek medical attention as necessary. The FOL and/or SSO will determine when and if you may return to the field based on the extent of the immune response and hazards or potential hazards identified in these locations. To the FOL and SSO, this is a serious decision you have to make as to whether to take someone vulnerable to these hazards into a remote location where you may not be able to carry them out. Consider it wisely.

Poisonous Plants – To minimize the potential of encountering poisonous plants in the field, at least one member of the field team needs to have basic knowledge of what these plants look like so that they can be recognized, pointed out to other field personnel, and avoided if at all possible. If the field team cannot avoid contact and must move through an area where these plants exist, the level of personal protective equipment (PPE) shall include Tyvek coveralls and enhanced decontamination procedures for the removal of oils from the tooling and/or equipment.

Temperature-Related Stress – Excessively cold temperatures may result in cold stress, especially when entering the water either intentionally or by accident. Provisions for combating this hazard should be maintained at the sample location during this activity. Excessively hot temperatures may result in heat stress especially in scenarios where equipment is packed through the marsh.

Because all of these activities are conducted outside, electrical storms are a significant concern. The following measures will be incorporated to minimize this hazard:

- Where possible, utilize commercial warning systems and weather alerts to detect storms moving into the area.
- If on or in the water, get out of the water. Move to vehicles or preferably into enclosed buildings with plumbing and wiring.
- Where warning systems are not available, follow the 30/30 Rule (*if there are less than 30 seconds between thunder and lightning, go inside for at least 30 minutes after the last thunder).*

See Section 4.0 of the Health and Safety Guidance Manual (HSGM) for additional protective measures.

6.0 PROCEDURES

6.1 Introduction

Collecting a representative sample of surface water or sediment may be difficult because of water movement, stratification, or heterogeneous distribution of the targeted analytes. To collect representative samples, one must standardize sampling methods related to site selection, sampling frequency, sample collection, sampling devices, and sample handling, preservation, and identification. Regardless of quality control applied during laboratory analyses and subsequent scrutiny of analytical data packages, reported data are no better than the confidence that can be placed in the representativeness of the samples. Consult Appendix C for guidance on sampling that should be considered during project planning and that may be helpful to field personnel.

6.1.1 ~~Surface Water Sampling Equipment~~

~~The selection of sampling equipment depends on the site conditions and sample type to be acquired. In general, the most representative samples are obtained from mid-channel at a stream depth of 0.5 foot in a well-mixed stream; however, project-specific planning documents will address site-specific sampling requirements including sample collection points and sampling equipment. The most frequently used samplers include the following:~~

- ~~Peristaltic pump~~
- ~~Bailer~~
- ~~Dip sampler~~
- ~~Weighted bottle~~
- ~~Hand pump~~
- ~~Kemmerer~~
- ~~Depth-integrating sampler~~

~~The dip sampler and weighted bottle sampler are used most often, and detailed discussions for these devices and the Kemmerer sampler are addressed subsequently in this section~~.

~~The criteria for selecting a sampler include:~~

1. ~~Disposability and/or easy decontamination~~.
2. ~~Inexpensive cost (if the item is to be disposed).~~
3. ~~Ease of operation.~~

4. ~~Non-reactive/non-contaminating properties - Teflon-coated, glass, stainless steel or polyvinyl chloride (PVC) sample chambers are preferred (in that order).~~

~~Measurements collected for each sample (grab or each aliquot collected for compositing) shall include but not be limited to:~~

- ~~Specific conductance~~
- ~~Temperature.~~
- ~~pH.~~
- ~~Dissolved oxygen.~~

~~Sample measurements shall be conducted as soon as the sample is acquired. Measurement techniques described in SOP SA-1.1 shall be followed. All pertinent data and results shall be recorded in a field notebook or on sample log sheets (see Attachment A) or an equivalent electronic form(s). These analyses may be selected to provide information on water mixing/stratification and potential contamination. Various types of water bodies have differing potentials for mixing and stratification.~~

~~In general, the following equipment if necessary for obtaining surface water samples:~~

- ~~Required sampling equipment, which may include a remote sampling pole, weighted bottle sampler, Kemmerer sampler, or other device.~~
- ~~Real-time air monitoring instrument (e.g., PID, FID) as directed in the project-specific planning document.~~
- ~~Required PPE as directed in the project-specific planning document, which may include:~~
  - ~~Nitrile surgeon's or latex gloves (layered as necessary).~~
  - ~~Safety glasses.~~
  - ~~Other items identified on the Safe Work Permit that may be required based on location-specific requirements (e.g., hearing protection, steel-toed work boots, hard hat). These provisions will be listed in the HASP or addressed by the FOL and/or SSO.~~

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

  - ~~Required paperwork (see SOP SA-6.3 and Attachments A and B to this SOP).~~
  - ~~Required decontamination equipment.~~
  - ~~Required sample containers.~~
  - ~~Sealable polyethylene bags (e.g., Ziploc® baggies).~~
  - ~~Heavy duty cooler.~~
  - ~~Ice.~~

- ~~Paper towels and garbage bags~~.
- ~~Chain-of-custody records and custody seals~~.

~~Dip Sampling~~

~~Specific procedures for collecting a dip or grab sample of surface water can vary based on site-specific conditions (e.g., conditions near the shore and how closely a sampler can safely get to the shore). The general procedure for collecting a sample using a pole or directly from the water body is as follows:~~

1. ~~If using a remote sampling pole, securely attach the appropriate sample container to a pole of sufficient length to reach the water to be sampled. Samples for volatile analysis should be collected first. Use PPE as described in the HASP. When sample containers are provided pre-preserved or if the pole cannot accommodate a particular sample container, use a dedicated, clean, unpreserved bottle/container for sampling and transfer to an appropriately preserved container.~~
2. ~~Remove the cap. Do not place the cap on the ground or elsewhere where it might become contaminated.~~
3. ~~Carefully dip the container into the water just below the surface (or as directed by project-specific planning documents), and allow the bottle to fill. Sample bottles for volatile analysis must be filled with no headspace. Avoid contacting the bottom of the water body because this will disturb sediment that may interfere with the surface water sample.~~
4. ~~Retrieve the container and carefully replace the cap securely. If using a container other than the sample bottle, pour the water from that container into the sample bottle and replace the cap securely.~~
5. ~~Use a clean paper towel to clean and dry the outside of the container~~.
6. ~~Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.~~
7. ~~Proceed with the handling and processing of each sample container as described in SOP SA-6.2.~~

~~Constituents measured in grab samples collected near the water surface are only indicative of conditions near the surface of the water and may not be a true representation of the total concentration distributed throughout the water column and in the cross section. Therefore, as possible based on site conditions, the sampler may be required to augment dip samples with samples that represent both dissolved and suspended constituents and both vertical and horizontal distributions.~~

CAUTION

In areas prone to natural hazards such as alligators and snakes, etc., always use a buddy as a watch. Always have and use a lifeline or throwable device to extract persons who could potentially fall into the water. Be attentive to the signs, possible mounds indicating nests, and possible slides into the water. Remember that although snakes are typically encountered on the ground, it is not unheard of to see them on low-hanging branches. Be attentive to your surroundings because these may indicate that hazards are nearby.

~~Weighted Bottle Sampling~~

~~A grab sample can also be collected using a weighted holder that allows a bottle to be lowered to any desired depth, opened for filling, closed, and returned to the surface. This allows discrete sampling with depth. Several of these samples can be combined to provide a vertical composite. Alternatively, an open bottle can be lowered to the bottom and raised to the surface at a uniform rate so that the bottle collects sample throughout the total depth and is just filled on reaching the surface. The resulting sample using either method will roughly approach what is known as a depth-integrated sample.~~

~~A closed weighted bottle sampler consists of glass or plastic bottle with a stopper, a weight and/or holding device, and lines to open the stopper and lower or raise the bottle. The general procedure for sampling with this device is as follows:~~

1. ~~Gently lower the sampler to the desired depth so as not to remove the stopper prematurely (watch for bubbles).~~
2. ~~When the desired depth is reached, pull out the stopper with a sharp jerk of the stopper line.~~
3. ~~Allow the bottle to fill completely, as evidenced by the absence of air bubbles.~~
4. ~~Raise the sampler and cap the bottle.~~
5. ~~Use a paper towel to clean and dry the outside of the container. This bottle can be used as the sample container as long as the bottle is an approved container type.~~
6. ~~Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.~~
7. ~~Proceed with the handling and processing of each sample container as described in SOP SA-6.2.~~

~~Kemmerer Sampler~~

~~If samples are desired at a specific depth, and the parameters to be measured do not require a Teflon-coated sampler, a standard Kemmerer sampler may be used. The Kemmerer sampler is a brass, stainless steel or acrylic cylinder with rubber stoppers that leave the ends open while it is lowered in a vertical position (thus allowing free passage of water through the cylinder). A "messenger" is sent down the line when the sampler is at the designated depth to cause the stoppers to close the cylinder, which is then raised. Water is removed through a valve to fill sample bottles. The general procedure for sampling with this device is as follows:~~

1. ~~Gently lower the sampler to the desired depth.~~
2. ~~When the desired depth is reached, send down the messenger to close the cylinder and then raise the sampler.~~
3. ~~Open the sampler valve to fill each sample bottle (filling bottles for volatile analysis first).~~
4. ~~Use a paper towel to clean and dry the outside of the container.~~
5. ~~Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.~~
6. ~~Proceed with the handling and processing of each sample container as described in SOP SA-6.2.~~

6.1.2 ~~Surface Water Sampling Techniques~~

~~Samples collected during site investigations may be grab samples or composite samples. The following general procedures apply to various types of surface water collection techniques:~~

- ~~If a clean, pre-preserved sample container is not used, rinse the sample container least once with the water to be sampled before the sample is collected. This is not applicable when sample containers are provided pre-preserved because doing so will wash some or all of the preservative out of the bottle.~~

- ~~For sampling moving water, collect the farthest downstream sample first, and continue sample collection in an upstream direction. In general, work from zones suspected of low contamination to zones of high contamination.~~

- ~~Take care to avoid excessive agitation of the water because loss of volatile constituents could result.~~

- ~~When obtaining samples in 40 mL vials with septum-lined lids for volatile organics analysis, fill the container completely (with a meniscus) to exclude any air space in the top of the bottle and to be sure that the Teflon liner of the septum faces in after the vial is filled and capped. Turn the vial upside down and tap gently on your wrist to check for air bubbles. If air bubbles rise in the bottle, add additional sample volume to the container.~~

- ~~Do not sample at the surface, unless sampling specifically for a known constituent that is immiscible and on top of the water. Instead, invert the sample container, lower it to the approximate depth, and hold it at about a 45-degree angle with the mouth of the bottle facing upstream.~~

6.2 ~~Onsite Water Quality Testing~~

~~Onsite water quality testing shall be conducted as described in SOP SA-1.1.~~

6.3 Sediment Sampling

6.3.1 General

If composite surface water samples are collected, sediment samples are usually collected at the same locations as the associated surface water samples. If only one sediment sample is to be collected, the sampling location shall be approximately at the center of the water body, in a depositional area if possible based on sample location restraints (see below), unless the SAP states otherwise.

Generally, coarser-grained sediments are deposited near the headwaters of reservoirs. Bed sediments near the center of a water body will be composed of fine-grained materials that may, because of their lower porosity and greater surface area available for adsorption, contain greater concentrations of contaminants. The shape, flow pattern, bathymetry (i.e., depth distribution), and water circulation patterns must all be considered when selecting sediment sampling sites. In streams, areas likely to have sediment accumulation (e.g., bends, behind islands or boulders, quiet shallow areas or very deep, low-velocity areas) shall be sampled, in general, and areas likely to show net erosion (i.e., high-velocity, turbulent areas) and suspension of fine solid materials shall be generally avoided. Follow instructions in the SAP, as applicable.

Chemical constituents associated with bottom material may reflect an integration of chemical and biological processes. Bottom samples reflect the historical input to streams, lakes, and estuaries with

respect to time, application of chemicals, and land use. Bottom sediments (especially fine-grained material) may act as a sink or reservoir for adsorbed heavy metals and organic contaminants (even if water column concentrations are less than detection limits). Therefore, it is important to minimize the loss of low-density "fines" during any sampling process.

Samples collected for volatile organic compound (VOC) analysis must be collected prior to any sample homogenization. Regardless of the method used for collection, the aliquot for VOC analysis must be collected directly from the sampling device (hand auger bucket, scoop, trowel), to the extent practical. If a device such as a dredge is used, the aliquot should be collected after the sample is placed in the mixing container prior to mixing.

In some cases, the sediment may be soft and not lend itself to collection by plunging Encore™ or syringe samplers into the sample matrix. In these cases, it is appropriate to open the sampling device, (Encore™ barrel or syringe) prior to sample collection, and carefully place the sediment in the device, filling it fully with the required volume of sample.

On active or former military sites, ordnance items may be encountered in some work areas. Care should be exercised when handling site media (such as if unloading a dredge as these materials may be scooped up). If suspected ordnance items are encountered, stop work immediately, move to shore and notify the Project Manager and Health and Safety Manager.

All relevant information pertaining to sediment sampling shall be documented as applicably described in SOP SA-6.3 and Attachment B or an equivalent electronic form.

6.3.2 Sampling Equipment and Techniques for Bottom Materials

A bottom-material sample may consist of a single scoop or core, or may be a composite of several individual samples in the cross section. Sediment samples may be obtained using onshore or offshore techniques.

SAFETY REMINDER

The following health and safety provisions apply when working on/over/near water:

- At least two people are required to be present at the sampling location in situations where the water depth and/or movement deem it necessary, each wearing a USCG-approved Personal Flotation Devices

- A minimum of three people are required if *any* of the following conditions are anticipated or observed:

  - Work in a waterway that is turbulent *or* swift that could sweep a sampler down stream should he or she fall in accidentally.

  - The underwater walking surface (e.g., stream/river bed) is suspected or observed to involve conditions that increase the potential for a worker to fall into the water. Examples include large/uneven rocks or boulders, dense mud or sediment that could entrap worker's feet, etc.

  - Waterway is tidal, and conditions such as those listed above could rapidly change.

The third person in the above condition must be equipped and prepared to render emergency support [e.g., lifeline, tethered Personal Flotation Device (Throwable Type IV, life saver), skiff, means to contact external emergency response support, etc.]

The following samplers may be used to collect sediment samples:

- ~~Scoop sampler~~
- ~~Dredge samplers~~
- Coring samplers

Each type of sampler is discussed below.

In general, the following equipment if necessary for obtaining sediment samples:

- Required sampling equipment, which may include a scoop sampler, dredge sampler, coring sampler, or stainless steel or pre-cleaned disposable trowel.
- Stainless bowl or pre-cleaned disposable bowl to homogenize sample.
- Real-time air monitoring instrument (e.g., PID, FID) as directed in the project-specific planning document.
- Required PPE as directed in the project-specific planning document, which may include:
  - Nitrile surgeon's or latex gloves (layered as necessary).
  - Safety glasses.
  - Other items identified on the Safe Work Permit that may be required based on location-specific requirements (e.g., hearing protection, steel-toed work boots, hard hat). These provisions will be listed in the HASP or addressed by the FOL and/or SSO.
  - Required paperwork (see SOP SA-6.3 and Attachments A and B to this SOP).
  - Required decontamination equipment.
  - Required sample containers.
  - Sealable polyethylene bags (e.g., Ziploc® baggies).
  - Heavy-duty cooler.
  - Ice.
  - Paper towels and garbage bags.
  - Chain-of-custody records and custody seals.

~~Scoop Sampler~~

~~A scoop sampler consists of a pole to which a jar or scoop is attached. The pole may be made of bamboo, wood, PVC, or aluminum and be either telescoping or of fixed length. The scoop or jar at the end of the pole is usually attached using a clamp.~~

~~If the water body can be sampled from the shore or if the sampler can safely wade to the required location, the easiest and best way to collect a sediment sample is to use a scoop sampler. Scoop sampling also reduces the potential for cross-contamination. The general scoop sampling procedure is as follows:~~

1. ~~Reach over or wade into the water body~~.

2. ~~While facing upstream (into the current), scoop the sampler along the bottom in an upstream direction. Although it is very difficult not to disturb fine-grained materials at the sediment-water interface when using this method, try to keep disturbances to a minimum.~~

~~Dredge Samplers~~

~~Dredges are generally used to sample sediments that cannot easily be obtained using coring devices (e.g., coarse grained or partially cemented materials) or when large quantities of sample are required. Dredges generally consist of a clam shell arrangement of two buckets. The buckets may either close upon impact or be activated by use of a "messenger." Some dredges are heavy and may require use of a winch and crane assembly for sample retrieval. The three major types of dredges are Peterson, Eckman and Ponar.~~

~~The Peterson dredge is used when the bottom is rocky, in very deep water, or when the flow velocity is high. The Peterson dredge shall be lowered very slowly as it approaches bottom, because it can force out and miss lighter materials if allowed to drop freely.~~

~~The Eckman dredge has only limited usefulness. It performs well where bottom material is unusually soft, as when covered with organic sludge or light mud. It is unsuitable, however, for sandy, rocky, and hard bottoms and is too light for use in streams with high flow velocities.~~

~~The Ponar dredge is a Peterson dredge modified by the addition of side plates and a screen on the top of the sample compartment. The screen over the sample compartment permits water to pass through the sampler as it descends, thus reducing the "shock wave." The Ponar dredge is easily operated by one person in the same fashion as the Peterson dredge. The Ponar dredge is one of the most effective samplers for general use on all types of substrates.~~

~~The general procedure for using dredge samplers is as follows:~~

1. ~~Gently lower the dredge to the desired depth.~~

2. ~~When the desired depth is reached, send the messenger down to cable to close the cylinder and then carefully raise the sampler~~.

3. ~~Open the sampler to retrieve the sediment.~~

4. ~~Transfer the sediment to the bowl in which it will be homogenized. Fill the sample bottle(s) for volatile analysis *prior to* homogenization. Homogenize the remainder of the sediment collected.~~

5. ~~Fill the containers for all analyses other and VOCs.~~

6. ~~Use a paper towel to clean and dry the outside of each container.~~

7. ~~Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.~~

8. ~~Proceed with the handling and processing of each sample container as described in SOP SA-6.2.~~

SAFETY REMINDER

Safety concerns using these dredges include lifting hazards, pinches, and compressions (several pinch points exist within the jaws and levers). In all cases, handle the dredge by the rope to avoid capturing fingers/hands.

Coring Samplers

Coring samplers are used to sample vertical columns of sediment. Many types of coring devices have been developed depending on the depth of water from which the sample is to be obtained, the nature of the bottom material, and the length of core to be collected. They vary from hand-push tubes to electronic vibrational core tube drivers.

Coring devices are particularly useful in pollutant monitoring because turbulence created by descent through the water is minimal, thus the fines at the sediment-water interface are only minimally disturbed. The sample is withdrawn intact, permitting the removal of only those layers of interest.

In shallow, wadeable waters, the use of a core liner or tube manufactured of Teflon or plastic is recommended for the collection of sediment samples. Caution should be exercised not to disturb the bottom sediments when the sample is obtained by wading in shallow water. The general procedure to collecting a sediment sample with a core tube is as follows:

1. Push the tube into the substrate until 4 inches or less of the tube is above the sediment-water interface. When sampling hard or coarse substrates, a gentle rotation of the tube while it is being pushed will facilitate greater penetration and decrease core compaction.

2. Cop the top of the tube to provide suction and reduce the chance of losing the sample.

3. Slowly extract the tube so as not to lose sediment from the bottom of the tube. Cap the bottom of the tube before removing it from the water. This will also help to minimize loss of sample.

4. Transfer the sediment to the bowl in which it will be homogenized. Fill the sample bottle(s) for volatile analysis prior to homogenization. Homogenize the remainder of the sediment collected.

5. Fill the containers for all analyses other and VOCs.

6. Use a paper towel to clean and dry the outside of each container.

7. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.

8. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

In deeper, non-wadeable water bodies, sediment cores may be collected from a bridge or boat using different coring devices such as Ogeechee Sand Pounders, gravity cores, and vibrating coring devices.

All three devices utilize a core barrel with a core liner tube system. The core liners can be removed from the core barrel and replaced with a clean core liner after each sample. Before extracting the sediment from the coring tubes, the clear supernatant above the sediment-water interface in the core should be decanted from the tube. This is accomplished by turning the core tube to its side and gently pouring the liquid out until fine sediment particles appear in the waste liquid. Post-retrieval processing of samples is the same as above.

7.0 REFERENCES

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U.S. EPA, 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Water Surveillance Branch, USEPA Surveillance and Analytical Division, Athens, Georgia.

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ATTACHMENT A
SURFACE WATER SAMPLE LOG SHEET

Tetra Tech NUS, Inc. **SURFACE WATER SAMPLE LOG SHEET**

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Stream
[] Spring
[] Pond
[] Lake
[] Other: _____
[] QA Sample Type: _____

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

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

SAMPLING DATA:

	Color	pH	S.C.	Temp.	Turbidity	DO	Salinity	Other
Date:	Visual	Standard	mS/cm	Degrees C	NTU	mg/l	%	NA
Time:								
Depth:								
Method:								

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

ATTACHMENT B
SOIL & SEDIMENT SAMPLE LOG SHEET

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

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

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

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

GRAB SAMPLE DATA:

Date:	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):

APPENDIX C
GUIDANCE ON SAMPLING DESIGN AND SAMPLE COLLECTION

C.1 Defining the Sampling Program

Many factors are considered in developing a sampling program for surface water and/or sediment, including study objectives, accessibility, site topography, physical characteristics of the water body (e.g., flow and mixing), point and diffuse sources of contamination, and personnel and equipment available to conduct the study. For waterborne constituents, dispersion depends on vertical and lateral mixing within the body of water. For sediment, dispersion depends on bottom current or flow characteristics, sediment characteristics (e.g., density, size), and geochemical properties (that affect adsorption/desorption). The hydrogeologist developing the sampling plan must therefore know not only the mixing characteristics of streams and lakes but must also understand the role of fluvial-sediment transport, deposition, and chemical sorption.

C.1.1 Sampling Program Objectives

The scope of the sampling program must consider the sources and potential pathways for transport of contamination to or within a surface water body. Sources may include point sources (leaky tanks, outfalls, etc.) or nonpoint sources (e.g., contaminated runoff). The major pathways for surface water contamination (not including airborne deposition) are overland runoff, leachate influx to the water body, direct waste disposal (solid or liquid) into the water body, and groundwater flow influx from upgradient. The relative importance of these pathways, and therefore the design of the sampling program, is controlled by the physiographic and hydrologic features of the site, the drainage basin(s) that encompasses the site, and the history of site activities.

Physiographic and hydrologic features to be considered include slopes and runoff direction, areas of temporary flooding or pooling, tidal effects, artificial surface runoff controls such as berms or drainage ditches (and when they were constructed relative to site operation), and locations of springs, seeps, marshes, etc. In addition, the obvious considerations such as the locations of man-made discharge points to the nearest stream (intermittent or flowing), pond, lake, estuary, etc. shall be considered.

A more subtle consideration in designing the sampling program is the potential for dispersion of dissolved or sediment-associated contaminants away from the source. The dispersion could lead to a more homogeneous distribution of contamination at low or possibly non-detectable concentrations. Such dispersion does not, however, always readily occur. For example, obtaining a representative sample of contamination from a main stream immediately below an outfall or a tributary is difficult because the inflow frequently follows a stream bank with little lateral mixing for some distance. Sampling alternatives to overcome this situation include: (1) moving the sampling location far enough downstream to allow for adequate mixing, or (2) collecting integrated samples in a cross section. Also, non-homogeneous distribution is a particular problem with regard to sediment-associated contaminants, which may accumulate in low-energy environments (coves, river bends, deep spots, or even behind boulders) near or distant from the source while higher-energy areas (main stream channels) near the source may show no contaminant accumulation.

The distribution of particulates within a sample itself is an important consideration. Many organic compounds are only slightly water soluble and tend to adsorb onto particulate matter. Nitrogen, phosphorus, and heavy metals may also be transported by particulates. Samples must be collected with a representative amount of suspended material; transfer from the sampling device shall include transferring a proportionate amount of the suspended material.

C.1.2 Location of Sampling Stations

Accessibility is the primary factor affecting sampling costs. The desirability and utility of a sample for analysis and consideration of site conditions must be balanced against the costs of collection as controlled by accessibility. Bridges or piers are the first choice for locating a sampling station on a stream because bridges provide ready access and also permit the sampling technician to sample any point across the stream. A boat or pontoon (with an associated increase in cost) may be needed to sample locations on lakes, reservoirs, or larger rivers. Frequently, however, a boat will take longer to cross a water body and will hinder manipulation of the sampling equipment. Wading for samples is not recommended unless it is known that contaminant levels are low so that skin contact will not produce adverse health effects. This provides a built in margin of safety in the event that wading boots or other protective equipment should fail to function properly. If it is necessary to wade into the water body to obtain a sample, the sampler shall be careful to minimize disturbance of bottom sediments and must enter the water body downstream of the sampling location. If necessary, the sampling technician shall wait for the sediments to settle before taking a sample.

Under ideal and uniform contaminant dispersion conditions in a flowing stream, the same concentrations of each contaminant would occur at all points along the cross section. This situation is most likely downstream of areas of high turbulence. Careful site selection is needed to ensure, as nearly as possible, that samples are taken where uniform flow or deposition and good mixing conditions exist.

The availability of stream flow and sediment discharge records can be an important consideration in choosing sampling sites in streams. Stream flow data in association with contaminant concentration data are essential for estimating the total contaminant loads carried by the stream. If a gaging station is not conveniently located on a selected stream, the project hydrogeologist shall explore the possibility of obtaining stream flow data by direct or indirect methods. Remember these locations are also where you may encounter natural hazards as these are areas where they hunt. Always exercise extreme caution.

C.1.3 Frequency of Sampling

The sampling frequency and objectives of the sampling event will be defined by the project planning documents. For single-event site or area characterization sampling, both bottom material and overlying water samples shall be collected at the specified sampling stations. If valid data are available on the distribution of a contaminant between the solid and aqueous phases, it may be appropriate to sample only one phase, although this is not often recommended. If samples are collected primarily for monitoring purposes (i.e., consisting of repetitive, continuing measurements to define variations and trends at a given location), water samples should be collected at a pre-established and constant interval as specified in the project plans (often monthly or quarterly and during droughts and floods). Samples of bottom material should generally be collected from fresh deposits at least yearly, and preferably seasonally, during both spring and fall.

The variability in available water quality data shall be evaluated before determining the number and collection frequency of samples required to maintain an effective monitoring program.

C.2 Surface Water Sample Collection

C.2.1 Streams, Rivers, Outfalls and Drainage Features

Methods for sampling streams, rivers, outfalls, and drainage features (ditches, culverts) at a single point vary from the simplest of hand-sampling procedures to the more sophisticated multi-point sampling techniques known as the equal-width-increment (EWI) method or the equal-discharge-increment (EDI) methods (see below).

Samples from different depths or cross-sectional locations in the watercourse taken during the same sampling episode shall be composited. However, samples collected along the length of the watercourse or at different times may reflect differing inputs or dilutions and therefore shall not be composited. Generally, the number and type of samples to be taken depend on the river's width, depth, and discharge and on the suspended sediment the stream or river transports. The greater the number of individual points that are sampled, the more likely that the composite sample will truly represent the overall characteristics of the water.

In small streams less than about 20 feet wide, a sampling site can generally be found where the water is well mixed. In such cases, a single grab sample taken at mid-depth in the center of the channel is adequate to represent the entire cross section.

For larger streams, at least one vertical composite shall be taken with one sample each from just below the surface, at mid-depth, and just above the bottom. The measurement of dissolved oxygen (DO), pH, temperature, conductivity, etc., shall be made on each aliquot of the vertical composite and on the composite itself. For rivers, several vertical composites shall be collected, as directed in the project planning documents.

C.2.2 Lakes, Ponds and Reservoirs

Lakes, ponds, and reservoirs have a much greater tendency to stratify than rivers and streams. The relative lack of mixing requires that more samples be obtained. The number of water sampling sites on a lake, pond, or impoundment will vary with the size and shape of the basin. In ponds and small lakes, a single vertical composite at the deepest point may be sufficient. Similarly, measurement of DO, pH, temperature, etc. is to be conducted on each aliquot of the vertical composite and on the composite itself. In naturally formed ponds, the deepest point may have to be determined empirically; in impoundments, the deepest point is usually near the dam.

In lakes and larger reservoirs, several vertical composites shall be composited to form a single sample if a sample representative of the water column is required. These vertical composites are often collected along a transect or grid. In some cases, it may be of interest to form separate composites of epilimnetic and hypolimnetic zones. In a stratified lake, the epilimnion is the thermocline that is exposed to the atmosphere. The hypolimnion is the lower, "confined" layer that is only mixed with the epilimnion and vented to the atmosphere during seasonal "overturn" (when density stratification disappears). These two zones may thus have very different concentrations of contaminants if input is only to one zone, if the contaminants are volatile (and therefore vented from the epilimnion but not the hypolimnion), or if the epilimnion only is involved in short-term flushing (i.e., inflow from or outflow to shallow streams). Normally, however, a composite consists of several vertical composites with samples collected at various depths.

In lakes with irregular shape and with bays and coves that are protected from the wind, separate composite samples may be needed to adequately represent water quality because it is likely that only poor mixing will occur. Similarly, additional samples are recommended where discharges, tributaries, land use characteristics, and other such factors are suspected of influencing water quality.

Many lake measurements are now made in situ using sensors and automatic readout or recording devices. Single and multi-parameter instruments are available for measuring temperature, depth, pH, oxidation-reduction potential (ORP), specific conductance, DO, some cations and anions, and light penetration.

C.2.3 Estuaries

Estuarine areas are, by definition, zones where inland freshwaters (both surface and ground) mix with oceanic saline waters. Knowledge of the estuary type may be necessary to determine sampling locations. Estuaries are generally categorized into one of the following three types dependent on freshwater inflow and mixing properties:

- Mixed Estuary - characterized by the absence of a vertical halocline (gradual or no marked increase in salinity in the water column) and a gradual increase in salinity seaward. Typically, this type of estuary is shallow and is found in major freshwater sheet flow areas. Because this type of estuary is well mixed, sampling locations are not critical.

- Salt Wedge Estuary - characterized by a sharp vertical increase in salinity and stratified freshwater flow along the surface. In these estuaries, the vertical mixing forces cannot override the density differential between fresh and saline waters. In effect, a salt wedge tapering inland moves horizontally back and forth with the tidal phase. If contamination is being introduced into the estuary from upstream, water sampling from the salt wedge may miss it entirely.

- Oceanic Estuary - characterized by salinities approaching full-strength oceanic waters. Seasonally, freshwater inflow is small, with the preponderance of the fresh-saline water mixing occurring near or at the shore line.

Sampling in estuarine areas is normally based on the tidal phase, with samples collected on successive slack tides (i.e., when the tide turns). Estuarine sampling programs shall include vertical salinity measurements at 1- to 5-foot increments, coupled with vertical DO and temperature profiles.

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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 (y261.30 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 (y261.20 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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		Effective Date 03/09/09	Revision 3
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject FIELD DOCUMENTATION		Approved Tom Johnston	

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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, Inc. (TtNUS) field activities.

2.0 SCOPE

Documents presented within this SOP (or equivalents) shall be used for all TtNUS field activities, as applicable. Other or additional documents may be required by specific client contracts or project planning documents.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager (PM) - The PM is responsible for obtaining hardbound controlled-distribution logbooks (from the appropriate source), as needed. In addition, the Project Manager is responsible for placing all field documentation used in site activities (i.e., records, field reports, sample data sheets, field notebooks, and the site logbook) in the project's central file upon the completion of field work.

Field Operations Leader (FOL) - The FOL is responsible for ensuring that the site logbook, notebooks, and all appropriate and current forms and field reports included in this SOP (and any additional forms required by the contract) are correctly used, accurately filled out, and completed in the required time frame.

General personnel qualifications for field documentation activities include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.
- Familiarity with appropriate procedures for documentation, handling, packaging, and shipping.

5.0 PROCEDURES

5.1 SITE LOGBOOK

5.1.1 General

The site logbook is a hard-bound, paginated, controlled-distribution record book in which all major on-site activities are documented. At a minimum, record or reference the following activities/events (daily) in the site logbook:

- All field personnel present
- Arrival/departure times and names of site visitors
- Times and dates of health and safety training
- Arrival/departure times of equipment
- Times and dates of equipment calibration

- Start and/or completion of borehole, trench, monitoring well installation activities, etc.
- Daily on-site activities
- Sample pickup information
- Health and safety issues (level of protection, personal protective equipment [PPE], etc.)
- Weather conditions

Maintain a site logbook for each project and initiate it at the start of the first on-site activity (e.g., site visit or initial reconnaissance survey). Make entries every day that on-site activities take place involving TtNUS or subcontractor personnel. Upon completion of the fieldwork, provide the site logbook to the PM or designee for inclusion in the project's central file.

Record the following information on the cover of each site logbook:

- Project name
- TtNUS project number
- Sequential book number
- Start date
- End date

Information recorded daily in the site logbook need not be duplicated in other field notebooks (see Section 5.2) but must summarize the contents of these other notebooks and refer to specific page locations in these notebooks for detailed information (where applicable). An example of a typical site logbook entry is shown in Attachment A.

If measurements are made at any location, either record the measurements and equipment used in the site logbook or reference the field notebook in which the measurements are recorded (see Attachment A).

Make all logbook, notebook, and log sheet entries in indelible ink (black pen is preferred). No erasures are permitted. If an incorrect entry is made, cross out the entry with a single strike mark, initial, and date it. At the completion of entries by any individual, the logbook pages used must be signed and dated by the person making the entries. The site logbook must also be signed by the FOL at the end of each day.

5.1.2 Photographs

Sequentially number movies, slides, or photographs taken of a site or any monitoring location to correspond to logbook/notebook entries. Enter the name of the photographer, date, time, site location, site description, and weather conditions in the logbook/notebook as the photographs are taken. A series entry may be used for rapid-sequence photographs. The photographer is not required to record the aperture settings and shutter speeds for photographs taken within the normal automatic exposure range. However, special lenses, films, filters, and other image-enhancement techniques must be noted in the logbook/notebook. If possible, such techniques shall be avoided because they can adversely affect the accuracy of photographs. Chain-of-custody procedures depend on the subject matter, type of camera (digital or film), and the processing it requires. Follow chain-of-custody procedures for film used for aerial photography, confidential information, or criminal investigation. After processed, consecutively number the slides of photographic prints and label them according to the logbook/notebook descriptions. Docket the site photographs and associated negatives and/or digitally saved images to compact disks into the project's central file.

5.2 FIELD NOTEBOOKS

Key field team personnel may maintain a separate dedicated field notebook to document the pertinent field activities conducted directly under their supervision. For example, on large projects with multiple investigative sites and varying operating conditions, the Health and Safety Officer may elect to maintain a

separate field notebook. Where several drill rigs are in operation simultaneously, each site geologist assigned to oversee a rig must maintain a field notebook.

5.3 FIELD FORMS

All TtNUS field forms (see list in Section 6.0 of this SOP) can be found on the company's intranet site (http://intranet.ttnus.com) under Field Log Sheets. Forms may be altered or revised for project-specific needs, subject to client approval. Care must be taken to ensure that all essential information can be documented. Guidelines for completing these forms can be found in the related sampling SOPs.

5.3.1 Sample Collection, Labeling, Shipment, Request for Analysis, and Field Test Results

5.3.1.1 Sample Log Sheet

Sample log sheets are used to record specified types of data while sampling. The data recorded on these sheets are useful in describing the sample as well as pointing out any problems, difficulties, or irregularities encountered during sampling. Complete a sample log sheet for each sample obtained, including field quality control (QC) samples.

5.3.1.2 Sample Label

A typical sample label is illustrated in Attachment B. Complete the required information on the adhesive labels and apply them to every sample container. Obtain sample labels from the appropriate program/project source, request that they be electronically generated in house, or request them the laboratory subcontractor.

5.3.1.3 Chain-of-Custody Record

The chain-of-custody record is a multi-part form that is initiated as samples are acquired and accompanies a sample (or group of samples) as they are transferred from person to person. This form must be used as follows for any samples collected for chemical or geotechnical analysis whether the analyses are performed on site or off site:

- Retain one carbonless copy of the completed chain-of custody form in the field.
- Send one copy is sent to the PM (or designee)
- Send the original to the laboratory with the associated samples. Place the original (top, signed copy) of the chain-of custody form inside a large Ziploc®-type bag taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one chain-of custody form, send the form with the cooler containing vials for volatile organic compound (VOC) analysis or the cooler with the air bill attached. Indicate on the air bill how many coolers are included with that shipment.

An example of a chain-of-custody form is provided as Attachment C. After the samples are received at the laboratory, the sample cooler and contents are checked and any problems are noted on the enclosed chain-of custody form (any discrepancies between the sample labels and chain-of custody form and any other problems that are noted are resolved through communication between the laboratory point-of-contact and the TtNUS PM). The chain-of custody form is signed and copied. The laboratory will retain the copy, and the original becomes part of the samples' corresponding analytical data package.

5.3.1.4 Chain-of-Custody Seal

Attachment D is an example of a custody seal. The custody seal is an adhesive-backed label that is part of a chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field and sealed in coolers for transport to the laboratory. Sign and date custody seals

and affix them across the lid and body of each cooler (front and back) containing environmental samples (see SOP SA-6.1). Obtain custody seals from the laboratory (if available) or purchase them from a supplier.

5.3.1.5 ~~Geochemical Parameters Log Sheets~~

~~Complete Field Analytical Log Sheets to record geochemical and/or natural attenuation field test results.~~

5.3.2 Hydrogeological and Geotechnical Forms

5.3.2.1 ~~Groundwater Level Measurement Sheet~~

~~Complete a Groundwater Level Measurement Sheet for each round of water level measurements made at a site.~~

5.3.2.2 ~~Data Sheet for Pumping Test~~

~~During the performance of a pumping test (or an in-situ hydraulic conductivity test), a large amount of data must be recorded, often within a short time period. Use a Pumping Test Data Sheet to facilitate this task by standardizing the data collection format for the pumping well and observation wells, and allowing the time interval for collection to be established in advance.~~

5.3.2.3 ~~Packer Test Report Form~~

~~Complete a Packer Test Report Form for each well at which a packer test is conducted.~~

5.3.2.4 ~~Boring Log~~

~~Complete a Summary Log of Boring, or Boring Log for each soil boring performed to document the materials encountered, operation and driving of casing, and locations/depths of samples collected. In addition, if volatile organics are monitored on cores, samples, cuttings from the borehole, or breathing zone, (using a photoionization detector [PID] or flame ionization detector [FID]), enter these readings on the boring log at the appropriate depth. When they become available, enter the laboratory sample number, concentrations of key contaminants, or other pertinent information in the "Remarks" column. This feature allows direct comparison of contaminant concentrations with soil characteristics.~~

5.3.2.5 ~~Monitoring Well Construction Details Form~~

~~Complete a Monitoring Well Construction Details Form for every monitoring well, piezometer, or temporary well point installed. This form contains specific information on length and type of well riser pipe and screen, backfill, filter pack, annular seal and grout characteristics, and surface seal characteristics. This information is important in evaluating the performance of the monitoring well, particularly in areas where water levels show temporal variation or where there are multiple (immiscible) phases of contaminants. Depending on the type of monitoring well (in overburden or bedrock, stick-up or flush mount), different forms are used.~~

5.3.2.6 ~~Test Pit Log~~

~~When a test pit or trench is constructed for investigative or sampling purposes, a Test Pit Log must be filled out by the responsible field geologist or sampling technician.~~

5.3.2.7 ~~Miscellaneous Monitoring Well Forms~~

~~Miscellaneous monitoring well forms that may be required on a project-specific basis include the Monitoring Well Materials Certificate of Conformance and Monitoring Well Development Record. Use a Monitoring Well Materials Certificate of Conformance to document all materials utilized during each monitoring well installation. Use a Monitoring Well Development Record to document all well development activities.~~

5.3.2.8 Miscellaneous Field Forms – Quality Assurance and Checklists

Miscellaneous field forms/checklists forms that may be required on a project-specific basis include the following:

- Container Sample and Inspection Sheet – use this form when a container (drum, tank, etc.) is sampled and/or inspected.
- QA Sample Log Sheet – use this form when a QA sample such as an equipment rinsate blank, source blank, etc. is collected.
- Field Task Modification Request (FTMR) – use this form to document deviations from the project planning documents. The FOL is responsible for initiating the FTMRs. Maintain copies of all FTMRs with the on-site planning documents, and place originals in the final evidence file.
- Field Project Daily Activities Checklist and Field Project Pre-Mobilization Checklist – used these during both the planning and field effort to ensure that all necessary tasks are planned for and completed. These two forms are not requirements but are useful tools for most field work.

5.3.3 Equipment Calibration and Maintenance Form

The calibration or standardization of monitoring, measuring, or test equipment is necessary to ensure the proper operation and response of the equipment, to document the accuracy, precision, or sensitivity of the measurements, and determine if correction should be applied to the readings. Some items of equipment require frequent calibration, others infrequent. Some are calibrated by the manufacturer, others by the user.

Each instrument requiring calibration has its own Equipment Calibration Log, which documents that the manufacturer's instructions were followed for calibration of the equipment, including frequency and type of standard or calibration device. Maintain an Equipment Calibration Log for each electronic measuring device used in the field; make entries for each day the equipment is used or in accordance with manufacturer recommendations.

5.4 FIELD REPORTS

The primary means of recording on-site activities is the site logbook. Other field notebooks may also be maintained. These logbooks and notebooks (and supporting forms) contain detailed information required for data interpretation or documentation but are not easily used for tracking and reporting of progress. Furthermore, the field logbook/notebooks remain on site for extended periods of time and are thus not accessible for timely review by project management. Other reports useful for tracking and reporting the progress of field activities are described below.

5.4.1 Daily Activities Report

To provide timely oversight of on-site contractors, complete and submit Daily Activities Reports (DARs) as described below.

5.4.1.1 Description

The DAR documents the activities and progress for each day's field work. Complete this report on a daily basis whenever there are drilling, test pitting, well construction, or other related activities occurring that involve subcontractor personnel. These sheets summarize the work performed and form the basis of payment to subcontractors. The DAR form can be found on the TtNUS intranet site.

5.4.1.2 Responsibilities

It is the responsibility of the rig geologist to complete the DAR and obtain the driller's signature acknowledging that the times and quantities of material entered are correct.

5.4.1.3 Submittal and Approval

At the end of the shift, the rig geologist must submit the DAR to the FOL for review and filing. The Daily Activities Report is not a formal report and thus requires no further approval. The DARs are retained by the FOL for use in preparing the site logbook and in preparing weekly status reports for submission to the PM.

5.4.2 Weekly Status Reports

To facilitate timely review by project management, photocopies of logbook/notebook entries may be made for internal use.

In addition to those described herein, other summary reports may also be contractually required.

All TtNUS field forms can be found on the company's intranet site at http://intranet.ttnus.com under Field Log Sheets.

6.0 LISTING OF FIELD FORMS ON THE TtNUS INTRANET SITE

- ~~Boring Log~~
- Container Sample and Inspection Sheet
- Daily Activities Checklist
- Daily Activities Record
- Equipment Calibration Log
- Field Task Modification Request
- Field Analytical Log sheet - Geochemical Parameters
- ~~Groundwater Level Measurement Sheet~~
- ~~Groundwater Sample Log Sheet~~
- ~~Hydraulic Conductivity Test Data Sheet~~
- ~~Low Flow Purge Data Sheet~~
- ~~Bedrock Monitoring Well Construction (Stick Up)~~
- ~~Bedrock Monitoring Well Construction Flush Mount~~
- ~~Bedrock Monitoring Well Construction Open Hole~~
- ~~Confining Layer Monitoring Well Construction~~
- ~~Monitoring Well Development Record~~

- ~~Monitoring Well Materials Certificate of Conformance~~
- ~~Overburden Monitoring Well Construction Flush Mount~~
- ~~Overburden Monitoring Well Construction Stick Up~~
- ~~Packer Test Report Form~~
- ~~Pumping Test Data Sheet~~
- ~~QA Sample Log Sheet~~
- Soil/Sediment Sample Log Sheet
- ~~Surface Water Sample Log Sheet~~
- ~~Test Pit Log~~
- Field Project Pre-Mobilization Checklist

ATTACHMENT A
TYPICAL SITE LOGBOOK ENTRY

START TIME: _____ DATE: _____

SITE LEADER: _____

PERSONNEL:

TtNUS	DRILLER	SITE VISITORS
_____	_____	_____
_____	_____	_____
_____	_____	_____

WEATHER: Clear, 68°F, 2-5 mph wind from SE

ACTIVITIES:

1. Steam jenney and fire hoses were set up.
2. Drilling activities at well ____ resumes. Rig geologist was _____. See Geologist's Notebook, No. 1, page 29-30, for details of drilling activity. Sample No. 123-21-S4 collected; see sample logbook, page 42. Drilling activities completed at 11:50 and a 4-inch stainless steel well installed. See Geologist's Notebook, No. 1, page 31, and well construction details for well _____.
3. Drilling rig No. 2 steam-cleaned at decontamination pit. Then set up at location of well _____.
4. Well _____ drilled. Rig geologist was _____. See Geologist's Notebook, No. 2, page ____ for details of drilling activities. Sample numbers 123-22-S1, 123-22-S2, and 123-22-S3 collected; see sample logbook, pages 43, 44, and 45.
5. Well _____ was developed. Seven 55-gallon drums were filled in the flushing stage. The well was then pumped using the pitcher pump for 1 hour. At the end of the hour, water pumped from well was "sand free."
6. EPA remedial project manger arrives on site at 14:25 hours.
7. Large dump truck arrives at 14:45 and is steam-cleaned. Backhoe and dump truck set up over test pit _____.
8. Test pit _____ dug with cuttings placed in dump truck. Rig geologist was _____. See Geologist's Notebook, No. 1, page 32, for details of test pit activities. Test pit subsequently filled. No samples taken for chemical analysis. Due to shallow groundwater table, filling in of test pit ___ resulted in a very soft and wet area. A mound was developed and the area roped off.
9. Express carrier picked up samples (see Sample Logbook, pages 42 through 45) at 17:50 hours. Site activities terminated at 18:22 hours. All personnel off site, gate locked.

_____

Field Operations Leader

ATTACHMENT B
SAMPLE LABEL

Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090	**Project:** **Site:** **Location:**	
Sample No:		**Matrix:**
Date:	**Time:**	**Preserve:**
Analysis:		
Sampled by:	**Laboratory:**	

ATTACHMENT C
CHAIN-OF-CUSTODY RECORD FORM

TETRA TECH NUS, INC.

CHAIN OF CUSTODY | NUMBER 3413 | PAGE ___ OF ___

PROJECT NO: | FACILITY: | PROJECT MANAGER | PHONE NUMBER | LABORATORY NAME AND CONTACT:

SAMPLERS (SIGNATURE) | FIELD OPERATIONS LEADER | PHONE NUMBER | ADDRESS

CARRIER/WAYBILL NUMBER | CITY, STATE

STANDARD TAT ☐
RUSH TAT ☐
☐ 24 hr. ☐ 48 hr. ☐ 72 hr. ☐ 7 day ☐ 14 day

DATE YEAR	TIME	SAMPLE ID	LOCATION ID	TOP DEPTH (FT)	BOTTOM DEPTH (FT)	MATRIX (GW, SO, SW, SD, QC, ETC.)	COLLECTION METHOD GRAP (G) COMP (C)	No. OF CONTAINERS	CONTAINER TYPE PLASTIC (P) or GLASS (G)	PRESERVATIVE USED	TYPE OF ANALYSIS	COMMENTS

	DATE	TIME		DATE	TIME
1. RELINQUISHED BY			1. RECEIVED BY		
2. RELINQUISHED BY			2. RECEIVED BY		
3. RELINQUISHED BY			3. RECEIVED BY		

COMMENTS

DISTRIBUTION: WHITE (ACCOMPANIES SAMPLE) YELLOW (FIELD COPY) PINK (FILE COPY)

FORM NO. TtNUS-001 4/02R

ATTACHMENT D
CHAIN-OF-CUSTODY SEAL

CUSTODY SEAL	**CUSTODY SEAL**
Date	**Date**
Signature	**Signature**

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ATTACHMENTS

A INVESTIGATION-DERIVED WASTE LABEL 15

1.0 PURPOSE

Decontamination is the process of removing and/or neutralizing site contaminants that have contacted and/or accumulated on equipment. The purpose of this Standard Operating Procedure (SOP) is to protect site personnel, the general public, and the environment while preserving or maintaining sample integrity. It is further intended through this procedure to describe the steps necessary for proper decontamination of drilling equipment, earth-moving equipment , chemical sampling equipment and field operation and analytical equipment.

2.0 SCOPE AND APPLICABILITY

This procedure applies to all equipment used to provide access to/acquire environmental samples that may have become contaminated through direct contact with contaminated media including air, water, and soil. This equipment includes drilling and heavy equipment and chemical sampling and field analytical equipment. Where technologically and economically feasible, single-use sealed disposable equipment will be employed to minimize the potential for cross-contamination. This SOP also provides general reference information on the control of contaminated materials.

Decontamination methods and equipment requirements may differ from one project to another. General equipment items are specified in Section 6.0, but project-specific equipment must be obtained to address the project-specific decontamination procedures presented in Section 7.0 and applicable subsections.

3.0 GLOSSARY

Alconox/Liquinox - A brand of phosphate-free laboratory-grade detergent.

Decontamination Solution - A solution selected/identified in the Health and Safety Plan or Project-Specific Quality Assurance Plan. The solution is selected and employed as directed by the project chemist/health and safety professional.

Deionized Water (DI) - Tap water that has been treated by passing through a standard deionizing resin column. This water may also pass through additional filtering media to attain various levels of analyte-free status. The DI water should meet College of American Pathologists (CAP) and National Committee for Clinical Laboratory Standards (NCCLS) specifications for reagent-grade Type I water.

Potable Water - Tap water from any municipal water treatment system. Use of an untreated potable water supply is not an acceptable substitute for tap water.

Pressure Washing - Process employing a high-pressure pump and nozzle configuration to create a high-pressure spray of potable water. High-pressure spray is employed to remove solids from equipment.

Solvent – A liquid in which solid chemicals or other liquids are dissolved. The solvent of choice is pesticide-grade isopropanol. Use of other solvents (methanol, acetone, or hexane) may be required for particular projects or for a particular purpose (e.g., removal of concentrated waste) and must be justified in the project planning documents. For example, it may be necessary to use hexane when analyzing for trace levels of pesticides, PCBs, or fuels. In addition, because many of these solvents are not miscible in water, the equipment should be air dried prior to use. Solvents should not be used on PVC equipment or well construction materials.

Steam Pressure Washing - A cleaning method employing a high-pressure spray of heated potable water to remove various organic/inorganic chemicals from equipment.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - Responsible for ensuring that all field activities are conducted in accordance with approved project plan(s) requirements.

Decontamination Personnel - Individuals assigned the task of decontamination. It is the responsibility of these individuals to understand the use and application of the decontamination process and solutions as well as the monitoring of that process to ensure that it is working properly. This is accomplished through visual evaluation, monitoring instrument scanning of decontaminated items, and/or through the collection of rinsate blanks to verify contaminant removal.

Field Operations Leader (FOL) - Responsible for the implementation of project-specific planning documents. This includes on-site verification that all field activities are performed in compliance with approved SOPs or as otherwise dictated by the approved project plan(s). The FOL is also responsible for the completion and accuracy of all field documentation.

Site Safety Officer (SSO) - Exercises shared responsibility with the FOL concerning decontamination effectiveness. All equipment arriving on site (as part of the equipment inspection), leaving the site, and moving between locations is required to go through a decontamination evaluation. This is accomplished through visual examination and/or instrument screening to determine the effectiveness of the decontamination process. Improper or incomplete decontamination is sufficient to restrict equipment from entering the site, exiting the site, or moving to a new location on the site until the objectives are successfully completed.

General personnel qualifications for decontamination activities include the following:

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

5.0 HEALTH AND SAFETY

In addition to the health and safety issues and reminders specified in subsections of this SOP, the following considerations and requirements must be observed as SOPs for field equipment decontamination activities:

- If any solvents or hazardous chemicals (e.g., isopropyl alcohol) are to be used in equipment decontamination activities, the FOL must first obtain the manufacturer's/supplier's Material Safety Data Sheet (MSDS) and assure that it is reviewed by all users (prior to its use), added to the site Hazardous Chemical Inventory, and maintained on site as part of the project Hazard Communication Program.
- Review and observe specific health and safety requirements (e.g., personal protective equipment [PPE]) specified in the project-specific health and safety plan for this activity.

6.0 EQUIPMENT LIST

- ~~Wood for decontamination pad construction, when applicable (see Section 7.1).~~

- ~~Tools for constructing decontamination pad frame, when applicable (see Section 7.1).~~
- ~~Visqueen sheeting or comparable material to cover decontamination pad frame, when applicable~~ (see Section 7.1).
- Wash/drying racks for auger flights and drill/drive rods, when applicable (see Section 7.2).
- PPE as specified in the project health and safety plan.
- Soap and water for washing and rinsing.
- Deionized water for final rinsing.
- Solvents (e.g., pesticide-grade isopropanol) for rinsing (see applicable portions of Section 7.2).
- Tubs, buckets, etc. for containerizing rinse water (see applicable portions of Section 7.2).
- Sample bottles for collecting rinsate blanks (see Section 7.2).
- Calibrated photoionization detector (PID) or flame ionization detector (FID) to monitor decontaminated equipment for organic vapors generated through the existence of residual contamination or the presence of decontamination solvent remaining after the piece was rinsed.
- Aluminum foil or clear clean plastic bag for covering cleaned equipment (see applicable portions of Section 7.2).
- Paper towels or cloths for wiping.
- Brushes, scrapers, or other hand tools useful for removing solid materials from equipment.
- Clear plastic wrap for covering or wrapping large decontaminated equipment items (see Section 7.2.2).
- Drum-moving equipment for moving filled waste drums (optional) (see Section 7.3).
- Drum labels for waste drums (see Attachment A).

7.0 PROCEDURES

The process of decontamination is accomplished through the removal of contaminants, neutralization of contaminants, or isolation of contaminants. To accomplish this activity, preparation is required including site preparation, equipment selection, and evaluation of the decontamination requirements and processes. Site contaminant types, concentrations, and media types are primary drivers in the selection of the types of decontamination and where it will be conducted. For purposes of this SOP, discussion is limited to decontamination procedures for general environmental investigations.

Decontamination processes will be performed at the location(s) specified in project-specific planning documents. Typical decontamination locations include the following:

- Temporary decontamination pads/facilities
- Sample locations
- Centralized decontamination pad/facilities

- Combination of some or all of the above

The following discussion includes general considerations for the decontamination process. Specific construction and implementation procedures will be as specified in the project-specific planning documents and/or may be as dictated by site-specific conditions as long as the intent of the requirements in the planning documents is met. This intent is to contain any residual fluids and solids generated through the decontamination process.

7.1 ~~Decontamination Pad Design/Construction Considerations~~

7.1.1 ~~Temporary Decontamination Pads~~

~~Temporary decontamination pads may be constructed at satellite locations within the site area in support of temporary work areas. These structures are generally constructed to support the decontamination of heavy equipment such as drill rigs and earth-moving equipment but can be employed for smaller articles.~~

~~The purpose of the decontamination pad is to contain wash waters and potentially contaminated soil generated during decontamination procedures. Therefore, construction of these pads should take into account the following considerations:~~

- Site location – The decontamination site selected should be far enough from the work site to maximize decontamination effectiveness while minimizing travel distance. The location of the decontamination site shall be selected to provide, in the judgment of the FOL or FOL designee, compliance with as many of the following characteristics as practicable:

  - Well removed from pedestrian/vehicle thoroughfares.

  - Avoidance of areas where control/custody cannot be maintained.

  - Avoidance of areas where potential releases of contaminated media or decontamination fluids may be compounded through access to storm water transport systems, streams, or other potentially sensitive areas.

  - Avoidance of potentially contaminated areas.

  - Avoidance of areas too close to the ongoing operation, where cross-contamination may occur.

  The selected decontamination site should include the following, where possible:

  - Areas where potable water and electricity are provided.

> ***Safety Reminder***
>
> When utilizing electrical power sources, either hard-wired or portable-generated sources, ensure that:
>
> - All power is routed through a Ground Fault Circuit Interrupter (GFCI).
>
> - All power cords are in good condition (no physical damage), rated for the intended energy load, and designated for outdoor use.
>
> In situations where accomplishing these elements is not possible, it will be necessary to implement a site electrical grounding program.

- Areas where support activities such as removing decontamination waters soil and sediment are possible without entering an active exclusion zone.

- Areas that offer sufficient size to carry out the specific decontamination sequence.

- ~~Decontamination pad (decon pad) – The decon pad shall be constructed to meet the following characteristics:~~

  - ~~Size – The size of the pad should be sufficient to accept the equipment to be decontaminated as well as permitting free movement around the equipment by the personnel conducting the decontamination. The size should permit these movements utilizing pressure/steam washer wands and hoses and minimizing splash due to work in close quarters.~~

  - ~~Slope – An adequate slope will be constructed to permit the collection of water and potentially contaminated soil within a trough or sump constructed at one end. The collection point for wash waters should be of adequate distance that the decontamination workers do not have to walk through the wash waters while completing their tasks. Because the pad will be sloped, place a light coating of sand over the plastic to minimize potential slips and falls. See the text about liners below.~~

  - ~~Sidewalls – The sidewalls shall be at least 6 inches in height (or as high as possible if 6 inches is not achievable) to provide adequate containment for wash waters and soil. If splash represents a potential problem, splash guards should be constructed to control overspray. Sidewalls may be constructed of wood, inflatables, sand bags, etc. to permit containment. Splash guards are typically wood frames with Visqueen coverings to control overspray.~~

  - ~~Liner – Depending on the types of equipment and decontamination method to be used, the liner should be of sufficient thickness to provide a puncture-resistant barrier between the decontamination operation and the unprotected environment. Care should be taken to examine the surface area prior to placing the liner to remove sharp articles (sticks, stones, debris) that could puncture the liner. Liners are intended to form an impermeable barrier. The thickness may vary from a minimum recommended thickness of 10 mil to 30 mil. The desired thickness may be achieved through layering materials of lighter construction. It should be noted that various materials (rubber, polyethylene sheeting) become slippery when wet. To minimize this potential hazard associated with a sloped liner, a light coating of sand shall be applied to provide traction, as necessary.~~

  - ~~Wash/drying racks – Auger flights, drill/drive rods, and similar equipment require racks positioned off of the ground to permit these articles to be washed, drained, and dried while secured from falling during this process.~~

For decontamination of direct-push technology (DPT) equipment, the pad may be as simple as a mortar tub containing buckets of soapy water for washing and an empty bucket to capture rinse waters. Decontamination may be conducted at the rear of the rig to permit rapid tool exchange.

- Maintenance – Maintain the decontamination area by:

  - Periodically clearing the work area of standing water, soil, and debris, and coiling hoses to aid in eliminating slip, trip, and fall hazards. In addition, these articles will reduce potential backsplash and cross-contamination.

- Regularly changing the decontamination fluids to ensure proper cleaning and prevent cross-contamination.

- PPE – Periodically evaluate the condition of, and maintain the decontamination equipment, including regular cleaning of face shields and safety glasses. This is critical to ensuring the safety of decontamination personnel and the integrity of the decontamination process, and it will ensure that equipment is functioning properly.

7.1.2 Decontamination Activities at Drill Rigs/DPT Units

During subsurface sampling activities including drilling and DPT activities, decontamination of drive rods, Macro Core Samplers, split spoons, etc. is typically conducted at an area adjacent to the operation. Decontamination is generally accomplished using a soap/water wash and rinse utilizing buckets and brushes. This area requires sufficient preparation to accomplish the decontamination objectives.

Buckets shall be placed within mortar tubs or similar secondary containment tubs to prevent splash and spills from reaching unprotected environmental media. Drying racks shall be employed as directed for temporary pads to permit parts to dry and be evaluated prior to use/reuse. Methodology regarding this activity is provided in Section 7.2.

7.1.3 Decontamination Activities at Remote Sample Locations

When sampling at remote locations, sampling equipment such as trowels and pumps/tubing should be evacuated of potentially contaminated media to the extent possible. This equipment should be wrapped in plastic for transport to the temporary/centralized decontamination location for final cleaning and disposition. Flushing and cleaning of single-use equipment such as disposable trowels, tubing, and surgeon's gloves may allow disposal of this equipment after visible soil and water remnants have been removed.

7.2 Equipment Decontamination Procedures

The following represents procedures to be employed for the decontamination of equipment that may have contacted and/or accumulated contamination through site investigation activities.

7.2.1 ~~Monitoring Well Sampling Equipment~~

7.2.1.1 ~~Groundwater sampling equipment – This includes pumps inserted into monitoring wells such as bladder pumps, Whale pumps, and Redi-Flo pumps and reusable bailers, etc.~~

1. ~~Evacuate to the extent possible, any purge water within the pump/bailer.~~

2. ~~Scrub using soap and water and/or steam clean the outside of the pump/bailer and, if applicable, the pump tubing.~~

3. ~~Insert the pump and tubing/bailer into a clean container of soapy water. Pump/run a sufficient amount of soapy water through the pump/bailer to flush out any residual well water. After the pump is flushed, circulate soapy water through the pump to ensure that the internal components are thoroughly flushed.~~

4. ~~Remove the pump and tubing/bailer from the container~~

5. ~~Rinse external pump components using tap water.~~

6. ~~Insert the pump and tubing/bailer into a clean container of tap water. Pump/run a sufficient amount of tap water through the pump/bailer to evacuate all of the soapy water (until clear).~~

> ***CAUTION***
> Do not rinse PE, PVC, and associated tubing with solvents –
> Use the procedures defined in the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 7 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

7. ~~If groundwater contains or is suspected to contain oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse the equipment to be cleaned with pesticide-grade isopropanol~~.

8. ~~Pass deionized water through the hose to flush out the tap water and solvent residue as applicable.~~

9. ~~Drain residual deionized water to the extent possible.~~

10. ~~Allow components of the equipment to air dry.~~

11. ~~For bladder pumps, disassemble the pump and wash the internal components with soap and water, then rinse with tap water, isopropanol, and deionized water and allow to dry. After the parts are dry, conduct a visual inspection and a monitoring instrument scan to ensure that potential contaminants and all decontamination solvent have been removed. Collect a rinsate blank in accordance with the project-specific planning documents to ensure that the decontamination process is functioning as intended. The typical frequency of collection for rinsate blanks is 1 per 20 field samples. In addition, wipe samples or field tests such as UV light may be used.~~

12. ~~Wrap pump/bailer in aluminum foil or a clear clean plastic bag for storage.~~

> ***SAFETY REMINDER***
> Remember when handling powered equipment to disconnect the power source and render the equipment to a zero energy state (both potential and kinetic) before opening valves, disconnecting lines, etc.

7.2.1.2 ~~Electronic Water Level Indicators/Sounders/Tapes~~

~~During water level measurements, rinsing the extracted tape and probe with deionized water and wiping the surface of the extracted tape between locations is acceptable. However, periodic full decontamination should be conducted as follows:~~

1. ~~Wash with soap and water~~
2. ~~Rinse with tap water~~
3. ~~Rinse with deionized water~~

> ***NOTE***
> In situations where oil, grease, free product, other hard to remove materials are encountered, probes and exposed tapes should be washed in hot soapy water. If probes or tapes cannot be satisfactorily decontaminated (they are still stained, discolored, etc.), they should be removed from service.

7.2.1.3 Miscellaneous Equipment

Miscellaneous equipment including analytical equipment (water quality testing equipment) shall be cleaned per manufacturers' instructions. This generally includes wiping the sensor housing and rinsing with tap and deionized water.

Coolers/shipping containers employed to ship samples are received from the laboratory in a variety of conditions including marginal to extremely poor. Coolers shall be evaluated prior to use for the following:

- Structural integrity – Coolers missing handles or having breaks in the outer housing should be removed and not used. Notify the laboratory that the risk of shipping samples in the cooler(s) provided is too great and request a replacement unit.

- Cleanliness – As per protocol, only volatile organic samples are accompanied by a trip blank. If a cooler's cleanliness is in question (visibly dirty/stained) or if there are noticeable odors, the cooler should be decontaminated prior to use as follows:

  1. Wash with soap and water
  2. Rinse with tap water
  3. Dry

If these measures fail to clean the cooler to an acceptable level, remove the unit from use as a shipping container and ask the cooler provider (e.g., the analytical laboratory) to provide a replacement unit.

7.2.2 Downhole Drilling Equipment

This includes any portion of the drill rig that is over the borehole, including auger flights, drill stems, rods, and associated tooling that would extend over the borehole. The following procedure is to be employed prior to initiating the drilling/sampling activity, then between locations:

> ***CAUTION***
> Exercise care when using scrapers to remove soil and debris from downhole drilling equipment. Inadvertent slips of scrapers have resulted in cuts, scrapes, and injured knuckles, so use scrapers carefully when removing soil from these items.

1. Remove loose soil using shovels, scrapers, etc.

2. Through a combination of scrubbing using soap and water and/or steam cleaning or pressure washing, remove visible dirt/soil from the equipment being decontaminated.

> ***CAUTION***
> In Step 3, do not rinse PE, PVC, and associated tubing with solvents. The appropriate procedures should be defined within the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 4 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

3. Rinse the equipment with tap water, where applicable (steam cleaning and pressure washing incorporate rinsing as part of the process).

4. If the equipment has directly or indirectly contacted contaminated sample media and is known or suspected of being contaminated with oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse equipment with pesticide-grade isopropanol

5. To the extent possible, allow components to air dry.

6. If the decontaminated equipment is to be used immediately after decontamination, screen it with a calibrated photoionization detector (PID)/flame ionization detector (FID) to ensure that all contaminants and possible decontamination solvents (if they were used) have been adequately removed.

7. Wrap or cover equipment in clear plastic until it is time to be used.

SAFETY REMINDER

Even when equipment is disconnected from power sources, dangers such as the following may persist:

Falls - An auger flight standing on its end may fall and injure someone. Secure all loose articles to prevent heavy articles from falling onto people or equipment.

Burns - Steam cleaner water is heated to more than 212 °F and exhibits thermal energy that can cause burns. Prevent contact of skin with hot water or surfaces.

High water pressure - Pressure washer discharge can have 2,000 to 4,000 psi of water pressure. Water under this amount of pressure can rupture skin and other human tissues. Water at 4,000 psi exiting a 0° tip can be dangerous because of its relatively high cutting power. The exit velocity and cutting power of the water are reduced when exiting a 40° fan tip, but damage to soft tissues is still possible.

In general, follow the rules below to avoid injury, equipment damage, or incomplete decontamination:

1. Read the operating manual and follow the manufacturers' recommended safety practices before operating pressure washers and steam cleaners.

2. Never point the pressure washer or steam cleaner at another person or use to clean your boots or other parts of your body. Water lacerations and burns may appear to be minor at first but can be life threatening. Do not attempt to hold small parts in your hand while washing them with high-temperature or high-pressure water.

3. Always wear PPE as specified in the HASP such as:

   - Hard hat, safety glasses, splash shield, impermeable apron or splash suit, and hearing protection. Remember that excessive noise is a hazard when operating gas-powered engines and electrically driven pressure washers. PPE will be identified in your project specific planning documents.

4. Inspect each device before use. An inspection checklist will be provided in the project-specific planning documents. If it is a rented device, safety measures are typically provided by the vendor. In all cases, if you are not familiar with the operation of a pressure washer/steam cleaner, do not operate it until you obtain and thoroughly review operating instructions and recommended safety practices.

5. Do not modify equipment unless the manufacturer has approved the modifications.

7.2.3 Soil/Sediment Sampling Equipment

This section applies to soil sampling equipment including but not limited to hand augers, stainless steel trowels/spoons, bowls, dredges, scoops, split spoons, Macro Core samplers, etc.

1. Remove all loose soil from the equipment through manual means.
2. Through a combination of scrubbing using soap and water and/or steam cleaning or pressure washing, remove visible dirt/soil from the equipment.
3. Rinse the equipment with tap water.

> ***CAUTION***
> Do not rinse PE, PVC, and associated tubing with solvents. The appropriate procedures should be defined within the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 4 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

4. If the equipment is contaminated or suspected to be contaminated with oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse the equipment with pesticide-grade isopropanol.
5. Rinse the equipment with deionized water.
6. To the extent possible, allow components to air dry.
7. If the equipment is to be used immediately after decontamination, screen it with a calibrated PID/FID to ensure that all solvents (if they were used) and trace contaminants have been adequately removed.
8. After the equipment has dried, wrap it in aluminum foil for storage until use.

Dredges employed in sediment sampling are typically decontaminated as follows:

- Remove the sediment sample from the sampling device
- If sufficient associated surface water is available at the sampling site, place the dredge in the water and flush to remove visible sediment.
- Extract the dredge and wash it in soap and water per the project-specific planning documents.

> ***CAUTION***
> When handling dredges, the primary safety concern is trapping fingers or extremities in the larger dredge samplers within the jaws or pinch points of the mechanical jaws. Keep hands, fingers, and extremities away from these pinch and compression points. Either handle the device by the rope or preferably lock the jaws in place to control the potential for closing during maintenance and/or cleaning.

7.3 Contact Waste/Materials

During the course of field investigations, disposable/single-use equipment becomes contaminated. These items include tubing, trowels, PPE (gloves, overboots, splash suits, etc.), and broken sample containers.

With the exception of the broken glass, single-use articles should be cleaned (washed and rinsed) of visible materials and disposed as normal refuse. The exception to this rule is that extremely soiled materials that cannot be cleaned shall be containerized for disposal in accordance with applicable federal, state, and local regulations.

7.3.1 Investigation-Derived Wastes - Decontamination Wash Waters and Sediments

> ***NOTE***
> Requirements for waste storage may differ from one facility to the next. Facility-specific directions for waste storage areas will be provided in project-specific documents, or separate direction will be provided by the Project Manager.

1. Assume that all investigation-derived waste (IDW) generated from decontamination activities contains the hazardous chemicals associated with the site unless there are analytical or other data to the contrary. Waste solution volumes could vary from a few gallons to several hundred gallons in cases where large equipment required cleaning.

2. Where possible, use filtering systems to extend the use of water within a closed system wash unit to recycle water and to reduce possible waste amounts.

> ***NOTE***
> Containerized waste rinse solutions are best stored in 55-gallon drums (or equivalent containers) that can be sealed until ultimate disposal at an approved facility.

3. Label waste storage containers appropriately labeled (see Attachment A).

4. Ensure that the IDW storage area is configured to meet the following specifications to permit access to the containers and to conduct spill/leak monitoring, sampling, and extraction when the disposal route is determined:

   - Enclose areas accessible by the general public using construction fencing and signs.
   - Stored materials in 55-gallon drums on pallets with four (or fewer) drums per pallet.
   - Maintain the retaining bolt and label on the outside of storage containers where readily visible.
   - Provide at least 4 feet of room between each row of pallets to allow access to containers for sampling, drum removal, and spill response.
   - As directed in project-specific planning documents, maintain an IDW Inventory List and provide the list to the site Point of Contact at the termination of each shift.
   - Maintain spill response equipment at the IDW storage area in case it is required for immediate access.

- Where possible, use equipment for moving containers. Where not possible, obtain help to manipulate containers.

CAUTION

Each container of water can weigh up to 490 pounds. Each 55-gallon drum of wet soil can weigh more than 750 pounds. Fill drums and temporary containers to 80 percent capacity to minimize spill and handling difficulties. Use drum carts to move filled drums.

See safe lifting techniques provided in Section 4.4 of the Tetra Tech NUS, Inc. Health and Safety Guidance Manual.

When placing drums, keep your fingers out of pinch and smash points such as between the drums. In some cases such as well development and/or purge water, you can place the drums to be filled on the pallet and transport materials in smaller easier to handle containers.

7.4 Decontamination Evaluation

Upon decontamination of equipment, determine the effectiveness of the decontamination process in the following manner:

- Visual evaluation – A visual evaluation will be conducted to ensure the removal of particulate matter. This shall be done to ensure that the washing/rinsing process is working as intended.

- Instrument Screening – A properly calibrated PID/FID should be used to evaluate the presence of site contaminants and solvents used in the cleaning process. The air intake of the instrument shall be passed over the article to be evaluated. Avoid placing the instrument probe into residual waters. A PID/FID reading greater than the daily established background level requires a repeat of the decontamination process, followed by rescreening with the PID/FID. This sequence must be repeated until no instrument readings greater than the daily established background level are observed. It should be noted that the instrument scan is only viable if the contaminants are detectable within the instrument's capabilities.

NOTE

When required by project-specific planning documents, collection of rinsate blanks (see next step) shall be completed without exception unless approval to not collect these samples is obtained from the Project Manager.

- Collection of Rinsate Blanks – It is recommended that rinsate samples be collected to:

  - Evaluate the decontamination procedure representing different equipment applications (pumps versus drilling equipment) and different decontamination applications.

  - Single-use disposable equipment – The number of samples should represent different types of equipment as well as different lot numbers of single-use articles.

  - The collection and the frequency of collection of rinsate samples are as follows unless specified differently in the project-specific planning documents:

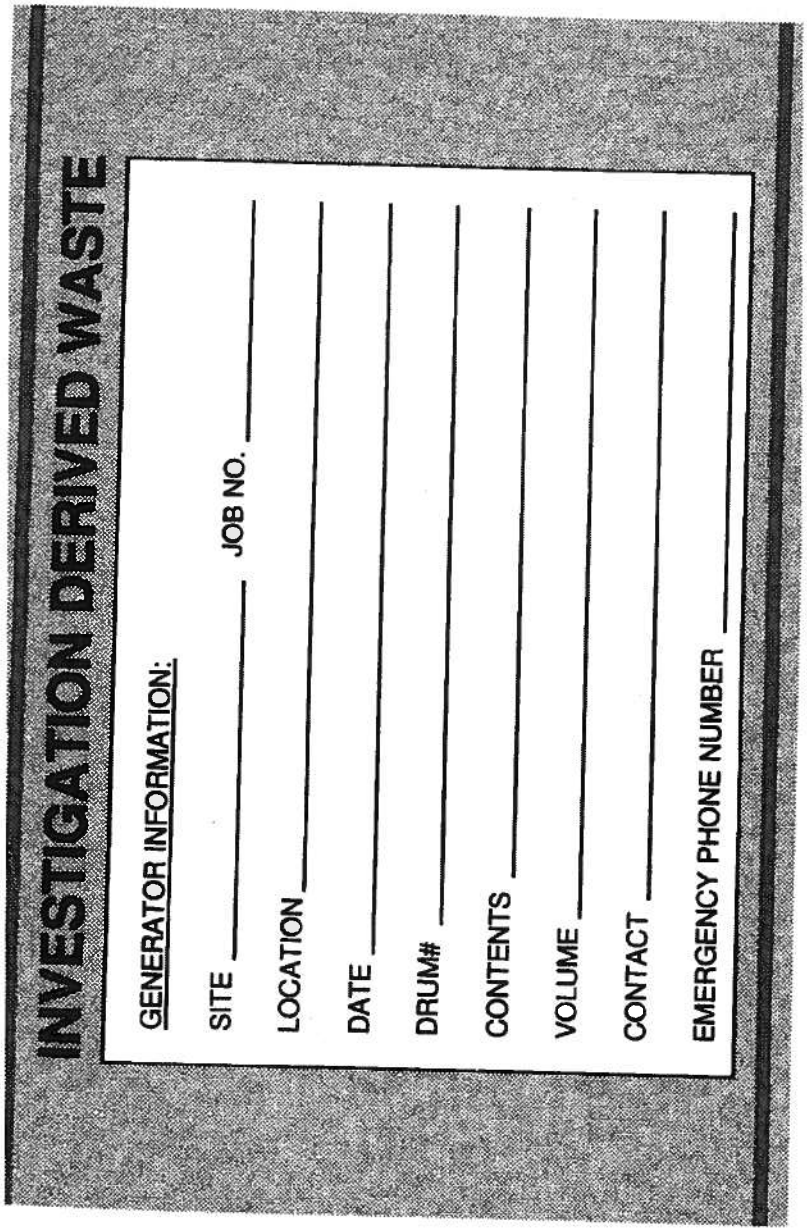
    - Per decontamination method
    - Per disposable article/batch number of disposable articles

NOTE

It is recommended that an initial rinsate sample be collected early in the project to ensure that the decontamination process is functioning properly and to avoid using a contaminated batch of single-use articles. It is recommended that a follow-up sample be collected later during the execution of the project to ensure that those conditions do not change.

Rinsate samples collection may be driven by types of and/or levels of contaminant. Difficult to remove contaminants, oils/greases, some PAHs/PCBs, etc. may also support the collection of additional rinsates due to the obvious challenges to the decontamination process. This is a field consideration to be determined by the FOL.

Attachment A
iDW Label



APPENDIX B

Field Documentation Forms

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.

QA SAMPLE LOG SHEET

Project Site Name: Derecktor Shipyard Offshore
Project Number: 112G02747
Sample Location:
QA Sample Type:

Sample ID Number:
Sampled By:
C.O.C. Number:

[ ] Trip Blank
[ ] Source Water Blank (Field Blank)
[ ] Rinsate Blank
[ ] Other Blank

SAMPLING DATA:

Date:
Time:
Method: Direct Pour

WATER SOURCE:

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

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

Product Name: Reagent Grade Water (DIUF)
Supplier:
Manufacturer:
Order Number:
Lot Number:
Expiration Date:

RINSATE INFORMATION (If Applicable):

Media Type:
Equipment Used:
Equipment Type:
[ ] Dedicated
[ ] Reusable
[ ] Disposable

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected
VOC	HCl	3 X 40 mL vials	YES / NO
GRO	HCl	2 X 40 mL vials	YES / NO
DRO		2 x 1 liter amber glass jars	YES / NO
SVOC		2 x 1 liter amber glass jars	YES / NO
Pesticides, PCBs		2 x 1 liter amber glass jars	YES / NO
Metals (filtered/unfiltered)	HNO3	1 x 2 500 mL poly bottle	YES / NO

OBSERVATIONS / NOTES:

Signature(s):

TETRA TECH NUS, INC.	SITE ENTRY LOG

Site Name: DERECKTOR SHIPYARD OFFSHORE Date:_____.

Location: NAVSTA NEWPORT Project Number: 112G02747

NAME	REPRESENTING	TIME IN (HOURS)	TIME OUT (HOURS)	INITIALS

TtNUS Form 0002

SITE-SPECIFIC TRAINING DOCUMENTATION

My signature below indicates that I am aware of the potential hazardous nature of performing remedial investigation activities at DERECKTOR SHIPYARD OFFSHORE and that I have received site-specific training which included the elements presented below:

- Names of designated personnel and alternates responsible for site safety and health
- Safety, health, and other hazards present at the sites
- Use of personal protective equipment
- Safe use of engineering controls and equipment
- Medical surveillance requirements
- Signs and symptoms of overexposure
- Contents of the Health and Safety Plan
- Emergency response procedures (evacuation and assembly points)
- Initial response procedures
- Review of the contents of relevant Material Safety Data Sheets
- Review of the use of Safe Work Permits

I have been given the opportunity to ask questions and all of my questions have been answered to my satisfaction. I further state, that the dates of my training (introductory, refresher, and supervisory, as applicable) and my medical surveillance requirements are accurate and correct to the best of my knowledge.

Name (Printed and Signature)	Site-Specific Training Date	40-Hour Training (Date)	8-Hour Refresher Training (Date)	8-Hour Supervisory Training (Date)	Medical Exam

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Project Site Name: Derecktor Shipyard Offshore
Project No.: 112G02747

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

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

Type of Sample:
[X] 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:

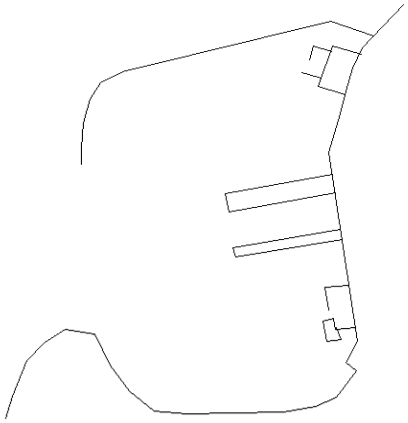
Date:	Time	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Method:				
Monitor Readings (Range in ppm):				

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

MAP:



Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

APPENDIX C

Notes from Planning Meetings

ANALYTICAL RESULTS FOR CONTAMINANTS OF CONCERN IN SEDIMENT
SITE 19 - DERECKTOR SHIPYARD
NS NEWPORT
NEWPORT, RHODE ISLAND

Pre FS Data

SAMPLE_ID		DSY-1	DSY-2	DSY-3	DSY-4	DSY-5	DSY-6	DSY-7	DSY-8	DSY-9	DSY-10	DSY-11	DSY-12	DSY-13	DSY-14	DSY-15	DSY-16	DSY-17	DSY-18	DSY-19	DSY-20	DSY-21	DSY-22	DSY-23	DSY-24
LOCATION_ID		DSY-01	DSY-02	DSY-03	DSY-04	DSY-05	DSY-06	DSY-07	DSY-08	DSY-09	DSY-10	DSY-11	DSY-12	DSY-13	DSY-14	DSY-15	DSY-16	DSY-17	DSY-18	DSY-19	DSY-20	DSY-21	DSY-22	DSY-23	DSY-24
TOP_DEPTH		0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
BOTTOM_DEPTH		0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
SAMPLE_DATE		11/03/93	11/03/93	11/03/93	11/03/93	11/03/93	11/03/93	11/03/93	11/03/93	11/03/93	11/03/93	11/03/93	11/03/93	06/13/94	06/13/94	06/13/94	06/13/94	06/13/94	06/13/94	06/13/94	06/13/94	06/13/94	06/13/94	06/13/94	06/13/94
SACODE																									
QC_TYPE	**CALCULATED PRG**																								
BENZO(A)PYRENE	539	163.83	3320	4710	431.43	401.78	488.3	301.91	480.98	49.13	38.47	206.33	248.81	43.73	26.07	245.08	11.76	310.59	1190	496.21	880.34	109.63	182.008	239.46	54.52
HIGH MOLECULAR WEIGHT PAHS	13903	4013.39	63994.3	72956	5531.95	4525.58	5336.06	4197.97	5855.67	616.58	535.87	4991.43	4493.61	872.66	435.52	3129.21	237.28	3417.68	12859.78	9211.87	20405.51	1923.4	3270.568	4408.67	1162.21
TOTAL PCBs (total Aroclor or Sum congners x 2	1060	67.5772	209.0887	733.3312	194.56	105.4064	132.0812	73.3573	148.3893	28.1474	11.728	658.1613	176.009	22.3161	22.9766	54.835	9.4	243.7826	292.7514	216.559	366.9521	92.3076	178.2045	150.1339	25.869
LEAD	168	35.393	180.764	201.061	51.348	43.296	48.579	31.699	50.604	14.856	22.132	46.082	45.99	35.2	28.9404	42.025	31.5291	32.9018	60.2299	57.8157	76.9119	41.8304	52.5276	54.1921	39.1522
TRIBUTYLTIN	228 sv*	32	372	255	155	202	125	61	131	72	64	139	116	138	94	110	83	81	167	220	197	109	244	220	120

Post FS Data (2004)

SAMPLE_ID		No Sample	DSY-SD-02-	DSY-SD-03-	DSY-SD-04-	DSY-SD-05-	DSY-SD-06-	No Sample	DSY-SD-08-	DSY-SD-09-	No Sample	DSY-SD-11-	No Sample	No Sample	No Sample	No Sample	No Sample	No Sample	No Sample	No Sample	DSY-SD-20-	No Sample	No Sample	No Sample	No Sample
LOCATION_ID		DSY-01	DSY-02	DSY-03	DSY-04	DSY-05	DSY-06	DSY-07	DSY-08	DSY-09	DSY-10	DSY-11	DSY-12	DSY-13	DSY-14	DSY-15	DSY-16	DSY-17	DSY-18	DSY-19	DSY-20	DSY-21	DSY-22	DSY-23	DSY-24
TOP_DEPTH			0	0	0	0	0		0	0		0									0				
BOTTOM_DEPTH			0.5	0.5	0.5	0.5	0.5		0.5	0.5		0.5									0.5				
SAMPLE_DATE			08/25/04	08/26/04	08/26/04	08/26/04	08/25/04		08/26/04	08/26/04		08/26/04									08/26/04				
SACODE	**CALCULATED PRG**		AVG OF DUP			AVG OF DUP																			
QC_TYPE			FD			FD																			
BENZO(A)PYRENE	539		180	1000	150 J	104	370		190 J	46 J		320 J									230 J				
HIGH MOLECULAR WEIGHT	13903		1521	9760	1644	813.5	2945		1525	378.5		2776									2048				
TOTAL PCBS (SUM OF PCB	1060		110	349	198	104	498		179	23.8		385									202				
LEAD	168		49.5	114 J	50.4 J	46.4	49.9 J		46.5 J	29.1 J		65.6 J									47.9 J				
TRIBUTYLTIN	228 sv*		NA	NA	NA	NA	NA		NA	NA		NA									NA				

Key:

COC for HH Risk (shellfish ingestion)
COC for bedded sed
COC for Suspended Sed
*Screening Value only
Detected, below PRG

GREY BACKGROUND - DETECTED; U - NOT DETECTED; UJ - DETECTION LIMIT APPROXIMATE;
J - QUANTITATION LIMIT APPROXIMATE; R - REJECTED; NA - NOT ANALYZED

Pre FS Data

				DSY 27			DSY 28					DSY 29			DSY 30			DSY 31					DSY 34		
SAMPLE_ID		DSY-25-SUR	DSY-26-SUR	DSY-27-SUR	DSY-V9-MID	DSY-V9-BOT	DSY-28-SUR	DSY-28-MID	DSY-28-BOT	DSY-V4-MID	DSY-V4-BOT	DSY-29-SUR	DSY-29-MID	DSY-29-BOT	DSY-30-SUR	DSY-30-MID	DSY-30-BOT	DSY-31-SUR	DSY-31-MID	DSY-31-BOT	DSY-32-SUR	DSY-33-SUR	DSY-34-SUR	DSY-34-MID	DSY-34-BOT
LOCATION_ID		DSY-25	DSY-26	DSY-27	DSY-27	DSY-27	DSY-28	DSY-28	DSY-28	DSY-28	DSY-28	DSY-29	DSY-29	DSY-29	DSY-30	DSY-30	DSY-30	DSY-31	DSY-31	DSY-31	DSY-32	DSY-33	DSY-34	DSY-34	DSY-34
TOP_DEPTH		0	0	0	0.3	1.3	0	0.8	2.5	3.4	4.3	0	0.5	1.4	0	0.6	2.2	0	1.5	3.3	0	0	0	0.7	2.3
BOTTOM_DEPTH		0.5	0.5	0.5	0.7	1.5	0.5	1.1	2.8	3.8	4.6	0.5	0.9	1.8	0.5	0.9	2.6	0.5	1.8	3.6	0.5	0.5	0.5	1	2.6
SAMPLE_DATE		09/28/95	10/19/95	10/12/95	01/11/96	01/11/96	10/19/95	11/16/95	11/16/95	01/11/96	01/11/96	10/19/95	11/16/95	11/16/95	10/12/95	11/16/95	11/16/95	10/12/95	11/16/95	11/16/95	09/28/95	09/28/95	10/12/95	11/16/95	11/16/95
SACODE																									
QC_TYPE	**CALCULATED PRG**											avg of dups												avg of dups	
BENZO(A)PYRENE	539	395.8064	434.2685	923.9997	2380	235.6422	377.4684	697.5214	4130	0	6.2333	2790	1550.0409	9.529	811.8407	137.7264	2.415	420.8157	147.0902	2.0163 J	494.6919	67.8487	147.6253	21.0301	2.415
HIGH MOLECULAR WEIGHT PAHS	13903	2343.5877	3031.3942	5526.7383	21497.2799	2075.2226	2210.2406	4116.0924	34320.7401	19.2034	64.6812	23345.1	11413.4112	82.1203	5594.3696	846.8089	9.0553	2408.6018	743.8501	13.3997	2897.783	391.2466	860.3718	126.7275	7.3587
TOTAL PCBs (total Aroclor or Sum congners x 2	1060	93.6121	98.2409	3310	1380	91.254	133.7077	430.457	203.8593	16.7737	7.4336	741.1858	81.4807	2.854	315.2884	12.9621	5.3362	220.9876	146.6901	3.1993	200.912	39.9302	64.5813	20.9327	4.9628
LEAD	168	35.9	40.4	150.7	182	15.5	77.7	148.4	192.6	16.1	12.8	179.2	87.1	19	80	34.9	21.7	81	52.7	29.9	124.8	40	47.6	23.8	25.7
TRIBUTYLTIN	228 sv*	0.73 J	2.27	8.52	7.27	0.5	65.36	12.87	140.54	0.5	0.5	63.53	0.5	0.5	6.86	0.5	0.5	228	0.5	0.5	4.26	0.5	4.13	0.5	0.5

Post FS Data (2004)

SAMPLE_ID		No Sample	DSY-SD-26-	DSY-SD-27-	No Sample	No Sample	DSY-SD-28-	No Sample	No Sample	No Sample	No Sample	DSY-SD-29-	No Sample	No Sample	No Sample	No Sample	No Sample	DSY-SD-31-	No Sample	No Sample	DSY-SD-32-	No Sample	No Sample	No Sample	No Sample
LOCATION_ID		DSY-25	DSY-26	DSY-27	DSY-27	DSY-27	DSY-28	DSY-28	DSY-28	DSY-28	DSY-28	DSY-29	DSY-29	DSY-29	DSY-30	DSY-30	DSY-30	DSY-31	DSY-31	DSY-31	DSY-32	DSY-33	DSY-34	DSY-34	DSY-34
TOP_DEPTH			0	0			0					0						0			0				
BOTTOM_DEPTH			0.5	0.5			0.5					0.5						0.5			0.5				
SAMPLE_DATE			08/26/04	08/26/04			08/25/04					08/26/04						08/26/04			08/26/04				
SACODE																									
QC_TYPE	**CALCULATED PRG**																								
BENZO(A)PYRENE	539		89 J	530			220 J					1200						120			250 J				
HIGH MOLECULAR WEIGHT	13903		816	3796			1656					10970						1055			2334				
TOTAL PCBS (SUM OF PCB	1060		80.1	2960			79.8					410						130			102				
LEAD	168		27 J	138 J			51.3 J					113 J						47.9 J			43.6 J				
TRIBUTYLTIN	228 sv*		NA	NA			NA					NA						NA			NA				

Key:

COC for HH Risk (shellfish ingestion)
COC for bedded sed
COC for Suspended Sed
*Screening Value only
Detected, below PRG

GREY BACKGROUND - DETECTED; U - NOT DETECTED; UJ - DETECTION LIMIT APPROXIMATE;
J - QUANTITATION LIMIT APPROXIMATE; R - REJECTED; NA - NOT ANALYZED

Pre FS Data

			DSY 36								NEW STATIONS ADDED 2004						REFERENCE AREA DATA JAMESTOWN					
SAMPLE_ID		DSY-35-SUR	DSY-36-SUR	DSY-36-MID	DSY-36-BOT	DSY-37-SUR	DSY-38-SUR	DSY-39-SUR	DSY-40-SUR	DSY-41-SUR	No Sample	No Sample	No Sample	No Sample	No Sample	No Sample	JPC-1-SUR-AVG	JPC-2-SUR-AVG	No Sample	No Sample	No Sample	No Sample
LOCATION_ID		DSY-35	DSY-36	DSY-36	DSY-36	DSY-37	DSY-38	DSY-39	DSY-40	DSY-41	DSY-SD-101	DSY-SD-101	DSY-SD-103	DSY-SD-103	DSY-SD-104	DSY-SD-104	DSY-JPC-01	DSY-JPC-02	DSY-SD-JPC03	DSY-SD-CC02	DSY-SD-CH01	DSY-SD-CH02
TOP_DEPTH		0	0	1.3	3	0	0	0	0	0							0	0				
BOTTOM_DEPTH		0.5	0.5	1.6	3.3	0.5	0.5	0.5	0.5	0.5							0.5	0.5				
SAMPLE_DATE		10/12/95	09/28/95	11/16/95	11/16/95	09/28/95	09/28/95	10/12/95	10/12/95	10/12/95							10/19/95	10/27/95				
SACODE																						
QC_TYPE	**CALCULATED PRG**				avg of dups												avg of dups	avg of dups				
BENZO(A)PYRENE	539	4.1449 J	319.4442	432.4069	10.3104	163.8001	119.8243	142.9988	316.6461	18.5537							62.8722	NA				
HIGH MOLECULAR WEIGHT PAHS	13903	25.1363	1703.2945	2129.0725	53.1417	1115.872	843.8954	830.6092	3016.6921	143.5151							365.1931	NA				
TOTAL PCBs (total Aroclor or Sum congners x 2	1060	6.6972	113.2718	393.4579	4.1091	99.9017	59.3308	58.1578	84.1312	13.7532							18.9124	NA				
LEAD	168	14	78.8	112.5	27.2	56.9	62.2	54	42.1	17							29	49.6				
TRIBUTYLTIN	228 sv*	0.5	5.01	0.5	0.5	0.5	0.5	0.5	4.21	0.5							0.28 J	0.41 J				

Post FS Data (2004)

SAMPLE_ID		No Sample	No Sample	No Sample	No Sample	No Sample	No Sample	No Sample	No Sample	No Sample	DSY-SD-101-	DSY-SD-101-	DSY-SD-103-	DSY-SD-103-	DSY-SD-104-	DSY-SD-104-	DSY-SD-	No Sample	DSY-SD-JPC03-	DSY-SD-CC02-	DSY-SD-CH01-	DSY-SD-CH02-
LOCATION_ID		DSY-35	DSY-36	DSY-36	DSY-36	DSY-37	DSY-38	DSY-39	DSY-40	DSY-41	DSY-SD-101		DSY-SD-103		DSY-SD-104		DSY-JPC-01	DSY-JPC-02	DSY-SD-JPC03	DSY-SD-CC02	DSY-SD-CH01	DSY-SD-CH02
TOP_DEPTH											0	0.5	0	0.5	0	0.5	0		0	0	0	0
BOTTOM_DEPTH											0.5	1	0.5	1	0.5	1	0.5		0.5	0.5	0.5	0.5
SAMPLE_DATE											08/25/04	08/25/04	08/25/04	08/25/04	08/25/04	08/25/04	08/26/04		08/26/04	08/26/04	08/26/04	08/26/04
SACODE																						
QC_TYPE	**CALCULATED PRG**																					
BENZO(A)PYRENE	539										270	420	360	1400 *	140	320 J	46 J		26.5 J	42 J	470 J	60 J
HIGH MOLECULAR WEIGHT	13903										2541	4743	4223	23070	1316	2966	413		232.55	384	3775	497.5
TOTAL PCBS (SUM OF PCB	1060										307	410	764	749	144	203	16.4		0.8125	4.32	86.2	11.1
LEAD	168										40.7 J	49 J	71.6 J	168 J	58.4 J	75.6 J	11.3 J		4.35 J	8.4 J	65.7 J	21.3 J
TRIBUTYLTIN	228 sv*										NA	NA	NA	NA	NA	NA	NA		NA	NA	NA	NA

Key:

COC for HH Risk (shellfish ingestion)
COC for bedded sed
COC for Suspended Sed
*Screening Value only
Detected, below PRG

GREY BACKGROUND - DETECTED; U - NOT DETECTED; UJ - DETECTION LIMIT APPROXIMATE;
J - QUANTITATION LIMIT APPROXIMATE; R - REJECTED; NA - NOT ANALYZED

DRAFT

Meeting Notes
Discussion of Path Forward
Derecktor Shipyard NAVSTA Site 19
NAVSTA Newport, Newport Rhode Island

Attachments:

1. Presentation handout
2. Table of Historic Data

Participating:

Dave Barclift, NAVFAC LANT
Pamela Crump, RIDEM
Ken Finkelstein, NOAA
Todd Finlayson, Gannett Fleming (via phone)
Arun Gavaskar, NAVFAC LANT
Gary Jablonski, RIDEM
Mike Horton, Tetra Tech
Nina Johnson, NAVFAC Mid Atlantic
Winoma Johnson, NAVFAC Mid Atlantic
Kymberlee Keckler, USEPA
Greg Kemp, Gannett Fleming
Cornelia Mueller, NAVSTA
Ken Munney, US Fish and Wildlife
Brian Olsen, USEPA
Stephen Parker, Tetra Tech

The meeting convened at 10:00 AM

W. Johnson opened the meeting and thanked everyone for coming. She stated that the purpose of the meeting was to review existing data, determine additional data needs, determine a path forward, and discuss a schedule to achieve a ROD and remedy.

Brian Olson stated that this is a necessary discussion to have and he expects that all parties will speak freely and not be afraid that they will be held to what is said in open conversation. This needs to be a flow of ideas, so keep an open mind as the discussions progress.

Most of the early part of the discussion was led by S. Parker, who presented the first portion of the presentation (Attachment 1) and the historic data (Attachment 2).

Regarding Slide 14, there was discussion about the PRGs that are being followed at this site. G. Jablonski indicated that RIDEM had provided a letter dated (January 2?) 2008 that had asked a question on Step 9 of the PRG development process. He did not believe that the Navy had responded to that letter.

Navy action – find the 2008 RIDEM letter and respond.
Follow up: Letter was incorrectly dated January 2, 2008, was actually received on January 6, 2009. Records show that a phone call to discuss the letter and the proposal and PRG development was planned and the need to hold that call was noted in the May 2009 RPM Meeting notes. However, the call was not held because no proposed dates were agreed on. The Navy finally provided a written response on October 29, 2009 which explained the PRG development and included a sample calculation. No additional comments were received. In response to this action item, Navy provided RIDEM January, 2, 2009 letter and Navy October 29, 2009 responses on November 8, 2010.

Regarding Slide 14, It was clarified that the four "Limiting COCs" that were selected for the site and have "Recommended PRGS" were selected from a list of ten COCs for which "Baseline PRGs" were developed originally by SAIC. K. Munney, K. Finkelstein, and K. Keckler stated that they would prefer to see all contaminants analyzed for in any future investigation. K.Keckler stated that she would prefer to see extent of contamination measured below the three- foot depth proposed.

Regarding Slide 24, Brian Olson stated that the time required to go from data gaps investigation to ROD should not be as long as are stated. He felt that a ROD was possible for this site in 2011. N. Johnson and W. Johnson indicated that this would be a difficult feat to achieve, given the FFA review cycles, and stated that they could provide breakdown of times

Regarding Slide 30, K. Finkelstein stated that based on 2004 data (and unless future sampling reveals much higher concentrations, say under the piers), dredging does not appear to be justified and may unnecessarily disturb the existing equilibrium. B. Olson stated that he did not believe the data were sufficient to make that determination.

There was a side discussion regarding the possibilities of dredging. D. Barclift stated that additional data needed to be collected and evaluated before a decision could be made as to whether dredging was needed. K. Munney also stated that the group needs to just define the sampling plan and see what that data shows: there is not enough data at this time to determine if dredging was necessary. It was agreed that a further investigation was appropriate at this point.

A. Gavaskar stated that the Navy was trying to model its approach on another Region 1 site that had recently been dredged, namely, New Bedford Harbor. In the New Bedford ROD, "although the ecological risk assessment pointed to a 1-ppm sediment PCB threshold for protection of marine organisms, achieving this TCL (threshold concentration level) was believed to cause more harm than good". More achievable thresholds of 10 ppm in the Upper Harbor and 50 ppm in the Lower Harbor were selected in the ROD to determine areas that needed to be dredged. Subsequently, EPA is looking for more cost effective alternatives for "moderately contaminated" areas and has tested a capping remedy in 20 acres of sediment with concentrations as high as 94 ppm. B. Olson said that Bedford was different because it was a big site, whereas Derecktor is a smaller site.

A. Gavaskar mentioned that dredging had not been very successful at low-contamination sites. K. Munney said that the Army had recently dredged a low-contamination site at

Natick. A. Gavaskar mentioned that his understanding was that the Natick site had been dredged to a cove-wide average of 1 ppm. The Navy would like to use a similar surface-weighted area concentration (SWAC) approach that had been used at Natick and many other sites. K. Keckler said that EPA' preference would be to dredge all the sediment that was above the PRG. B. Olson said that EPA would be receptive to some approach akin to multi-increment sampling, whereby the cove is divided into parcels and a composite sample within each parcel is used to determine cleanup.

G. Kemp asked if there were on shore inputs that could be providing additional contaminants. It was stated that there are storm drain outfalls that do flow in this area, and "scuppers" which drain overland runoff to the near shore areas. G. Kemp indicated that this could potentially represent a different source. There was also some discussion about the contaminant deposition from the berthed ships , particularly copper. Navy agreed to include copper as an analyte in areas which have not been previously sampled.

Regarding Slide 31, there was open conversation about sampling approaches. B. Olson requested that the group consider multi-increment (MI) sampling. The Navy agreed to consider the idea. It was noted that area averaging of data may be appropriate or composite sampling may be useful in some data uses. Other ideas were discussed openly, including doing more cores in the hatched areas, less in unhatched areas, fewer in the outer portions of the cove, etc. There was general agreement that locations which did not have PRG exceedances would not need to be sampled again. G. Kemp asked if the data from the Corps of Engineers collected in 1987 had been considered (this data had been considered in selection of stations for the BERA, but it is not in the electronic database provided in the handouts).

N. Johnson asked the group to decide how much of each type of sampling (surface samples versus deeper cores) should be done, as the Navy had limited funds.

Also regarding slide 31, K. Keckler stated that EPA had provided a letter to the Navy with additional samples requested for the PDI (no date cited). **Navy Action :** The Navy agreed to find this letter. **EPA Action:** K. Keckler stated she would resend anyway. **Follow-up:** Navy did not find this item in their records, and EPA resent it on 10/29/10.

Regarding slide 32, B. Olson stated he would like to see some information provided on the effects of propeller wash against sediments. It was acknowledged that there were some papers done on this issue, and they would be sought for evaluation of the possibility of resuspension and scouring.

Regarding slides 37-40, the possibility of a DQO meeting was discussed, and it was determined that it would have to be at a later time. Some dates were discussed, the following was agreed on:

The Navy would deliver a revised sampling plan for discussion on November 5, 2010
The EPA and RIDEM would provide comments for discussion on November 15, 2010
The parties would would have a conference call on November 22 to discuss in detail.

The goals of the meeting were reviewed, and it was agreed that a path forward could begin, with the data gaps investigation.

Summary of Actions:

1. **All parties to consider something similar to Multi Increment Sampling for sediments**
2. **Navy to find RIDEM Letter 1/2/08 and determine if a response was sent (done, see above)**
3. **EPA to resend requested additional sediment samples (done, see above)**
4. **Navy to look for information prop washing.**
5. **Navy to submit a revised sampling plan by November 5.**
6. **EPA and RIDEM to review sampling plan and provide comments by November 15.**
7. **Navy to schedule a conference call for November 22, to discuss sampling plan and comments.**

Meeting adjourned at 1:45 PM.

Conceptual Sampling Plan

Data Gaps Investigation, Site 19: Former Derecktor Shipyard Marine Sediment

Newport RI

Data Quality Objectives

Data will be collected to support a decision as to whether a remedial action is required in portions of the study area that are affected by site related contaminants. Data needs to be adequate to support the selection of the action that would be required. These actions could include, but may not be limited to: removal (dredging), cover (thin layer cap), monitored natural recovery, and/or institutional controls.

It has been determined that contaminants present in the sediment do or did pose unacceptable risk to human and ecological receptors (SAIC 1997). Based on these calculated risks, "Baseline Preliminary Remediation Goals" (BPRGs) were developed based on a HQ of 1.0, as published in SAIC, 1998. A subset of "recommended PRGs" was developed to best represent site related contaminants as a first step in risk management. Those that were not selected for calculation of recommended PRGs were:

- **o,p'-DDE –**
  - Estimated to pose risk to amphipods from suspended sediment modeled through an elutriate test, however baseline PRG was only exceeded in historical data at one sample station, not considered a contaminant that originated at the site.
- **Arsenic –**
  - Estimated to pose risk to human health through ingestion of shellfish and lobster, however most arsenic in shellfish is present in a non toxic organic form (SAIC 1998, pp 30-31).
  - Estimated to pose risk to avian predators, however data did not show arsenic above the BPRG level for this receptor.
  - Estimated to pose risk to amphipods from suspended sediment modeled through an elutriate test, however because locations of high risk did not match the locations of high concentrations, this PRG was not recommended for implementation.
- **Copper-**
  - Estimated to pose risk to avian predators, however site data only showed two exceedances above the BPRG level for this receptor.
  - Estimated to pose risk to amphipods from suspended sediment modeled through an elutriate test, however because locations of high risk did not match the locations of high concentrations, this PRG was not recommended for implementation.
- **Silver -**
  - Estimated to pose risk to avian predators, however data did not show silver above the BPRG level for this receptor.
- **Zinc –**
  - Estimated to pose risk to avian predators, and exceeded BPRGs at most stations. However, exceedances were slight, and limiting process showed that other, more toxic COCs were co-located with zinc that would be addressed.

Four COCs were recommended to be retained for use to direct remedial actions. These are:

Benzo(a)pyrene
Total HMW PAHs
Total PCBs
Lead

Secondary constituents of concern have been identified recently and include:

TBT – highly toxic, but not found to be a COC for the site
Asbestos – known to have been released from under Pier 1
Copper – possibly deposited from the 10 year berthing of the Ex Saratoga, and Ex Forrestal at Pier 1.

Approach for Data Collection:

To support the decision as to whether to take an action as stated above, The following approach is proposed:

Establish five decision units throughout the study area as shown on the attached sketch. The decision units were developed based on location, confining site features, source areas, and previously collected data:

DU1 – Area under and north of Pier 2. Mostly composed of area not previously sampled, but included in a potential action area associated with DSY 27.

DU2 – Area between piers 1 and 2. Anticipated to have taken heavy ship traffic, and still trafficked by NOAA ocean buoy tenders. Includes former location of DSY 27 where PCBs were previously found in sediment exceeding PRGs

DU3 – Area of pier 1 where carriers were berthed, area under the carrier hulls and where dry docks previously were berthed and used for ship maintenance by Derecktor. Most portions not previously sampled except for metals in 1987 by corps of engineers. This DU Includes sediment under this active section of the pier.

DU4 – location of DSY 28, PAHs exceed PRGs to a depth of 3 feet below sediment surface. Former location of storm drain north of Building 42.

DU5 – location around stations DSY 29 and DSY 3. Former location of the Greenport Ferry and location where a large storm drain outfall discharges from Building 234 area. Also downgradient of the former setup shop (Building 234). PAHs and lead previously exceeded PRGs in sediment.

Each decision unit may be broken down into subunits that are comprised of squares 200x200 feet. Each larger Decision Unit will be made up of between 9 and 15 of these subunits. The size of the subunit was selected based on the size of the area to be addressed.

Chemical data will be collected at one foot intervals in one position within each of the subunits to a total depth of 2 feet through coring. The one foot interval is selected because that is the effective vertical precision of most dredging equipment. This may become important if dredging is found to be necessary within subunits or Decision Units. The vertical limit of two feet is selected because at most stations cored (with the exception of DSY 28) clean conditions were previously found in the 1-2 foot sample interval.

Four cores within each Decision Unit will be continued to four feet below sediment surface, and sampled at one foot intervals. The four foot depth limit for the subset of cores was selected based on a) the previous finding that sediments in the one core at DSY 28 that was previously advanced to this depth did not exceed PRGs below three feet, and b) by EPA request. The location of these four cores within each Decision Unit is not determined at this time, and will be selected based on presence of depositional sediment found during field efforts.

Decision Points

1. The chemical data from all the subunits taken collectively within each interval will be used to determine whether an action is required for the larger Decision Unit at that interval.

2. If a hot spot is defined within the Decision Unit, then that hot spot may be addressed individually.

3. If the sediment exceeds PRGs at depth but does not exceed PRGs in the surface interval, physical data will be used to determine if that sediment is at risk of being exposed under expected current conditions.

Chemical Data Collection

In each subunit, one core will be collected to a depth of two feet below sediment surface. These cores will be sampled at depth intervals of 0-1 foot and 1-2 feet, as stated above.

Reference attached figure for proposed station locations.

Chemical analysis shall be limited to the four Recommended COCs at all stations (Sum HMW PAHs, benzo(a)pyrene, total PCBs, and total lead). Secondary constituents of interest will be added as follows:

- Copper will be added at stations not previously sampled (under Piers, under carrier berthing locations). These include five stations at DU1, nine stations at DU3, and six stations at DU4.

- Asbestos analysis will be added to the surface intervals at 6 stations under Pier 1.

- By previous agreement TBT will be included at all sample stations.

The location of SD22 which showed an exceedance of the TBT screening value in 1993 will have surface grabs conducted around it to determine presence of COCs in surface sediment, by regulatory request.

Physical Data Collection

A multibeam sonar survey will be conducted within an area approximately 200 feet north of Pier 2, and between Pier 1 south to the CCRF surface discharge outfall (small peninsula to the south of the site). This survey will be tied to the survey conducted in 2007 and mapped as one area. Data will be used to identify depositional areas, scour areas, and overall changes in depth over time.

The hydrographic survey conducted by URI in 1996 will be augmented by deployed ADCP profilers during a 15 day period. Deployments at six stations near and around piers will assist in determination of strength of fluctuating and short term currents at the sediment surface.

Sediment stability testing is proposed for the same six stations. Stablity testing is proposed to be performed by ERDC Coastal and Hydraulics Laboratory, Vicksburg MS. The method to be employed will be a cohesive sediment erosion flume, proposed and planned for use at Gould Island (IR Site 17) during the same period of time. This data will provide information as to the current velocity required to move sediments from different depths within the sediment strata

Radioisotope analysis of cores will be conducted at six stations where the flume testing is being done. Analysis will include lead 210 which has a steady decay rate and a half life of 22 years, as well as a relatively steady deposition rate that should not be impacted by the presence of lead contaminants from site discharge. The lead 210 isotope will be measured every few centimeters down the core. Radioisotope analysis will be used to determine accumulation rate of sediment within each decision unit.

References:

SAIC 1998, Preliminary Remediation Goals for Derecktor Shipyard, Coddington Cove Prepared under Contract to Tetra Tech NUS, for US Navy Northern Division. Final. November 1998.

SAIC and Univeristy of Rhode Island, Ecological Risk Assessment for Derecktor Shipyard, Coddington Cove. Prepared under Contract to Tetra Tech NUS, for US Navy Northern Division. Final. May 1997.

Tetra Tech NUS, Inc. 1998, Marine Human Health Risk Assessment, Offshore Areas of the Former Robert E. Derecktor Shipyard, NETC Newport September, Final, 1998

Kincaid, et. al. 1996. Hydrographic Survey for the Offshore Ecological Risk Assessment at Derecktor Shipyard / Coddington Cove, Naval Education and Training Center, Newport RI. June.

Tetra Tech 2008. Summary of TBT at Site 19, Derecktor Shipyard, Naval Station Newport, Newport RI. Revised November 7, 2008

NFESC, 1998. Deep Draft Berthing Facility Site Plan, Naval Facilities Engineering Service Center, East Coast Detachment. Washington DC. July.

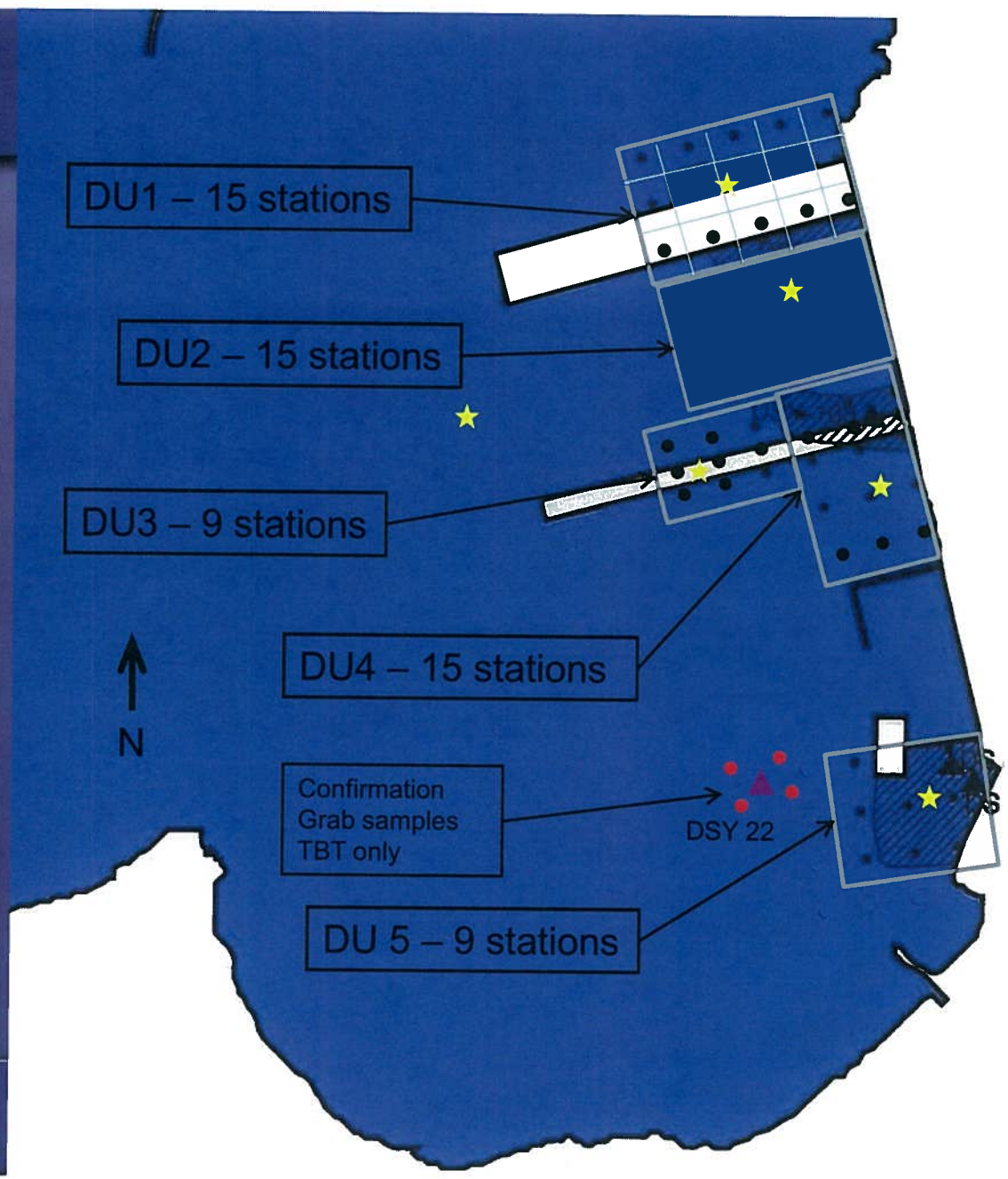
Conceptual Sampling Plan

- Establish five Decision Units. Each unit can be broken into subunits of 200 foot squares (subunits are on DU 1 only)
- Collect one core at each station to two feet. Samples 0-1 foot, 1-2 feet
- Conduct four cores at each DU to 4 feet max, analyze them in one foot intervals
- Conduct four confirmation grab samples around the former location DSY22 due to a previous hit of TBT.
- Conduct geophysical sampling at one station within each DU, and one outside the area

● Grab sample

★ Geophysical sample station

• Dots are illustrative only



SITE 19 - FORMER DERECKTOR SHIPYARD

PLANNING MEETING,

OCTOBER, 2010

INTRODUCTION

- Part 1
  - Site Orientation and Existing Data
- Part 2
  - Site Path Forward – Proposal from the Navy
  - Schedule to Achieve ROD and Remedy in Place
- Part 3 –
  - Discuss Additional Data Needs

TETRA TECH

pier 1
pier 2
NAVAL COMPLEX NEWPORT, R.I. Co

1977 Prior to Lease

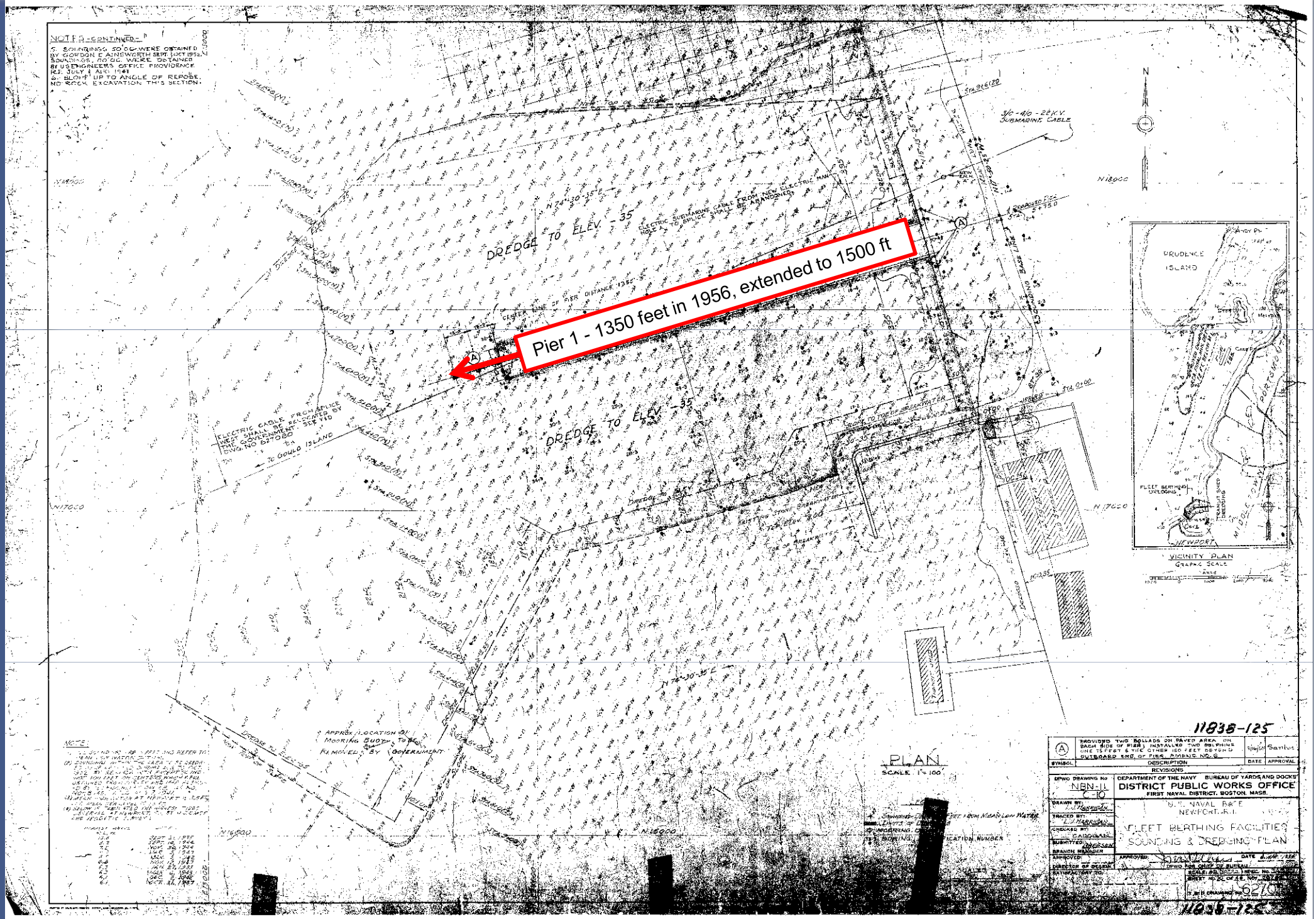
Derecktor Lease Area 1988

TETRA TECH

Coddington Cove

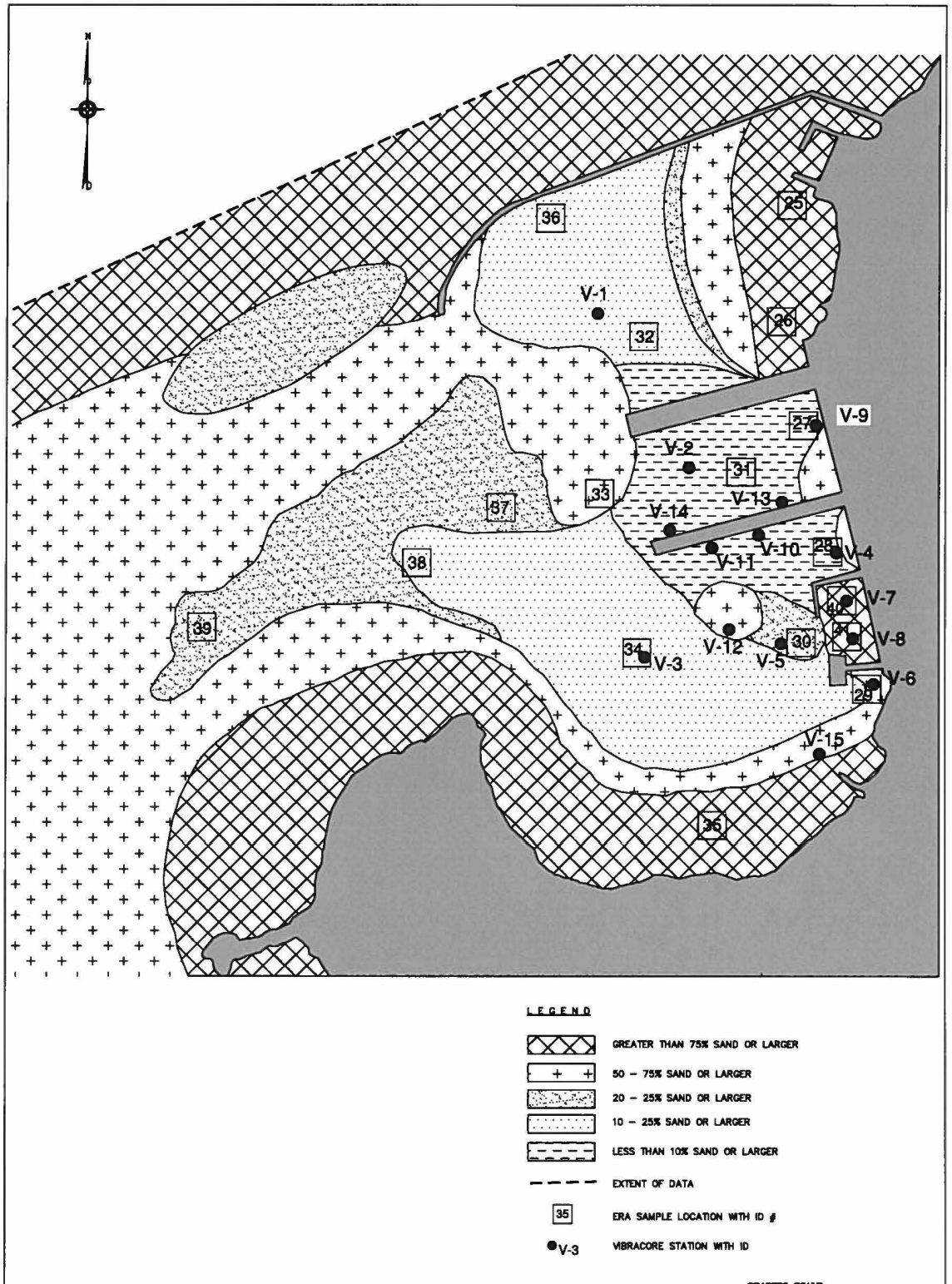
- 1956 - Portions of the cove were planned to be dredged in to “elevation –35 feet, relative to 0.0 MLLW” which would be 35-39 feet deep.
- Piers 1 and 2 Constructed in the 1950s. Area under the piers does not appear to have been dredged.
- Sediment Bathymetry Mapped in 1996 by URI
- Recent soundings in 2007 show depths between Pier 1 and Pier 2 to be 33’ to 39’

TETRA TECH

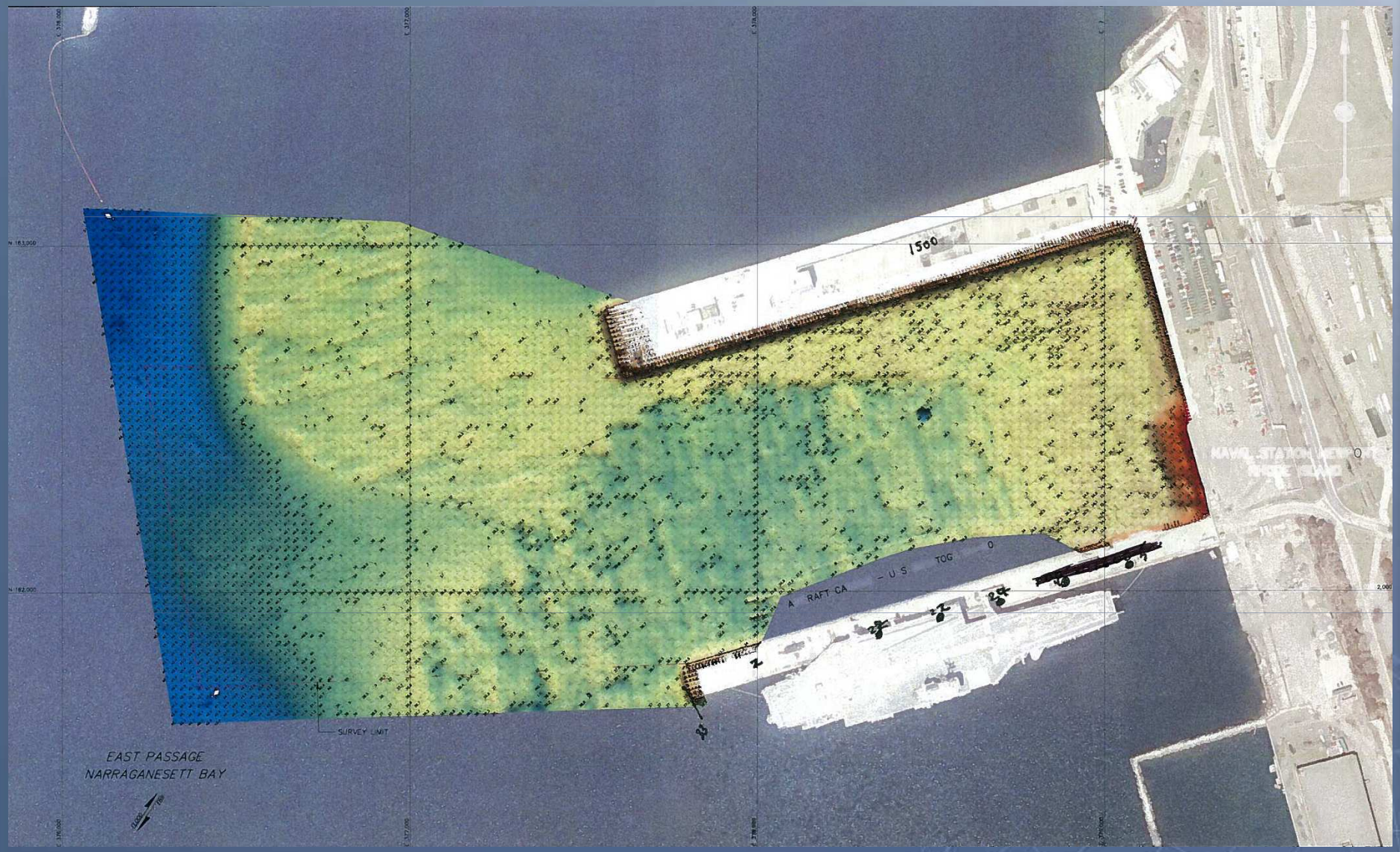

Pier 1 - 1350 feet in 1956, extended to 1500 ft
DREDGE TO ELEV -35
DREDGE TO ELEV -35
3/c-4/c-22K.V. SUBMARINE CABLE
ELECTRIC CABLE PROFILE WILL SHALL BE RELOCATED BY GOVERNMENT SEE DWG NO 827080
TO GOULD ISLAND
APPROX LOCATION OF MOORING BUOY TO BE REMOVED BY GOVERNMENT
PLAN
SCALE 1" = 100'
VICINITY PLAN
GRAPHIC SCALE
PRUDENCE ISLAND
NEWPORT
PORTSMOUTH
MIDDLE GRAVEL
FLEET BERTHING DREDGING
11835-125
PROVIDED TWO BOLLARDS ON PAVED AREA ON EACH SIDE OF PIER INSTALLED TWO DOLPHINS ONE 75 FEET & ONE OTHER 150 FEET BEYOND OUTBOARD END OF PIER AMENDS NO. 6
DESCRIPTION
REVISIONS
DEPARTMENT OF THE NAVY BUREAU OF YARDS AND DOCKS
DISTRICT PUBLIC WORKS OFFICE
FIRST NAVAL DISTRICT, BOSTON, MASS.
NBN-11
C-10
U.S. NAVAL BASE NEWPORT, R.I.
FLEET BERTHING FACILITIES
SOUNDING & DREDGING PLAN
DRAWN BY
TRACED BY
CHECKED BY
SUBMITTED
BRANCH MANAGER
APPROVED
DIRECTOR OF DESIGN
SATISFACTORY TO
DATE
SCALE
SHEET NO. 1 OF 4
827080
11835-125

Grain size data interpreted by URI in 1996.

V-1 is the location where deposition was measured at 1 cm/yr.



Multibeam Hydrography (OSI Inc. 2007)



TETRA TECH

Summary of Events

- Sediment Evaluation by URI, 1993
- Preliminary Assessment by ENSR, 1994
- On Shore SASE by Brown and Root, 1997
- Baseline Ecological Risk Assessment by SAIC and URI, 1997 (Sidebar?)
- Marine Sediment PRGs Developed by SAIC, 1998
- FS - Tetra Tech, 1999
- Sediment sampling & analysis 2004
- FS Revision 1 – Tetra Tech, Draft – March 2007, Draft Final – February 2008

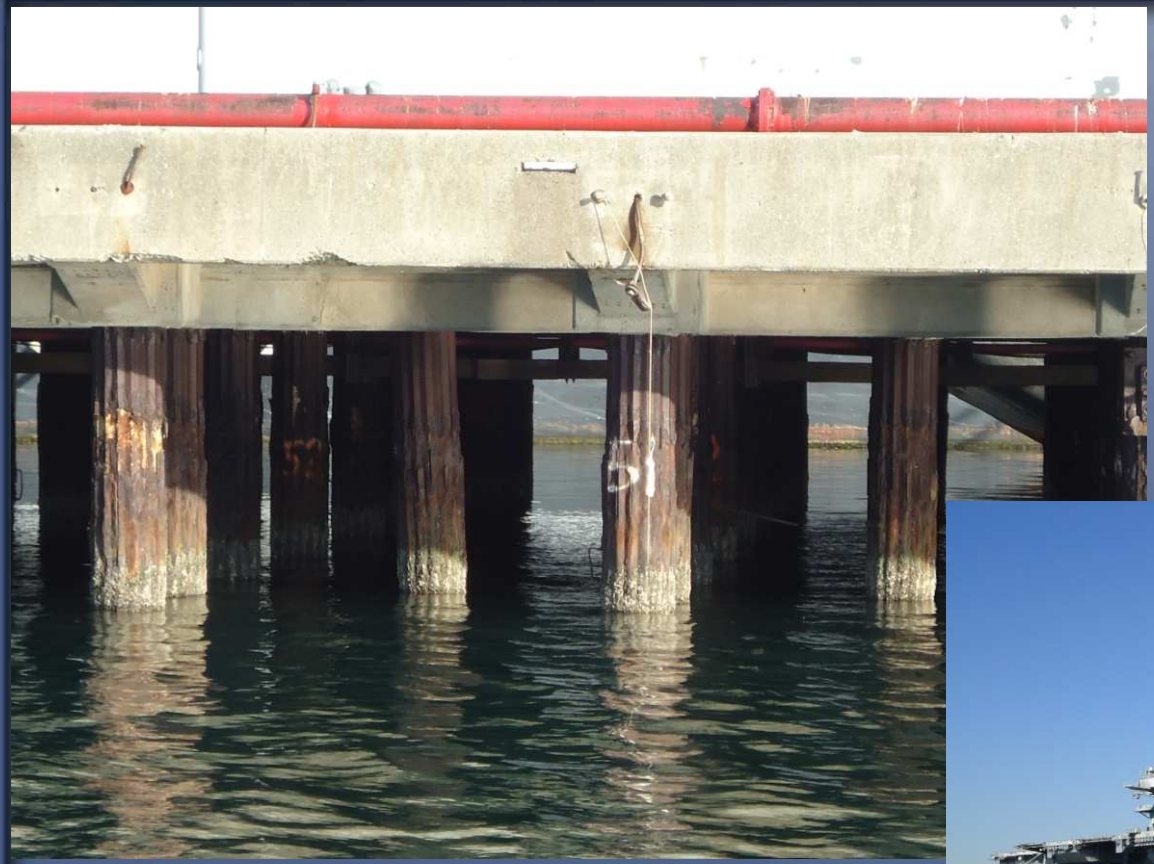
TETRA TECH

Current/Future use of Piers

- Pier 1
  - EX- Forrestal removed 2010
  - EX- Saratoga scheduled for removal 2011
  - No planned use for pier after departure of Saratoga
  - Pier 1 demolition planned but not funded.
- Pier 2
  - USCG buoy tenders to be removed to improved waterfront 2012
  - Visiting Ships on occasion
  - Weight handling operations on Pier 2 to cease 2013

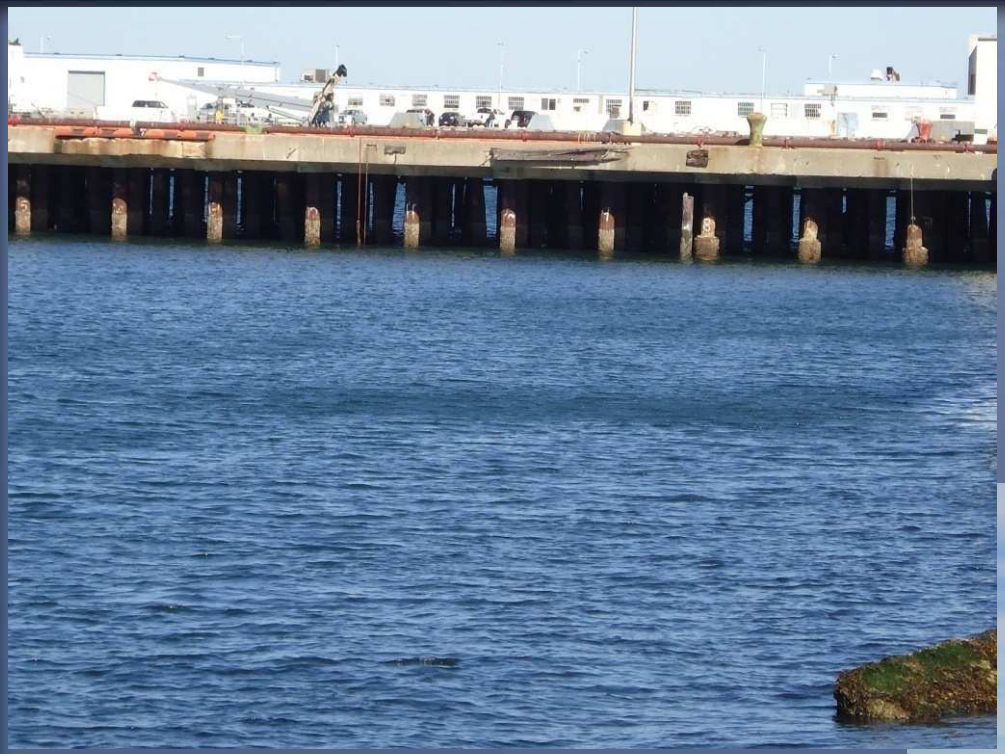
TETRA TECH

Pier 1



TETRA TECH

Pier 2



TETRA TECH

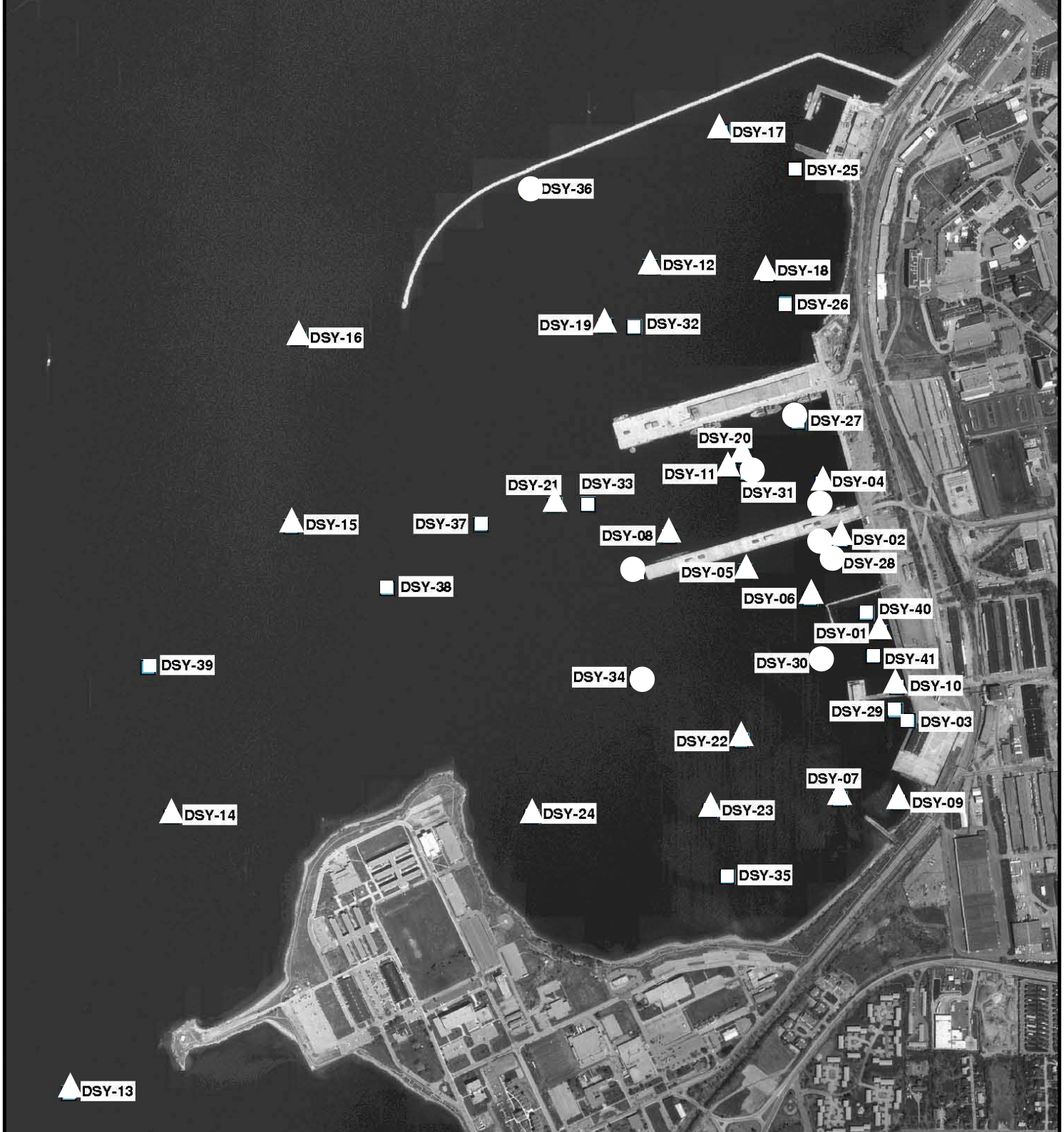
Data and PRGs

- Reference handout data table
- PRGs:
  - Benzo(a)Pyrene (HHRA – shellfish ingestion) 539 ug/kg
  - HMW PAHs (ERA, bedded sediment) – 13,903 ug/kg
  - Total PCBs (ERA, suspended sediment) – 1,060 ug/kg
  - Lead (ERA, suspended sediment) – 168 mg/kg
- Screening value for TBT calculated in 2009 – 228 ug/kg
- Combination of exceedances

TETRA TECH

All stations sampled

- ▲ 0-2 cm grab
- ■ 0- 6 inch grab
- ● Core



All Samples Exceeding PRGs, 0’ – 1’

- △ 0-2 cm grab
- ▫ 0- 6 inch grab
- ○ Core

- C < PRG
- No Data
- C > PRG

B(a)P
HMW PAHs
PCBs
Lead
TBT
Multiple

All Samples Exceeding PRGs, 1’ – 2’

- △ 0-2 cm grab
- ▫ 0- 6 inch grab
- ○ Core

- C < PRG
- No Data
- C > PRG
  - B(a)P
  - HMW PAHs
  - PCBs
  - Lead
  - TBT
  - Multiple

All Samples Exceeding PRGs, 2’ – 3’

- △ 0-2 cm grab
- ▫ 0- 6 inch grab
- ○ Core

- C < PRG
- No Data
- C > PRG
  - B(a)P
  - HMW PAHs
  - PCBs
  - Lead
  - TBT
  - Multiple

All Samples Exceeding PRGs, 3’ – 4’

- △ 0-2 cm grab
- ▫ 0- 6 inch grab
- ○ Core

- C < PRG
- No Data
- C > PRG
  - B(a)P
  - HMW PAHs
  - PCBs
  - Lead
  - TBT
  - Multiple

All Samples Exceeding PRGs, 4’ – 5’

- △ 0-2 cm grab
- ▫ 0- 6 inch grab
- ○ Core

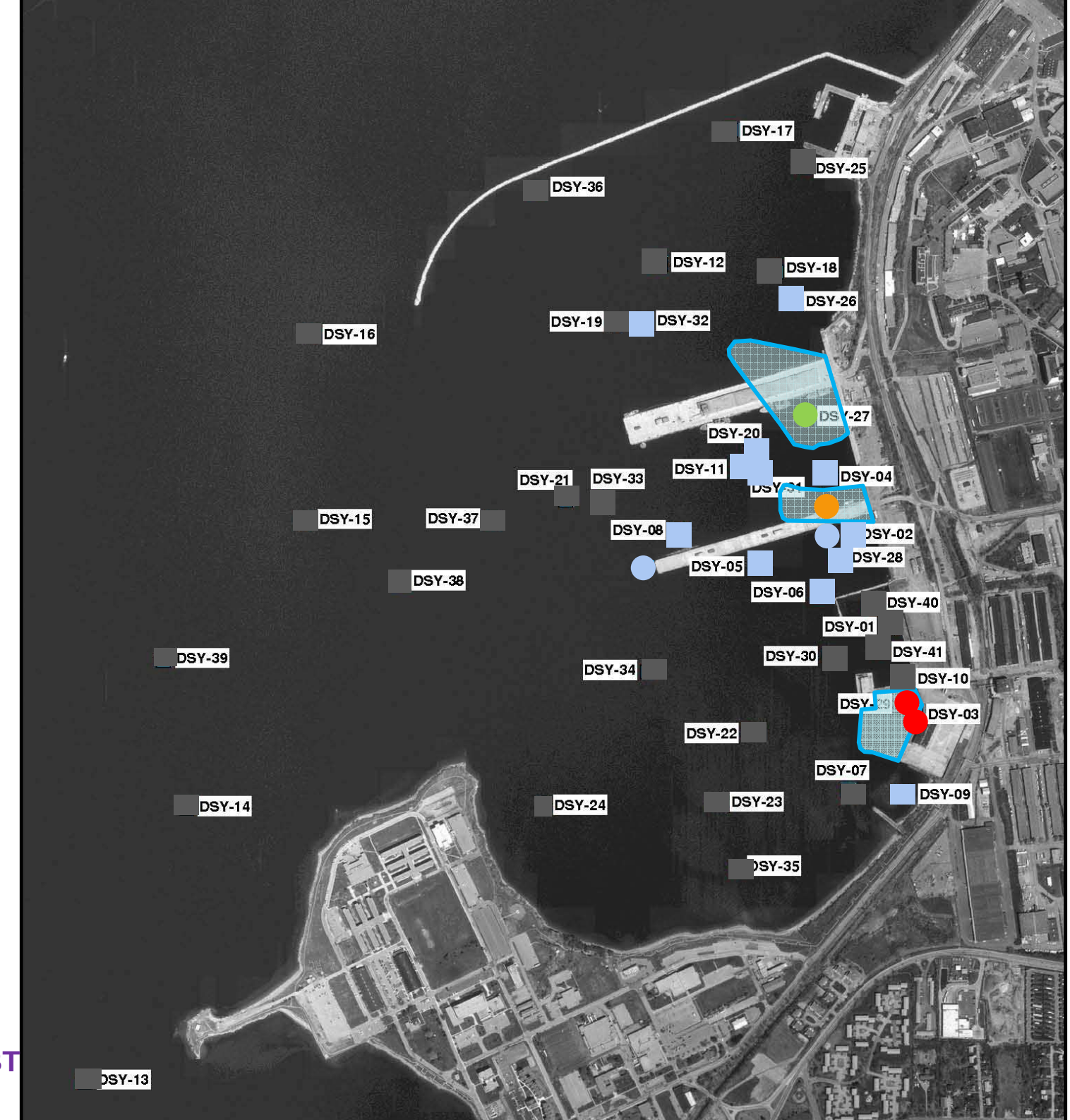
- C < PRG
- No Data
- C > PRG
  - B(a)P
  - HMW PAHs
  - PCBs
  - Lead
  - TBT
  - Multiple

Stations exceeding PRGs in 2004

Areas shown in 2008 FS report

- C < PRG
- No Data
- C > PRG

B(a)P
HMW PAHs
PCBs
Multiple
Not sampled for TBT



PART 2

PROPOSED PATH FORWARD

TETRA TECH

Proposed Path Forward

- Initiate Data Gaps Investigation
  - Further characterize extent of contamination in areas previously exceeding PRGs.
  - Characterize extent of contamination in areas not previously accessible (under piers).
  - Use this information to determine if a Removal Action is necessary or other alternatives should be considered.
- Conduct Removal Action if needed.
- Prepare Focused FS if needed.
- PRAP and ROD

TETRA TECH

Proposed path forward – Possible schedule

- Supplemental Investigation March 2011 – April 2012
- Non Time Critical Removal Action
  - EECA, Action Memo (February 2012 – November 2012)
  - Design, Removal (October 2012 - complete)
- Focused FS if necessary (October 2012 – July 2013)
- PRAP (June 2013 – February 2014)
- ROD (Sept 2013 – March 2014)

TETRA TECH

PART 3

ADDITIONAL DATA NEEDS FOR SUPPLEMENTAL INVESTIGATION (BASIC DQOs)

TETRA TECH

Data to be considered

- Physical data needs
  - Detailed sediment bathymetry mapping (multi-beam sonar)
  - Detailed hydrographics (flow velocity via ADCP deployments where scouring is feared)
  - Sediment Stability Analysis such as flume testing
  - Geochemical (radioisotope) analysis within selected cores

TETRA TECH

Data to be considered

- Chemical data needs
  - COCs* on surface
  - COCs* at depth
  - COCs* under piers not previously accessible
  - Asbestos at a subset of stations (surface sediment under piers).

*COCs = Total PCBs, HMW PAHs, lead, benzo(a)pyrene.

TBT analysis is proposed as a screening parameter

TETRA TECH

DQOs for Supplemental Investigation Chemical Data Needs

- What analytes to sample for
  - COCs determined by BERA (+ TBT)
  - Asbestos in sediment under pier 1
- What concentrations are important
  - PRG Concentrations and the TBT screening value.
- Where do we look
  - Areas where PRGs were exceeded on the surface during 2004.
  - Propose to look 3 feet in depth in these areas (no exceedance below this depth).

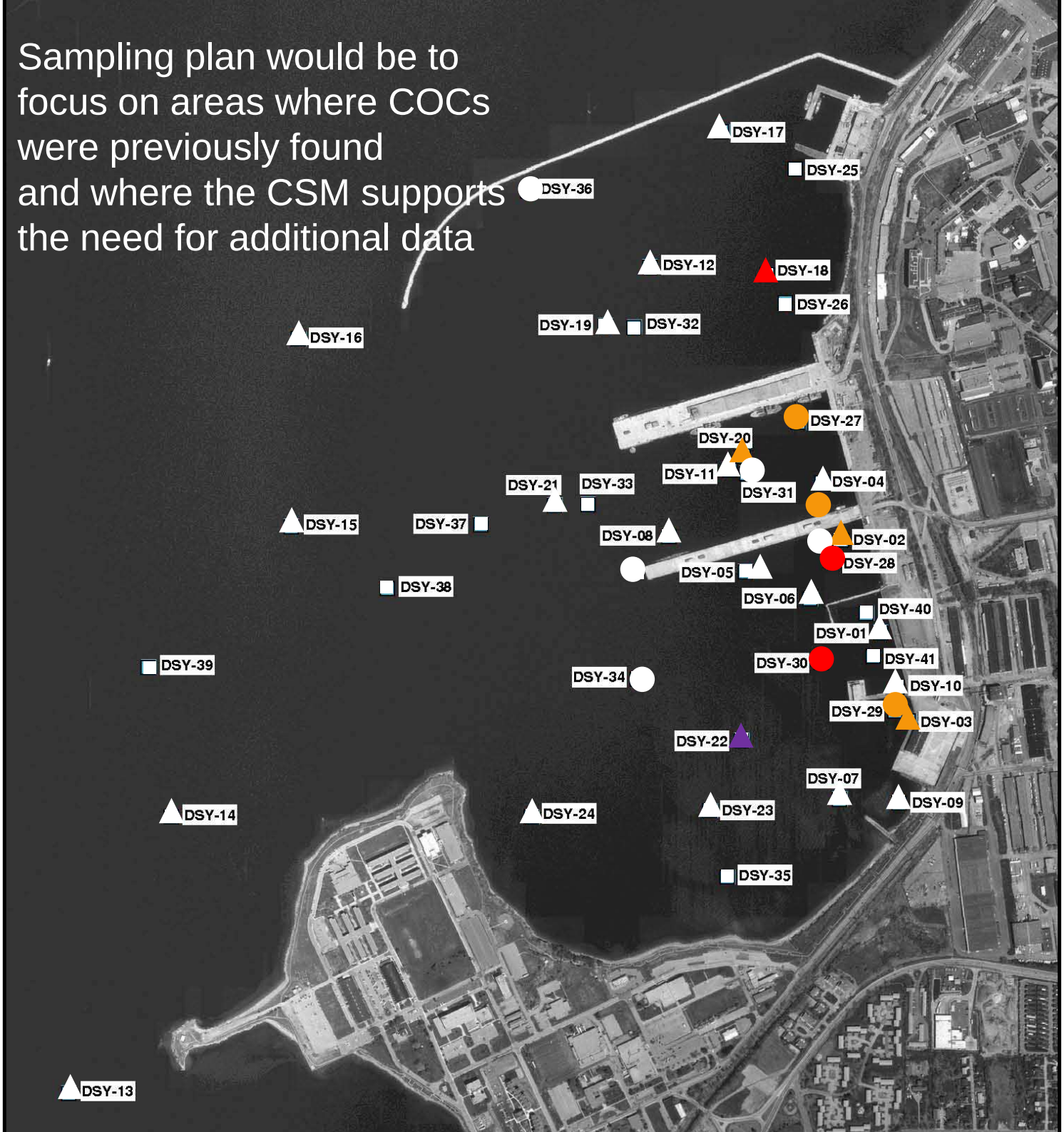
TETRA TECH

All Samples Exceeding PRGs, 0’ – 1’

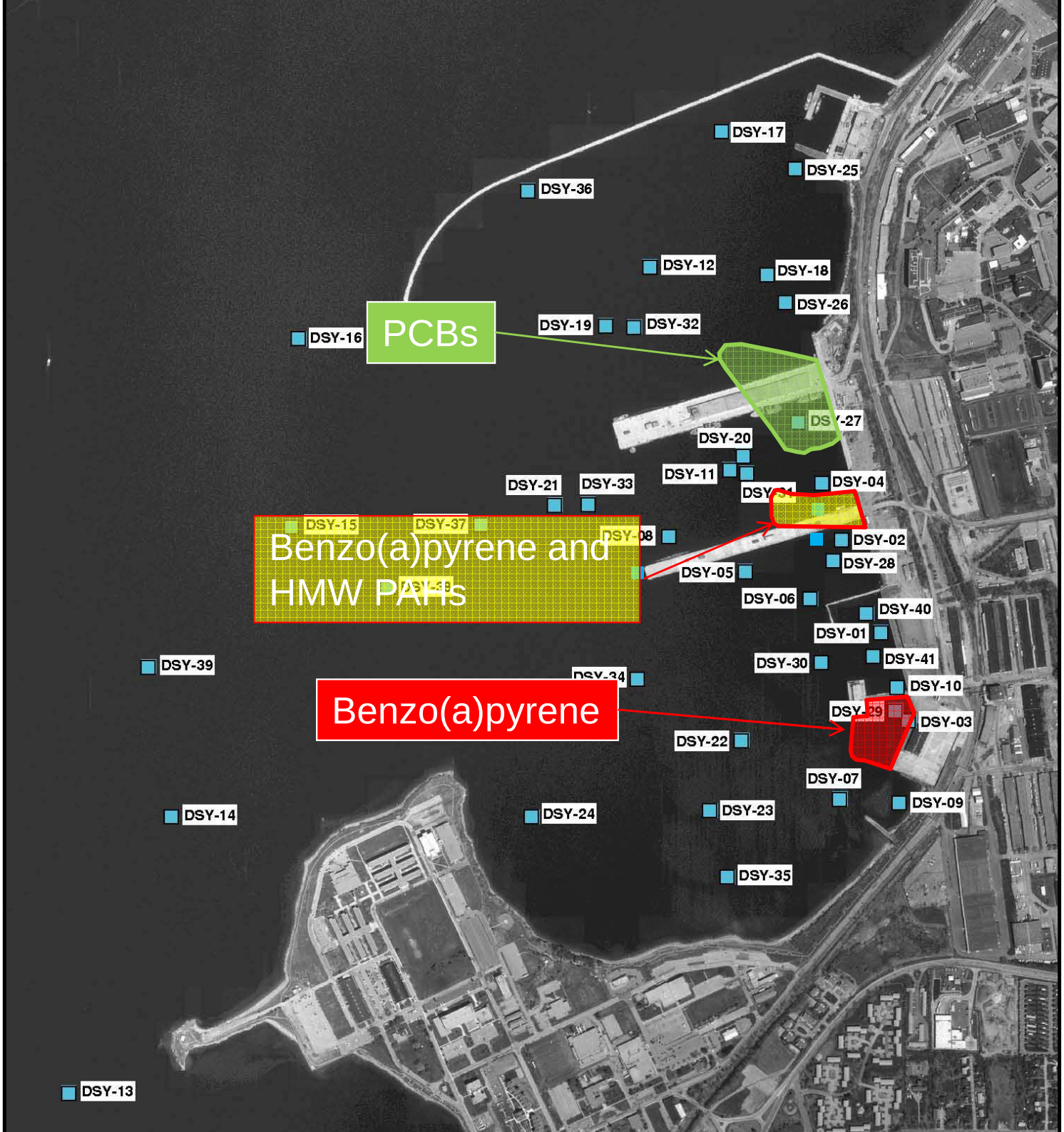
- △ 0-2 cm grab
- ▫ 0- 6 inch grab
- ○ Core

- ● C < PRG
- ● No Data
- ● C > PRG
  - B(a)P
  - HMW PAHs
  - PCBs
  - Lead
  - TBT
  - Multiple

Sampling plan would be to focus on areas where COCs were previously found and where the CSM supports the need for additional data

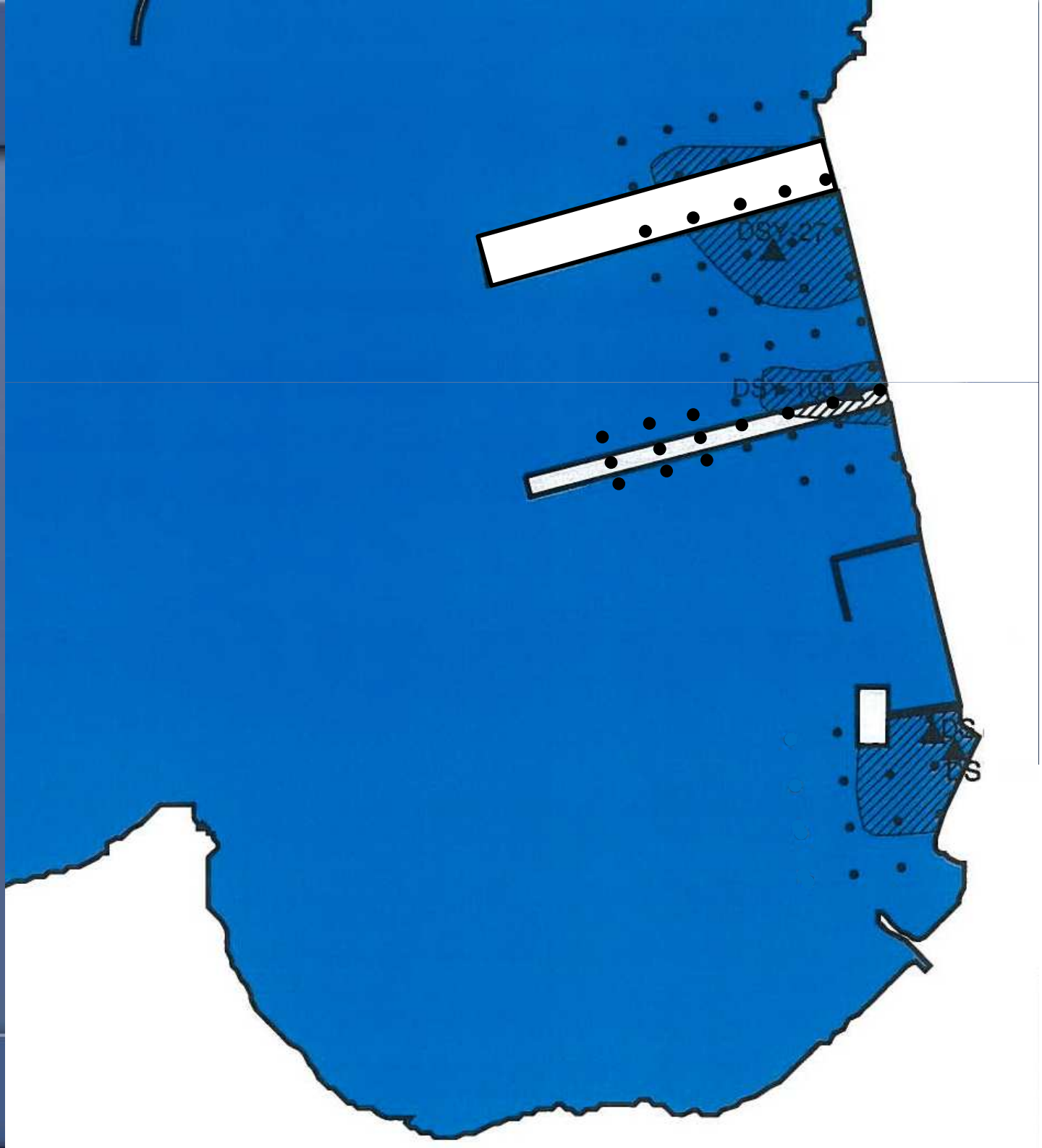


Areas exceeding PRGS on surface sediment, 2004 sampling



Sample Scheme

- PDI from FS, expanded.
- Grabs to 6 inches
- Core samples - 1’-2’ and 2’-3’
- 200 foot grid spacing shown, with exception of cores at Pier 1



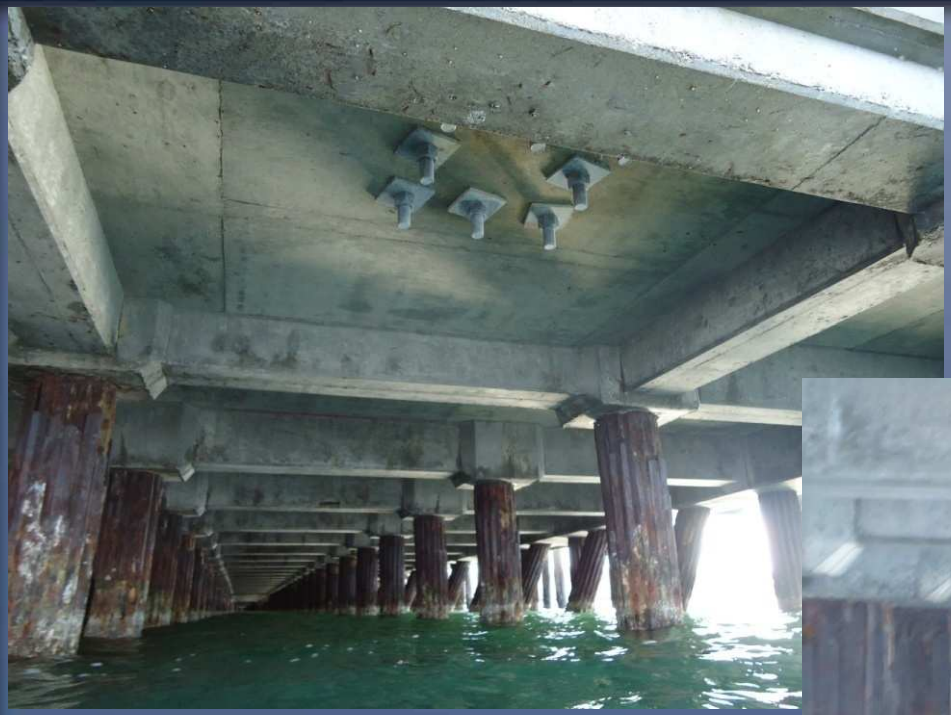
Discussion: Basic Decision Rules

- SURFACE:
  - If a hot spot* is encountered in the surface sediment, Removal Actions will be conducted (cap or dredge).
  - If PRGs are exceeded on the surface, but there are no hot spots, site average concentrations will be evaluated to see if area risk merits an aggressive action.
- SUBSURFACE:
  - If there is potential for scouring* (based on physical data) and hot spots are present in both surface and deeper sediment, then a Removal Action will be required for that sediment.
  - If there is no potential for scouring (based on physical data) and hot spots are present at depths below six inches and up to 3 feet, then monitoring is adequate.

* Terms to be defined

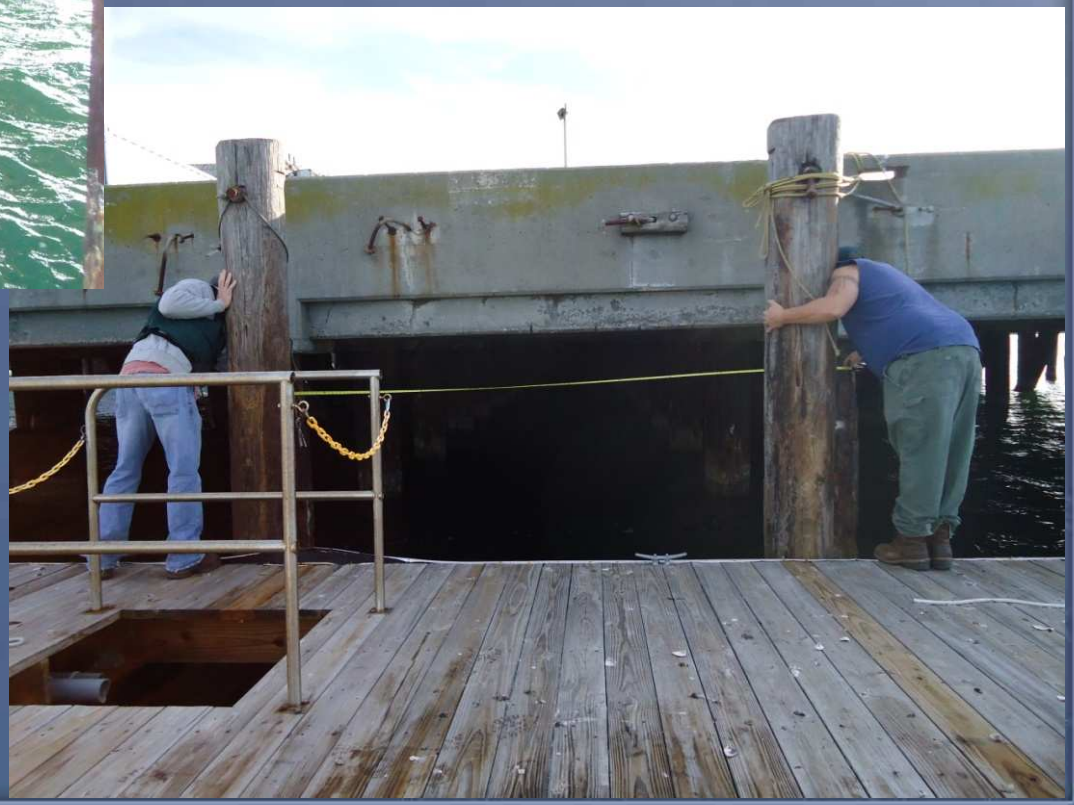
TETRA TECH

Access Under Piers



TETRA TECH

Pier 1 Inspection



TETRA TECH

Physical Limitations and Considerations

- Working under Piers
  - Height clearance approximately 4 feet at high tide, 7 feet at low tide.
  - Deck is approximately 12 inches thick at minimum points.
  - Framing members appear to be constructed as shown on design plans.
  - Water depths under pier 1 ranges from 14 feet at eastern end to 33 feet at western end. Appears to match original grade prior to dredging.
  - No access to small boats or equipment from sides of pier 1, only from west end.
  - NOT an issue for sample collection, however will be an issue for dredging, if selected.

TETRA TECH

Discussion: What is an acceptable action level for RA? (A PRG is not necessarily an RG)

- Define hot spot

TETRA TECH

Future DQO Meeting (technical details – 1 of 2)

- To address hot spots, determine depth and extent ahead of time and don't do confirmation samples (many PDI samples, but no post-excavation samples).

Or

- Design for a generalized area (few PDI samples) and conduct post excavation samples to document the condition, but not to direct additional passes with the dredge. If resulting conditions are still in excess of the RG, provide a secondary solution – thin layer cover.

TETRA TECH

Future DQO Meeting (technical details – 2 of 2)

- Define “potential for scouring”
- Physical data needs
  - Density of physical data points
  - Locations of data points

TETRA TECH

Future DQO Meeting: Plan a date, select attendees.

- Date:
- Attending:
- Goals:

TETRA TECH

THANK YOU

TETRA TECH

Notes from the Conference Call November 22, 2010

Conceptual Sediment Sampling Plan Dated 11/9/10
Derecktor Shipyard NAVSTA Site 19
NAVSTA Newport, Newport Rhode Island

Reference: 1. Conceptual Plan and Illustration dated 11/9/10.
2. EPA comments on same 11/18/11

Participating:

Dave Barclift, NAVFAC LANT
Ken Finkelstein, NOAA
Todd Finlayson, Gannett Fleming
Arun Gavaskar, NAVFAC LANT
Bart Hoskins, USEPA
Gary Jablonski, RIDEM
Winoma Johnson, NAVFAC Mid Atlantic
Kymberlee Keckler, USEPA
Greg Kemp, Gannett Fleming
Ken Munney, US Fish and Wildlife
Stephen Parker, Tetra Tech

The meeting convened at 10:14 AM

W. Johnson opened the meeting and asked for everyone to keep the team informed with their thoughts as the sampling plan develops. It was noted that based on the conceptual plan submitted by the Navy 11/9/10 there were comments from NOAA, USF&W, RIDEM and EPA. W. Johnson stated that the objective of todays call is to understand what everyone was thinking for the conceptual sampling plan.

S. Parker presented the proposed analyte list and submittal dated 11/9/10 (attached). In regards to the analyte list, K.Keckler stated that the Army Corps data from (1987?) shows that zinc exceeded the BPRG for this analyte in sediment near both Pier 1 and Pier 2. The Navy took it as action to review this data (provided in the BERA) and provide a response to EPA.

S. Parker explained that each subunit (or decision unit) of the site would be represented by a series of samples, and within each subunit those samples would be collected to a depth of 2 feet below the sediment surface. K. Keckler stated that she felt that the subunits were too large to be practical, for a decision on how to develop a remedy for each. K. Munney asked how hot spots would be identified in this type of a plan. S. Parker stated that the identification of hot spots were not a target of this plan and acknowledged that the data would have to be utilized without the subunit boundaries to identify hot spots.

K. Finkelstein stated that some of the PRGs are not supportable. He believes that all the stations should be analyzed for zinc and copper as well as the RPRGs that were identified in the previous FS version. However, he also stated that using these BPRGs

for remediation goals was not appropriate. It was noted that if the data was not used for remediation goals, it was uncertain what it would be used for.

W. Johnson stated that the PRG document is finalized. K. Keckler stated that this may not be the case, and indicated that a separate meeting may be appropriate on that subject. It was agreed that for the time being, discussion on the PRGs would be tabled.

K. Keckler presented her sampling plan (11/18) showing cross hated areas and non. The cross-hatched areas should be individual cores, and other areas not hatched should be addressed with composite samples. The plan shows 26 decision units, and therefore there would be 26 samples. In addition, there should be composites for the different depth intervals collected.

After some discussion on this plan, it was agreed that the Navy would take it into consideration, and come up with a combined plan using the EPA approach and composite data/sample collection. K. Keckler stated that individual cores were appropriate for the cross-hatched areas, and cores should be used on 100 foot grids to develop the composites described above.

There was a discussion about Pier 2. K. Munney stated that the outer portions of Pier 2 were also of concern, and though the source may not be from a CERCLA release, additional data should be sought from this area and considered.

There was discussion on comment no. 12 from EPA E-mail 11/18/10. The comment was in regards to testing the effluent from outfalls along the waterfront that may provide non-point source contaminants to the study area. K. Keckler stated that these outfalls need to be considered. W. Johnson stated that the site sources are addressed – the removal actions were conducted that removed the sand blast grit, drummed materials, and on shore investigations do not show soil contamination that provides additional sources of contaminants to the marine environment. K. Keckler noted that if the Navy wishes to consider MNA as a remedy for the site, they will need to consider contaminant inputs, including these non-point sources.

The NOAA Comments were reviewed

MIS – It was agreed by the group that Multi-increment sampling was not appropriate for sediment.

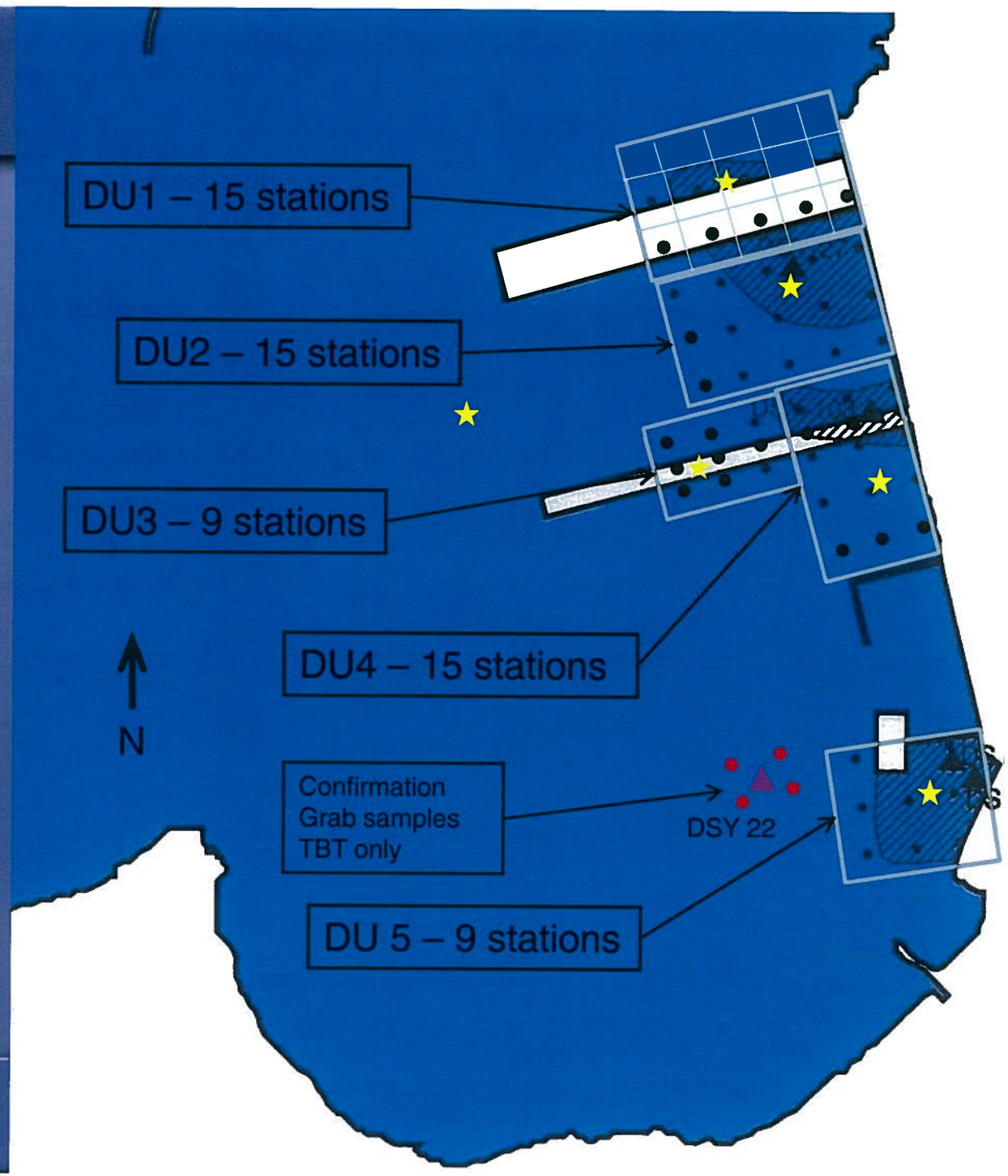
Pre-2004 injury – NOAA is considering pursuit, but it was acknowledged that the state would have to provide the lead, since they were the ones who suffered the injury. If the 2004 data is duplicated by this effort, and the ultimate remedy is MNR, then the injury would be the former release as documented by the older data, and NOAA would look for a restoration project in compensation. The state of Rhode Island would have to settle the claim. There was some discussion on what type of project would be appropriate. There could be an agreement without arbitration that would benefit all parties, avoid the legal costs. W. Johnson stated that this will be discussed in the future as needed. K. Finkelstein stated that his concern is in regards to the cumulative risk to the receptors present.

It was agreed that the Navy would provide a revised plan for sampling out to the work group by December 6, 2010 and there would be another call following to discuss it. The follow up call was tentatively agreed on for December 14.

Conceptual Sampling Plan

- Establish five Decision Units. Each unit can be broken into subunits of 200 foot squares (subunits are on DU 1 only)
- Collect one core at each station to two feet. Samples 0-1 foot, 1-2 feet
- Conduct four cores at each DU to 4 feet max, analyze them in one foot intervals
- Conduct four confirmation grab samples around the former location DSY22 due to a previous hit of TBT.
- Conduct geophysical sampling at one station within each DU, and one outside the area

- Grab sample
- ★ Geophysical sample station
- Dots are illustrative only



Conceptual Sampling Plan

Data Gaps Investigation, Site 19: Former Derecktor Shipyard Marine Sediment

Newport RI

Data Quality Objectives

Data will be collected to support a decision as to whether a remedial action is required in portions of the study area that are affected by site related contaminants. Data needs to be adequate to support the selection of the action that would be required. These actions could include, but may not be limited to: removal (dredging), cover (thin layer cap), monitored natural recovery, and/or institutional controls.

It has been determined that contaminants present in the sediment do or did pose unacceptable risk to human and ecological receptors (SAIC 1997). Based on these calculated risks, "Baseline Preliminary Remediation Goals" (BPRGs) were developed based on a HQ of 1.0, as published in SAIC, 1998. A subset of "recommended PRGs" was developed to best represent site related contaminants as a first step in risk management. Those that were not selected for calculation of recommended PRGs were:

- **o,p'-DDE –**
  - Estimated to pose risk to amphipods from suspended sediment modeled through an elutriate test, however baseline PRG was only exceeded in historical data at one sample station, not considered a contaminant that originated at the site.
- **Arsenic –**
  - Estimated to pose risk to human health through ingestion of shellfish and lobster, however most arsenic in shellfish is present in a non toxic organic form (SAIC 1998, pp 30-31).
  - Estimated to pose risk to avian predators, however data did not show arsenic above the BPRG level for this receptor.
  - Estimated to pose risk to amphipods from suspended sediment modeled through an elutriate test, however because locations of high risk did not match the locations of high concentrations, this PRG was not recommended for implementation.
- **Copper-**
  - Estimated to pose risk to avian predators, however site data only showed two exceedances above the BPRG level for this receptor.
  - Estimated to pose risk to amphipods from suspended sediment modeled through an elutriate test, however because locations of high risk did not match the locations of high concentrations, this PRG was not recommended for implementation.
- **Silver -**
  - Estimated to pose risk to avian predators, however data did not show silver above the BPRG level for this receptor.
- **Zinc –**
  - Estimated to pose risk to avian predators, and exceeded BPRGs at most stations. However, exceedances were slight, and limiting process showed that other, more toxic COCs were co-located with zinc that would be addressed.

Four COCs were recommended to be retained for use to direct remedial actions. These are:

Benzo(a)pyrene
Total HMW PAHs
Total PCBs
Lead

Secondary constituents of concern have been identified recently and include:

TBT – highly toxic, but not found to be a COC for the site
Asbestos – known to have been released from under Pier 1
Copper – possibly deposited from the 10 year berthing of the Ex Saratoga, and Ex Forrestal at Pier 1.

Approach for Data Collection:

To support the decision as to whether to take an action as stated above, The following approach is proposed:

Establish five decision units throughout the study area as shown on the attached sketch. The decision units were developed based on location, confining site features, source areas, and previously collected data:

DU1 – Area under and north of Pier 2. Mostly composed of area not previously sampled, but included in a potential action area associated with DSY 27.

DU2 – Area between piers 1 and 2. Anticipated to have taken heavy ship traffic, and still trafficked by NOAA ocean buoy tenders. Includes former location of DSY 27 where PCBs were previously found in sediment exceeding PRGs

DU3 – Area of pier 1 where carriers were berthed, area under the carrier hulls and where dry docks previously were berthed and used for ship maintenance by Derecktor. Most portions not previously sampled except for metals in 1987 by corps of engineers. This DU Includes sediment under this active section of the pier.

DU4 – location of DSY 28, PAHs exceed PRGs to a depth of 3 feet below sediment surface. Former location of storm drain north of Building 42.

DU5 – location around stations DSY 29 and DSY 3. Former location of the Greenport Ferry and location where a large storm drain outfall discharges from Building 234 area. Also downgradient of the former setup shop (Building 234). PAHs and lead previously exceeded PRGs in sediment.

Each decision unit may be broken down into subunits that are comprised of squares 200x200 feet. Each larger Decision Unit will be made up of between 9 and 15 of these subunits. The size of the subunit was selected based on the size of the area to be addressed.

Chemical data will be collected at one foot intervals in one position within each of the subunits to a total depth of 2 feet through coring. The one foot interval is selected because that is the effective vertical precision of most dredging equipment. This may become important if dredging is found to be necessary within subunits or Decision Units. The vertical limit of two feet is selected because at most stations cored (with the exception of DSY 28) clean conditions were previously found in the 1-2 foot sample interval.

Four cores within each Decision Unit will be continued to four feet below sediment surface, and sampled at one foot intervals. The four foot depth limit for the subset of cores was selected based on a) the previous finding that sediments in the one core at DSY 28 that was previously advanced to this depth did not exceed PRGs below three feet, and b) by EPA request. The location of these four cores within each Decision Unit is not determined at this time, and will be selected based on presence of depositional sediment found during field efforts.

Decision Points

1. The chemical data from all the subunits taken collectively within each interval will be used to determine whether an action is required for the larger Decision Unit at that interval.

2. If a hot spot is defined within the Decision Unit, then that hot spot may be addressed individually.

3. If the sediment exceeds PRGs at depth but does not exceed PRGs in the surface interval, physical data will be used to determine if that sediment is at risk of being exposed under expected current conditions.

Chemical Data Collection

In each subunit, one core will be collected to a depth of two feet below sediment surface. These cores will be sampled at depth intervals of 0-1 foot and 1-2 feet, as stated above.

Reference attached figure for proposed station locations.

Chemical analysis shall be limited to the four Recommended COCs at all stations (Sum HMW PAHs, benzo(a)pyrene, total PCBs, and total lead). Secondary constituents of interest will be added as follows:

- Copper will be added at stations not previously sampled (under Piers, under carrier berthing locations). These include five stations at DU1, nine stations at DU3, and six stations at DU4.

- Asbestos analysis will be added to the surface intervals at 6 stations under Pier 1.

- By previous agreement TBT will be included at all sample stations.

The location of SD22 which showed an exceedance of the TBT screening value in 1993 will have surface grabs conducted around it to determine presence of COCs in surface sediment, by regulatory request.

Physical Data Collection

A multibeam sonar survey will be conducted within an area approximately 200 feet north of Pier 2, and between Pier 1 south to the CCRF surface discharge outfall (small peninsula to the south of the site). This survey will be tied to the survey conducted in 2007 and mapped as one area. Data will be used to identify depositional areas, scour areas, and overall changes in depth over time.

The hydrographic survey conducted by URI in 1996 will be augmented by deployed ADCP profilers during a 15 day period. Deployments at six stations near and around piers will assist in determination of strength of fluctuating and short term currents at the sediment surface.

Sediment stability testing is proposed for the same six stations. Stablity testing is proposed to be performed by ERDC Coastal and Hydraulics Laboratory, Vicksburg MS. The method to be employed will be a cohesive sediment erosion flume, proposed and planned for use at Gould Island (IR Site 17) during the same period of time. This data will provide information as to the current velocity required to move sediments from different depths within the sediment strata

Radioisotope analysis of cores will be conducted at six stations where the flume testing is being done. Analysis will include lead 210 which has a steady decay rate and a half life of 22 years, as well as a relatively steady deposition rate that should not be impacted by the presence of lead contaminants from site discharge. The lead 210 isotope will be measured every few centimeters down the core. Radioisotope analysis will be used to determine accumulation rate of sediment within each decision unit.

References:

SAIC 1998, Preliminary Remediation Goals for Derecktor Shipyard, Coddington Cove Prepared under Contract to Tetra Tech NUS, for US Navy Northern Division. Final. November 1998.

SAIC and Univeristy of Rhode Island, Ecological Risk Assessment for Derecktor Shipyard, Coddington Cove. Prepared under Contract to Tetra Tech NUS, for US Navy Northern Division. Final. May 1997.

Tetra Tech NUS, Inc. 1998, Marine Human Health Risk Assessment, Offshore Areas of the Former Robert E. Derecktor Shipyard, NETC Newport September, Final, 1998

Kincaid, et. al. 1996. Hydrographic Survey for the Offshore Ecological Risk Assessment at Derecktor Shipyard / Coddington Cove, Naval Education and Training Center, Newport RI. June.

Tetra Tech 2008. Summary of TBT at Site 19, Derecktor Shipyard, Naval Station Newport, Newport RI. Revised November 7, 2008

NFESC, 1998. Deep Draft Berthing Facility Site Plan, Naval Facilities Engineering Service Center, East Coast Detachment. Washington DC. July.

EPA COMMENTS AND REVISION

Conceptual Sampling Plan

Data Gaps Investigation, Site 19: Former Derecktor Shipyard Marine Sediment

Newport RI

Data Quality Objectives

Data will be collected to support remedial decision-making in portions of the study area that are affected by site related contaminants. Data must be adequate to support remedy selection. These actions could include, but may not be limited to: removal (dredging), cover (full cap or thin layer cap), monitored natural recovery, and/or institutional controls. These actions will be fully evaluated in either an EE/CA or FS.

- It was previously determined that contaminants present in the sediment pose unacceptable risk to human and ecological receptors (SAIC 1997). Based on these calculated risks, "Baseline Preliminary Remediation Goals" (BPRGs) were developed based on a HQ of 1.0, as published in SAIC, 1998. The following PRGs will be used to determine whether cleanup is necessary and when it is completed.
  - Lead 168 ug/g dry weight
  - Benzo(a)pyrene 539 ng/g dry weight
  - HMW PAHs 13903 ng/g dry weight
  - Total PCBs 1060 ng/g dry weight
  - Copper 74 ug/g dry weight
  - Zinc 118 ug/g dry weight

Note: As discussed at the October 27, 2010 meeting, EPA believes that copper should be carried through as a recommended PRG. The Navy's Inactive Ship Deep Draft Berthing Facility Environmental Assessment dated December 8, 1997 estimated that "...the combined copper release rate is approximately 8 pounds per day for the three ships..." that were berthed at Derecktor Shipyard. These ships represent an additional loading of contaminants into the environment since the toxicity tests were taken.

The following constituents will also be measured:

TBT – highly toxic, but not found to be a COC for the site

Asbestos – known to have been released from under Pier 1

.

Approach for Data Collection:

To determine where remedial action is warranted, the following approach is proposed:

Establish twenty five decision units throughout the study area as shown on the attached sketch. The decision units were developed based on location, confining site features, source areas, and previously collected data:

DU1 – Area under and north of Pier 2. Mostly composed of area not previously sampled, but included in a potential action area associated with DSY 27.

DU2 – Area between piers 1 and 2. Anticipated to have taken heavy ship traffic, and still trafficked by NOAA ocean buoy tenders. Includes former location of DSY 27 where PCBs were previously found in sediment exceeding PRGs

DU3 – Area of pier 1 where carriers were berthed, area under the carrier hulls and where dry docks previously were berthed and used for ship maintenance by Derecktor. Most portions not previously sampled except for metals in 1987 by corps of engineers. This DU Includes sediment under, along, and adjacent to this active section of the pier.

DU4 – location of DSY 28, PAHs exceed PRGs to a depth of 3 feet below sediment surface. Former location of storm drain north of Building 42.

DU5 – location around stations DSY 29 and DSY 3. Former location of the Greenport Ferry and location where a large storm drain outfall discharges from Building 234 area. Also downgradient of the former setup shop (Building 234). PAHs and lead previously exceeded PRGs in sediment.

This description will obviously have to change based on our discussions and which approach(es) we decide to use.

Each decision unit may be broken down into subunits that are comprised of squares100x100 feet. Each larger Decision Unit will be made up of between 6 and 12 of these subunits. The size of the subunit was selected based on the size of the area to be addressed.

Chemical data will be collected at one foot intervals as 5-point composite samples within each of the subunits to a total depth of 2 to 4 feet through coring. The one foot interval is selected because that is the effective vertical precision of most dredging equipment. This may become important if dredging is necessary within subunits or Decision Units. A vertical limit of two feet was for most areas because at most stations cored (with the exception of DSY 28) clean conditions were previously found in the 1-2 foot sample interval. Deeper cores are required because contamination at depth that potentially could be mobilized because of vehicle traffic and periodic storms. Each core will be analyzed in three strata (0 to 1; 1 to 2; 2 to 4).

Four cores within each Decision Unit will be continued to four feet below sediment surface, and sampled at one foot intervals. The four foot depth limit for the subset of cores was selected based on a) the previous finding that sediments in the one core at DSY 28 that was previously advanced to this depth did not exceed PRGs below three feet, and b) by EPA request. The location of these four cores within each Decision Unit will be selected with consideration to the presence of depositional sediment found during field efforts.

Decision Points

1. The chemical data from all the subunits taken collectively within each interval will be used to determine whether an action is required over the extent of the larger Decision Unit at that interval. (How? Geometric mean? Median?)

2. If a hot spot (one or more subunits) is defined within the Decision Unit, then that hot spot will be addressed.

3. If the sediment exceeds PRGs at depth but does not exceed PRGs in the surface interval, physical data and sediment transport analyses will be used to determine if that sediment could be exposed under expected current, operational, or storm conditions.

Chemical Data Collection

In each subunit, one core will be collected to a depth of two feet below sediment surface. These cores will be sampled at depth intervals of 0-1 foot and 1-2 feet, as stated above.

Reference attached figure for proposed station locations.

Chemical analysis shall be limited to the six aforementioned PRGs at all stations (Sum HMW PAHs, benzo(a)pyrene, total PCBs, total lead, copper, zinc). Copper analysis will be limited to the areas under Piers and under berthing locations.

- Asbestos analysis will be added to all samples associated with Pier 1 and Pier 2. Additional sampling for asbestos may be required depending on the results of this sampling effort

- By previous agreement TBT analysis will be included at all sample stations. The location of SD22 which showed an exceedance of the TBT screening value in 1993 will have core samples collected around it to determine presence of COCs in surface and subsurface sediment, by regulatory request.

- TOC and grain size

Physical Data Collection

A multibeam sonar survey will be conducted within an area beginning approximately 200 feet north of Pier 2, and extending south to the CCRF surface discharge outfall (small peninsula to the south of the site) including the area between Piers 1 and 2 and extending from the eastern shore to the west end of the piers. This survey will be compared to the survey conducted in 2007 and anomalies noted. Data will be used to identify depositional areas, scour areas, and overall changes in depth over time. (The initial description provided by Tetra Tech was unclear and EPA has not checked on the extent of the 2007 survey to compare with the initial description for this planned survey.)

The hydrographic survey conducted by URI in 1996 will be augmented by deployed ADCP profilers during a 15 day period. Deployments at six stations near and around piers will assist in determination of strength of fluctuating and short term currents at the sediment surface.

Sediment stability testing is proposed for the same six stations (a single sample per DU does not seem to adequately represent sediment characteristics for such a large area). Stablity testing is proposed to be performed by ERDC Coastal and Hydraulics Laboratory, Vicksburg MS. The method to be employed will be a cohesive sediment erosion flume, proposed and planned for use at Gould Island (IR Site 17) during the same period of time. This data will provide information as to the current velocity required to move sediments from different depths within the sediment strata.

Radioisotope analysis of cores will be conducted at six stations where the flume testing is being done. Analysis will include lead 210 which has a steady decay rate and a half life of 22 years, as well as a relatively steady deposition rate that should not be impacted by the presence of lead contaminants from site discharge. The lead 210 isotope will be measured every few centimeters down the core. Radioisotope analysis will be used to determine accumulation rate of sediment within each decision unit.

Note: deposition rate using isotope analysis will only be determinable in undisturbed sediment.

The simplest model, Constant Flux: Constant Sedimentation (Robbins, 1978) assumes a constant flux of excess lead- 210 from the atmosphere and a constant dry-mass sedimentation rate. Where these assumptions are satisfied the lead-210 concentration will vary exponentially in accumulating sediment. The rate of accumulation is obtained from the following equation and solved for r, the dry mass accumulation rate.

C = C(0)e-km/r **(2),**

where C(0) is the excess lead-210, m is cumulative dry-mass, and k is the decay constant for lead-210 (0.03114). The resulting profile will be linear with the slope – k/r. The sedimentation rate can be determined graphically from the mean slope determined by a best or least squares fit.

References:

SAIC 1998, Preliminary Remediation Goals for Derecktor Shipyard, Coddington Cove Prepared under Contract to Tetra Tech NUS, for US Navy Northern Division. Final. November 1998.

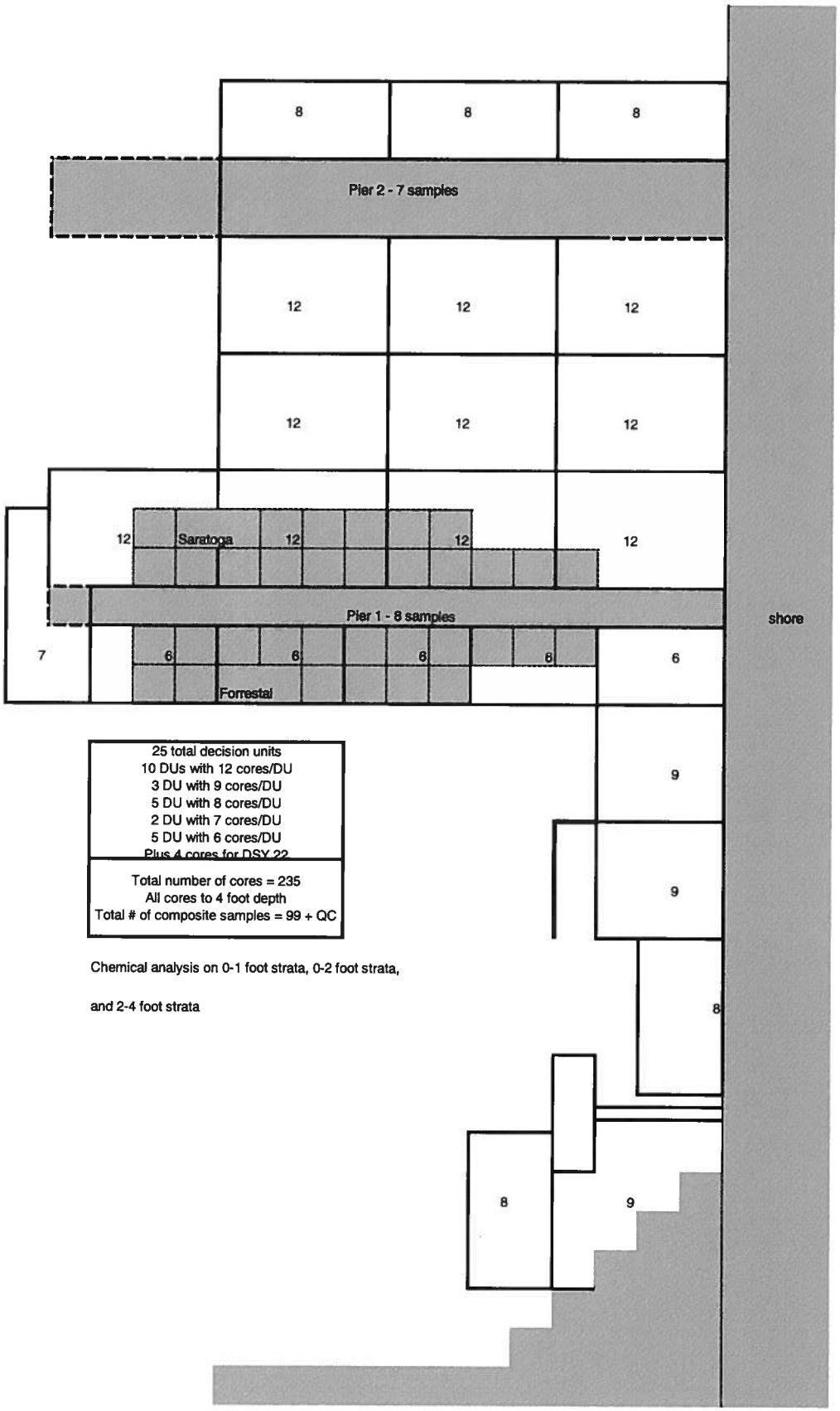
SAIC and Univeristy of Rhode Island, Ecological Risk Assessment for Derecktor Shipyard, Coddington Cove. Prepared under Contract to Tetra Tech NUS, for US Navy Northern Division. Final. May 1997.

Tetra Tech NUS, Inc. 1998, Marine Human Health Risk Assessment, Offshore Areas of the Former Robert E. Derecktor Shipyard, NETC Newport September, Final, 1998

Kincaid, et. al. 1996. Hydrographic Survey for the Offshore Ecological Risk Assessment at Derecktor Shipyard / Coddington Cove, Naval Education and Training Center, Newport RI. June.

Tetra Tech 2008. Summary of TBT at Site 19, Derecktor Shipyard, Naval Station Newport, Newport RI. Revised November 7, 2008

NFESC, 1998. Deep Draft Berthing Facility Site Plan, Naval Facilities Engineering Service Center, East Coast Detachment. Washington DC. July.



25 total decision units
10 DUs with 12 cores/DU
3 DU with 9 cores/DU
5 DU with 8 cores/DU
2 DU with 7 cores/DU
5 DU with 6 cores/DU
Plus 4 cores for DSY 22

Total number of cores = 235
All cores to 4 foot depth
Total # of composite samples = 99 + QC

Chemical analysis on 0-1 foot strata, 0-2 foot strata,

and 2-4 foot strata

TETRA TECH

C-NAVY-12-10-4068W

December 21, 2010

Project Number 112G02747

Ms. Kymberlee Keckler, Remedial Project Manager
U.S. EPA Region I
5 Post Office Square, Suite 100
Boston, Massachusetts 02114-3912

Mr. Gary Jablonski, Remedial Project Manager
Rhode Island Department of Environmental Management
235 Promenade Street
Providence RI, 02908-5767

Reference: CLEAN Contract No. N62470-08-D-0055
Contract Task Order No. WE61

Subject: Data Gaps Investigation,
IR Site 19, Former Derecktor Shipyard,
Naval Station Newport, Newport RI

Dear Ms. Keckler, Mr. Jablonski:

On behalf of Ms. Winoma Johnson, US Navy NAVFAC, I am providing to you the a follow-up from our conference call held December 14, 2010 in reference to the site identified above.

Attached to this letter, I am providing notes from the conference call referenced above. The Navy has considered and agreed to the requests for additional sample stations as discussed. Accordingly, the conceptual sampling plan and schematic have been revised and are also provided as attachments to this letter.

Based on these discussions and revisions, the Navy will move forward with the development of a sampling and analysis plan to provide details of the sampling program.

If you have any questions, please do not hesitate to contact me at 978-474-8434.

Very truly yours,

Stephen S. Parker, LSP
Project Manager

Enclosures

c: D. Barclift, NAVFAC (w/encl.)
K. Finkelstein. NOAA (w/encl.)
B. Hoskins, USEPA (w/encl.)
A. Gavaskar, NAVFAC (w/encl.)
J. Galler, TtNUS (w/encl.)
P. Golonka, Gannett Fleming (w/encl.)
W. Johnson, NAVFAC (w/encl.)
K. Munney, USF&W (w/encl.)
D. Ward, NAVSTA (w/encl.)
File 112G01474-3.1 (w/encl.)

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Notes from the Conference Call December 14, 2010

Conceptual Sediment Sampling Plan Dated 12/3/10
Derecktor Shipyard NAVSTA Site 19
NAVSTA Newport, Newport Rhode Island

Reference: Conceptual Plan and Illustration dated 12/3/10.

Participating:

Dave Barclift, NAVFAC LANT
Ken Finkelstein, NOAA
Todd Finlayson, Gannett Fleming (via phone)
Arun Gavaskar, NAVFAC LANT
Bart Hoskins, USEPA
Gary Jablonski, RIDEM
Winoma Johnson, NAVFAC Mid Atlantic
Kymberlee Keckler, USEPA
Greg Kemp, Gannett Fleming
Ken Munney, US Fish and Wildlife
Stephen Parker, Tetra Tech
Darlene Ward, NAVSTA

The meeting convened at 10:00 AM

W. Johnson opened the meeting and noted that this conference call was a follow-up to the previous call 11/22/10. After that meeting, the Navys action was to revise the plan considering comments to the previous version discussed that day, and issue the revision by December 6. The revision dated December 3 is the subject of the call today.

S. Parker introduced the plan dated 12/3/10. This plan utilizes the original approach of 200 foot grid cells with one discrete core located at the center of each cell to address most of the areas identified at the previous call – those being the area north of pier 2 to a distance of 200 feet, the area between piers 1 and 2, and areas around former stations SD-30 and SD-22. Additionally, a grid spacing of 100 feet would be utilized (with one discrete core representing each cell) in areas surrounding and shoreward of the sediment stations that exceeded PRGs in the most recent round.

All cores would be sampled using the depth scheme discussed on 11/22/10 – one sample 0-1 foot deep, one sample 1-2 feet deep, and one sample 2-4 feet deep. These samples would be analyzed for the suite described in the attachment.

S. Parker noted that additional sampling is not proposed around or near former station DSY-18, located approximately 500 feet north of Pier 2. This station had one exceedance of the PRG for Benzo(a)pyrene, in the 0-2 cm depth interval in 1993. It is also located well beyond the anticipated impact area supported by the conceptual site

model. G. Jablonski stated that he had previously requested that this location be included in this sampling effort so that it can be addressed permanently. The Navy took it as an action item to review RIDEM's request for sampling in this area and respond.

There was a side discussion about the PRGs. The PRG for benzo(a)pyrene was discussed as one with high uncertainty as to its origin being from the site or from other source inputs to the bay. The baseline PRGs for zinc and for copper were noted as was the lack of recommended PRG values for these analytes. A PRG for TBT would be calculated if the NOEC of 228 ug/kg is found.

The remainder of the sampling area was discussed, the following requests were made:

- One 200-foot sample grid was requested to be added to the west end, south side of pier 2, where a sample (ID:S4) was collected by the US Army Corps of Engineers in 1987 that contained high concentration of copper.
- Three 200-foot sample grids were requested to be added to the west end of pier 1, so that the entire pier area would have coverage. It was noted that the sample DSY101 was collected in this area in 2004.
- Three sample stations were requested to be added south of the former Building A18 near station DSY29.
- Three sample stations were requested to be added within the Stillwater Basin near to the waterfront (within 50 feet of the sheet piling wall).
- It was requested that the sample stations around DSY 22 be rearranged so that the samples surround the station as was previously proposed.

The sample analytical suite was discussed briefly. It was suggested that zinc and copper be collected at all the sample stations, but the group recalled that the previous agreement was to collect zinc and copper in areas not previously accessible to the sampling team in 2004 (Under the pier and under the carriers). G. Kemp stated that because sandblast grit was found in the former location of the ferry (DSY29) copper and zinc should at least be added to those sample stations. S. Parker noted that zinc and copper were collected from two samples in that area in 1993 and 1997. However, the Navy agreed to consider this request.

The physical data points were discussed, and it was requested that at least one additional sample be included at DSY 103 (mislabeled on the schematic as DSY 104), and the existing station between DSY 103 and DSY 27 be moved closer to DSY 27. It was noted that TOC and grain size sample analysis is included in the physical data analysis and was inadvertently left off the attached plan.

Actions:

1. **Navy to provide meeting notes and consider the requests stated within.**
2. **Navy to provide a revised sampling plan and schematic.**
3. **Navy to consider incorporation of requests and move ahead with a Sampling and Analysis Plan.**

Meeting adjourned at 1135 AM.

Conceptual Sampling Plan
Data Gaps Investigation, Site 19: Former Derecktor Shipyard Marine Sediment
Newport RI

Data Quality Objectives

Data will be collected to support remedial decision-making in portions of the study area that are affected by site related contaminants. Data will be adequate to support remedy selection and design. These actions could include, but may not be limited to: removal (dredging), cover (full cap or thin layer cap), monitored natural recovery, and/or institutional controls. These actions will be fully evaluated in either an EE/CA or FS.

- It was previously determined that contaminants present in the sediment pose risk to human and ecological receptors (SAIC 1997). Based on these calculated risks, "Baseline Preliminary Remediation Goals" (BPRGs) were developed based on a HQ of 1.0, as published in SAIC, 1998. The following "Recommended" PRGs (same document) will be used to guide the sampling, determine where remediation is necessary, and to determine where remediation is complete.

  - Lead 168 ug/g dry weight
  - Benzo(a)pyrene 539 ng/g dry weight
  - HMW PAHs 13,903 ng/g dry weight
  - Total PCBs 1,060 ng/g dry weight

- In accordance with EPA request, secondary constituent of interest will be sought:

  - TBT (NOEC) 228 ng/g dry weight

- In certain target areas, other specific analytes will also be sought:

  - Asbestos (under Pier 1) 1%
  - Copper (under former carriers and piers) 74 ug/g dry weight
  - Zinc (under former carriers and piers) 118 ug/g dry weight

The remainder of "Baseline PRGs" that were not selected for use to establish remediation areas included the constituents below:

o,p'-DDE –
Estimated to pose risk to amphipods from suspended sediment modeled through an elutriate test, however baseline PRG was only exceeded in historical data at one sample station, not considered a contaminant that originated at the site.

Arsenic –
Estimated to pose risk to human health through ingestion of shellfish and lobster, however most arsenic in shellfish is present in a non toxic organic form (SAIC 1998, pp 30-31).
Estimated to pose risk to avian predators, however data did not show arsenic above the BPRG level for this receptor.

Estimated to pose risk to amphipods from suspended sediment modeled through an elutriate test, however because locations of high risk did not match the locations of high concentrations, this PRG was not recommended for implementation

Silver -
Estimated to pose risk to avian predators, however data did not show silver above the BPRG level for this receptor.

Approach for Data Collection:

To determine where remedial action is warranted, the following approach is proposed:

1. A grid approach will be utilized to assign sample areas and designations.
2. Areas adjacent to and shoreward of sample stations where PRGs were exceeded in 2004 sediment sampling efforts will be sampled on a 100 foot grid. One core will be collected at the approximate center of each grid cell.
3. The area around sample station DSY 28 and shoreward will also be sampled on a 100 foot grid density. One core will be collected at the approximate center of each grid cell.
4. The area in the vicinity of the former Derecktor operations where recent (2004) sampling did not show any PRG exceedances will be sampled on a 200 foot grid. One core will be collected at the approximate center of each grid cell. This will include the area around DSY 22, DSY 30 and the location of DSY 18, in accordance with various regulator requests November 22 and December 14.
5. Samples will be collected from each core as described above from three intervals: 0-1 foot depth, 1-2 foot depth, and 2-4 foot depth.
6. No samples will be composited; all will be discrete samples.

Decision Points

The chemical data from the proposed grid network will be used to contour contaminant concentrations. If it is appropriate, the discrete data points may be combined into decision units, but this will be determined after the data are collected and evaluated.

Other decision points will be developed during preparation of the revised FS or EE/CA.

Chemical Data Analysis

Chemical analysis shall be limited to the eight analytes listed above at various stations:

- Four RPRGs (Sum HMW PAHs, benzo(a)pyrene, total PCBs, and lead) will be analyzed at all stations.

- Zinc and Copper analysis will be limited to the areas under Piers and under the Saratoga / Forrestal berthing locations.
- Asbestos analysis will be added to all samples associated with Pier 1.
- By previous agreement TBT analysis will be included at all sample stations. TBT data will be compared to the No Observed Effects Concentration (NOEC) of 228 ug/kg established November 2008, which was agreed to be used as a screening value.

Physical Data Collection

A multibeam sonar survey will be conducted within an area beginning approximately 200 feet north of Pier 2, and extending south to the CCRF surface discharge outfall (small peninsula to the south of the site) including the area between Piers 1 and 2 and extending from the eastern shore to the west end of the piers. This survey will be compared to the survey conducted in 2007 and anomalies noted. Data will be used to identify potential depositional areas, scour areas, and overall changes in depth over time.

The hydrographic survey conducted by URI in 1996 will be augmented by deployed ADCP profilers during a 15 day period. Deployments at nine stations near and around piers will assist in determination of strength of fluctuating and short term currents at the sediment surface. Stations were selected based on physical features that may create transitions at the site and coverage of the different areas of interest:

1. North side of Pier 2
2. South side of Pier 2
3. Inshore area between Piers 1 and 2
4. North side of Pier 1
5. South Side of Pier 1
6. DSY 28 area, near outfall, between Pier 1 and small stone breakwater
7. South of Stillwater Area
8. Between Stations DSY22 and DSY30
9. Outer cove (reference area)

Sediment stability testing is proposed for the same nine locations. Stability testing is proposed to be performed by ERDC Coastal and Hydraulics Laboratory, Vicksburg MS. The method to be employed will be a cohesive sediment erosion flume, proposed and planned for use at Gould Island (IR Site 17) during the same period of time. This data will provide information as to the current velocity required to move sediments from different depths within the sediment strata. Duplicate cores will be collected at these stations for grain size analysis and TOC for sediment.

Radioisotope analysis of cores is proposed to be conducted at the nine stations where the flume testing is being done. Analysis would include lead 210 which has a steady decay rate and a half life of 22 years, as well as a relatively steady deposition rate that should not be impacted by the presence of lead contaminants from site discharge. Radioisotope analysis will be used as one indicator to supplement other data collected at this site.

References:

SAIC 1998, Preliminary Remediation Goals for Derecktor Shipyard, Coddington Cove Prepared under Contract to Tetra Tech NUS, for US Navy Northern Division. Final. November 1998.

SAIC and Univeristy of Rhode Island, Ecological Risk Assessment for Derecktor Shipyard, Coddington Cove. Prepared under Contract to Tetra Tech NUS, for US Navy Northern Division. Final. May 1997.

Tetra Tech NUS, Inc. 1998, Marine Human Health Risk Assessment, Offshore Areas of the Former Robert E. Derecktor Shipyard, NETC Newport September, Final, 1998

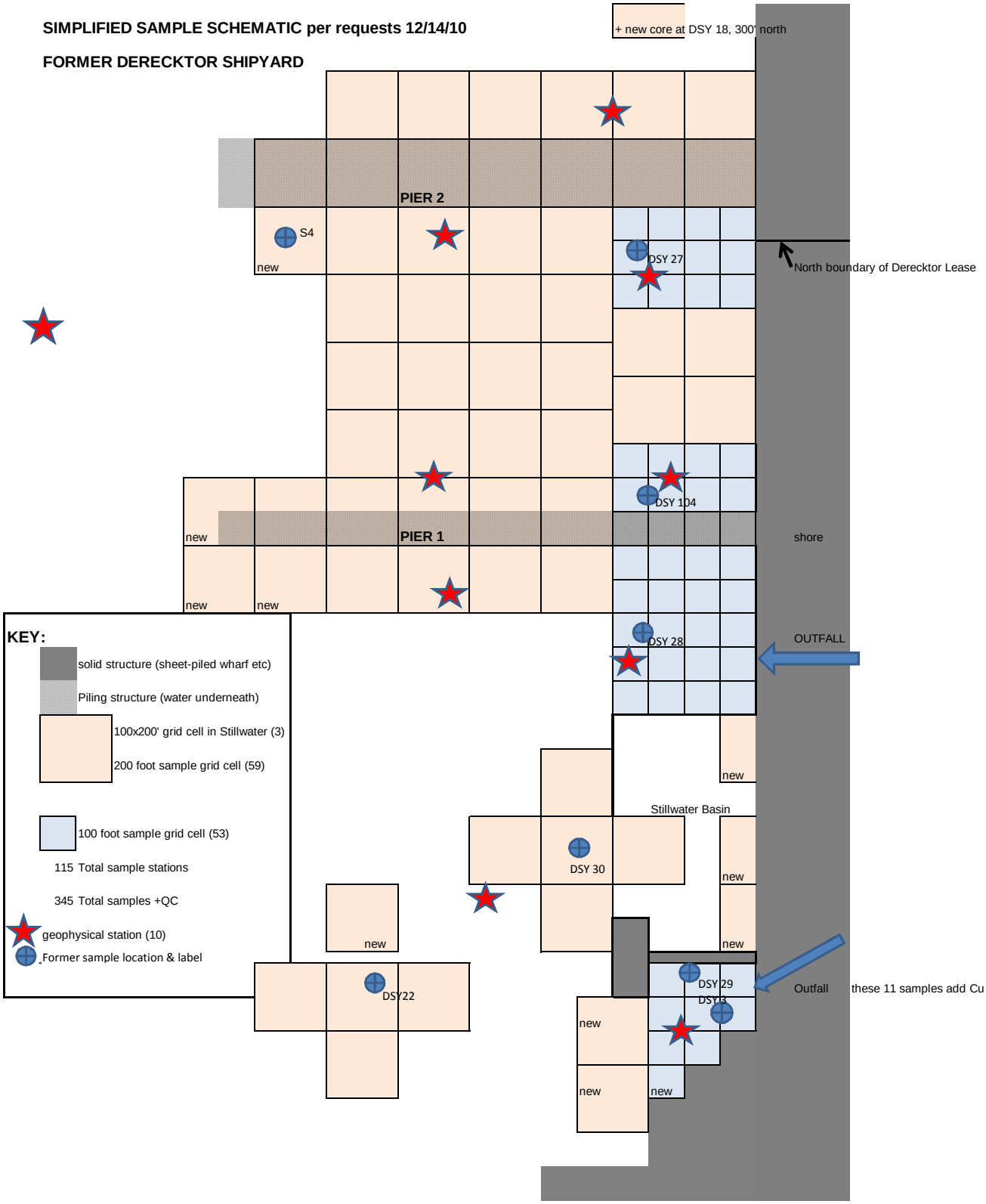
Kincaid, et. al. 1996. Hydrographic Survey for the Offshore Ecological Risk Assessment at Derecktor Shipyard / Coddington Cove, Naval Education and Training Center, Newport RI. June.

Tetra Tech 2008. Summary of TBT at Site 19, Derecktor Shipyard, Naval Station Newport, Newport RI. Revised November 7, 2008

NFESC, 1998. Deep Draft Berthing Facility Site Plan, Naval Facilities Engineering Service Center, East Coast Detachment. Washington DC. July.

SIMPLIFIED SAMPLE SCHEMATIC per requests 12/14/10

FORMER DERECKTOR SHIPYARD



APPENDIX D

Analytical Specifications

D-1 Katahdin

ATTACHMENT NO. 2

STATEMENT OF WORK/PRICE TABLES

TECHNICAL SPECIFICATION FOR LABORATORY SERVICES
DERECKTOR SHIPYARD, SITE 19
NAVAL STATION NEWPORT, NEWPORT, RHODE ISLAND

COMPREHENSIVE LONG-TERM ENVIRONMENTAL ACTION - NAVY (CLEAN)
CONTRACT N62470-08-D-1001, CONTRACT TASK ORDER (CTO) NO. WE61

DATA GAPS INVESTIGATION – MARINE SEDIMENT
CHEMICAL & PHYSICAL ANALYSES

1.0 INTRODUCTION

Tetra Tech NUS, Inc. (TtNUS) under CLEAN Contract N62470-08-D-1001, is procuring laboratory analytical services to support a Data Gaps Investigation at Naval Station Newport (NAVSTA), off-shore portions of Derecktor Shipyard, Site 19. Requested analyses include high molecular weight (HMW) polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), select metals, total organic carbon (TOC), grain size, and asbestos.

The laboratory performing the chemical analyses must provide a copy of its Department of Defense (DOD) Environmental Laboratory Accreditation Program (ELAP) accreditation letter; the scope of the ELAP accreditation must include all chemical methods and all analytes requested. The laboratory performing the asbestos analysis must participate in the in the National Voluntary Laboratory Accreditation Program (NVLAP).

The responding laboratory must submit limits of quantitation (LOQs), limits of detection (LODs), and detection limits (DLs) for all analyses and matrices requested by filling out the last three columns of the tables in Attachment A and including the completed attachment with the bid response.

After subcontract award, the laboratory will be required to submit Standard Operating Procedures (SOPs) and relevant precision and accuracy limits for all preparation and analytical methods required under this scope of work. The laboratory will also be asked to complete Uniform Federal Policy (UFP) Sampling and Analysis Plan (SAP) Worksheets 19, 24, 25, and 28 for inclusion in the SAP. The SAP will be prepared according to the UFP for Quality Assurance Project Plans (March 2005) and will utilize the 37 UFP-SAP worksheets.

2.0 SAMPLE INFORMATION

The approximate number of samples to be submitted, the type of analyses to be conducted, and the analytical methods to be used are summarized in Table 1. This investigation includes analysis of marine sediment samples and associated aqueous rinsate blanks.

The sampling is scheduled for June/July, 2011. The exact starting date of sample collection will be communicated to the laboratory at least 2 weeks in advance.

The samples are expected to be of low or moderate contaminant concentration except that because the sediment samples will be marine sediment, they may contain high concentrations of sodium, calcium, and magnesium. The field crew will attempt to identify any potentially high concentration samples and mark as such on the chain-of-custody forms.

Field duplicate samples will be submitted to the laboratory with "blinded" identification. The field crew will designate one per twenty sediment samples for matrix spike/matrix spike duplicate (MS/MSD) analysis (for organics) or matrix spike/laboratory duplicate (for metals). Additional volumes of these samples will be provided as necessary.

Percent Moisture

For the sediment analysis, the laboratory must decant any standing water, homogenize the sample, and determine the percent moisture before sample analysis. The sample aliquots must be increased to compensate for the moisture content of the samples or the sample may be centrifuged to eliminate more water. **The project LOQ goals listed in Attachment A must be met regardless the moisture content of the sediment samples**.

If the percent moisture content is too high and the sediment samples contain noticeable organic material, further dewatering must be performed at the laboratory prior to sample analysis. This could be accomplished by freeze-drying under controlled conditions proven to recover the analysis-specific target compounds; centrifugation and decanting free water; low temperature oven drying (below 60°C); or other procedure proposed by the laboratory and approved by Tetra Tech.

Metals Analysis

For the metals analyses, perform a matrix spike and a laboratory duplicate analysis instead of matrix spike/matrix spike duplicate. If a metals matrix spike recovery falls outside the control limits and the sample result does not exceed four times the spike added, a post-digestion spike analysis must be performed. In addition, a five-fold dilution analysis (ICP serial dilution) must be performed on the field-designated QC sample. Results for these QC analyses must be summarized in the corresponding CLP-equivalent forms (Attachment B).

PCB Analysis

All positive identifications for PCBs by gas chromatography (GC) with electron capture detector (ECD) MUST be confirmed on a second column that possesses retention characteristics different from those exhibited by the first column. Identification using a single column with dual detectors does not meet second column confirmation requirements. Confirmed positive results less than the laboratory LOQ but greater than the DL must be reported by the laboratory; the laboratory must “J” flag these results. The higher of two results generated from dual column analyses must be reported by the laboratory unless relative percent differences (RPDs) between columns are greater than 40% and interference is found to be present or there is some other justifiable reason to select the lower of the two results. The laboratory must narrate the reason for selection of the lower result.

Asbestos Analysis

Asbestos shall be analyzed by ASTM Method for Asbestos in Soil; Polarized Light Microscopy (PLM) Quantitative with Transmission Electron Microscopy (TEM) Qualitative. The laboratory shall quantify the percentage of asbestos in the sediment down to 1 percent (%) using PLM. If asbestos is not observed at or above 1% by PLM, the laboratory shall conduct a qualitative test for the presence of asbestos by TEM.

3.0 ANALYSIS/REPORTING INFORMATION

One hard copy data package deliverable and two PDF CD copies must be submitted, in addition to the electronic data deliverables to be provided in the format described in Attachment C. The original chain-of-custody form received with the samples and signed by the laboratory sample custodian must be returned with the hard copy data package.

The analytical requirements and approximate number of samples to be submitted for analysis are detailed in Table 1. Analysis and reporting requirements addressed in the Department of Defense (DOD) Quality Systems Manual (April 2009) and the requested methods must be followed. Additionally, it is a requirement of TtNUS that the associated PDF and hard copy data packages for PAH, PCB, and metal analyses must meet Contract Laboratory Program (CLP) format, reporting, and PDF/hard copy data package deliverable requirements. The PDF and hard copy data packages for the TOC analyses must be in a CLP-modified format and must include the appropriate summary forms and raw data for all samples and laboratory quality control samples. The summary forms should include the method-specific quality control limits (recoveries, relative percent differences, relative standard deviations, and/or percent differences, etc.). Second-source initial calibration verification results must be reported on a summary form.

The PDF and hard copy data packages for the grain size and asbestos analyses must contain raw data and appropriate summary forms for all samples and laboratory quality control samples.

The TtNUS sample identification numbers **must be included** on the raw data and summary forms.

Additionally, each hard copy and PDF data package must contain a summary data package. This summary data package shall consist of only the summary forms (i.e., the CLP-like equivalent of Forms 1 through 15).

Attachment A details the required target analyte list and LOQ goals that must be met. The laboratory must submit its sediment LOQs, LODs, and DLs for all chemical analyses requested by filling out the last three columns of the tables in Attachment A and including the completed attachment with the bid response. If the LOQ goal for a target compound is not technically achievable, the laboratory should propose the lowest LOQ technically possible for the requested method. Asbestos must be quantified to 1%.

Attachment B details the required summary forms for CLP-like data packages and requirements for organization/bookmarking of hard copy/PDF data packages.

Non-detected organic and metal results must be reported down to the laboratory's LODs. Positive results above the DL but below the laboratory's LOQ must be reported as estimated values qualified with a "J" **for both organic analyses and metal** analyses. Sediment samples must be reported on a **dry-weight basis**.

The hard copy/PDF data package deliverable must contain a detailed case narrative for all analytical fractions. This case narrative must also include the Contract Task Order (CTO) number, the site name, and the TtNUS Project Manager's name. Data from all analytical runs (i.e., original, dilution, re-analysis) must be reported in the raw data and Form Is for organic analyses. For metal analyses, only the final sample results should be reported in the Form Is, and data from all analytical runs must be included in the raw data.

As part of the laboratory case narrative, it is required that the Laboratory Quality Assurance Manager sign an attestation statement verifying that all electronic diskette deliverables exactly match the data summary forms (i.e. Form Is).

As stipulated in the CLEAN Basic Ordering Agreement (BOA), Sample Delivery Group (SDG) and fractionally-specific text (TXT) files containing all environmental sample and field quality control blank analysis results must be generated in accordance with the requirements outlined in Attachment C of this specification.

Maximum holding time allowances, as defined in the following table, are to be strictly observed. Calculation of holding time is in calendar days and is to begin from the time of sample collection. The holding times are as follows:

Analyses	Preservation	Holding Time
PAHs	Aqueous: Cool to ≤ 6 °C	7 days to extraction, 40 days to analysis
	Sediment: Cool to ≤ 6 °C	14 days to extraction, 40 days to analysis
PCBs	Aqueous and Sediment: Cool to ≤ 6 °C	30 days to extraction, 40 days to analysis
Metals (lead, copper, zinc)	Aqueous: HNO_3 to pH < 2	6 months to analysis
	Sediment: Cool to ≤ 6 °C	
TOC	Sediment: Cool to ≤ 6 °C	28 days to analysis
Grain Size	Not specified	Not specified
Asbestos	Not specified	Not specified

These holding times are based on 40 CFR 136, data validation criteria, and method specific requirements, and are measured from date of collection for samples preserved as requested in the analytical methods. The holding time criteria depicted apply to all analyses necessary to successfully determine the contaminant level contained in the sample. Hence, **the holding time criteria apply to any/all subsequent sample dilutions and re-analyses**.

The TtNUS Project Manager for this project is Mr. Stephen Parker and he must be contacted in the event of any laboratory problems that could impact project deadlines (i.e., late deliverables, technical problems in the lab that could lead to late deliverables.) To ensure good communication, it is required that the laboratory's appointed project manager contact Mr. Parker once a week for the entire project duration.

Contact information for Mr. Parker is as follows:

Tetra Tech NUS
250 Andover Street, Suite 200
Wilmington, MA 01887
Phone: 978-474-8434
Fax: 978-474-8499
Email: stephen.parker@tetratech.com

Technical, quality assurance, and data format concerns are to be directed to Ms. Lucy Guzman at 978-474-8416 or via e-mail lucy.guzman@tetratech.com. Ms. Guzman must be contacted and informed of any difficulties encountered during the conduct of the requested analyses.

Analytical data turnaround times are to be measured from receipt of each sample shipment. All hard copy/PDF (2 CDs) analytical data packages and associated electronic (TXT) deliverables are due within the standard BOA turnaround term of 21 calendar days from receipt of the last sample in a Sample Delivery Group (SDG).

The SDGs shall contain no more than 20 samples. The frequency with which SDGs contain fewer than 20 samples should be minimal. The hard copy data packages, PDF files (CDs), and electronic deliverables must be received at the same time or the deliverable will be considered incomplete and payment deductions may be imposed.

The hardcopy analytical data package, one PDF (CD) copy of the analytical data package, and the original chain-of-custody form (received with the samples and signed by the laboratory sample custodian) shall be sent to Ms. Lucy Guzman.

The electronic (TXT) deliverables, one PDF (CD) copy of the analytical data package, and a copy of the chain-of-custody form, should be sent to Ms. Tobrena Sedlmyer. The contact information for Ms. Sedlmyer is as follows:

Tetra Tech NUS, Inc.
661 Andersen Drive, Foster Plaza 7
Pittsburgh, PA 15220-2745
Phone: 412-921-8582
Fax: 412-921-4040
e-mail: tobrena.sedlmyer@tetratech.com

4.0 PERIOD OF PERFORMANCE/BOTTLEWARE INFORMATION

All samples will be shipped to the laboratory via express carrier within 48 hours of collection. **The laboratory must be capable of receiving samples on Saturdays.** Please circle the Yes or No at the bottom of Table 1 which will indicate if the laboratory will provide courier service from the collection site in Newport, Rhode Island at no extra charge. The laboratory will be notified at least 2 weeks prior to sample collection.

Bottleware shipments will be coordinated by the field operation leader.

The laboratory shall provide all necessary sample containers **(plus approximately 10% extra for breakage)**. All sample containers must meet ICHEM series 300 cleanliness criteria (or equivalent), and documentation of certified cleanliness must be provided. All of the appropriate sample bottleware must be pre-preserved. The bottleware must be shipped to the designated location in Coleman-like coolers. Each cooler must include a “temperature blank” vial. **The laboratory must also provide any extra coolers needed for return shipment of samples to the laboratory for analysis.** The laboratory is also requested to provide a packing slip indicating the analytical parameters for which each container type is designated, sample labels, and chain-of-custody forms.

The laboratory must provide Material Safety Data Sheets (MSDSs) for all preservatives sent with each bottleware shipment to the field. MSDSs must be representative of the chemicals provided as preservatives with regard to mixtures and/or purity of the chemicals. For example if a 35% sulfuric acid solution is the preservative, the MSDS provided should be for 35% sulfuric acid solution not 96% sulfuric acid.

5.0 ADDITIONAL COMMENTS/CONTACTS

Within the laboratory, the internal transfer of samples, extracts, and digestates must be accomplished and documented as controlled custody transfers. The laboratory must submit the documentation that supports an unbroken chain of custody for samples, digestates and extracts from time of receipt or production in the laboratory until disposal.

The laboratory is to provide a minimum of 60 days storage of sample extracts/digestates and 60 days storage of intact leftover sample aliquots, as stipulated in the BOA. **Additionally, the laboratory must store PDF data packages for 7 years.**

All analyses conducted under this subcontract assignment are to be performed at the solicited facility only. The laboratory is not permitted to lower-tier subcontract these analyses, or analyze these samples at a corporate facility other than the facility stipulated without prior notification and consent from the CLEAN Subcontracting Officer.

The unit cost for analysis is to include compensation for containers, preservatives, coolers, shipping costs, storage, disposal, and laboratory quality control analyses (such as matrix spike, matrix spike duplicate, laboratory duplicate, and laboratory control sample analyses.) These items are not to be billed as separate line items.

Contract concerns, and response to this solicitation, are to be directed to:

Ms. Meg Price
CLEAN Subcontracting Officer
Tetra Tech NUS, Inc.
234 Mall Boulevard, Suite 260
King of Prussia, PA 19406-1433
Phone: 610-382-1525
Fax: 610-491-9645
e-mail: meg.price@tetratech.com

Triplicate copies of invoices associated with the analyses contracted herein are to be submitted to the attention of the Accounting Supervisor:

Tetra Tech NUS, Inc.
661 Andersen Drive, Foster Plaza 7
Pittsburgh, PA 15220
Phone: 412-921-8506
Fax: 412-921-4040

Please confirm the laboratory's ability to perform the methodologies requested at the analyte quantitation limits indicated. Also confirm available laboratory capacity, and complete/confirm the

costing information indicated in Table 1. All costing information must reflect the terms and conditions established by the 2010 CLEAN BOA.

TABLE 1
NUMBER OF SAMPLES/ANALYTICAL METHODS
CTO WE61, OFF-SHORE DERECKTOR SHIPYARD
NAVSTA NEWPORT, NEWPORT, RI
DATA GAPS INVESTIGATION, CHEMICAL & PHYSICAL ANALYSES

Matrix	Parameter	Method	# Samples	Unit Price	Total Cost
Sediment	HMW PAHs(1)	SW-846 8270D or 8270D SIM(2)	380	$	$
	PCBs(1)	SW-846 8082A	380	$	$
	Lead	SW-846 6010C	277	$	$
	Lead, Copper, and Zinc	SW-846 6010C	103	$	$
	TOC	Lloyd Kahn	11	$	$
	Grain Size	ASTM D422-63	11	$	$
	Asbestos	ASTM Method for Asbestos in Soil; Polarized Light Microscopy (PLM) Quantitative with Transmission Electron Microscopy (TEM) Qualitative	33	$	$
Aqueous (Rinsate Blanks Associated with Sediment Samples)	HMW PAHs(1)	SW-846 8270D SIM	18	$	$
	PCBs(1)	SW-846 8082A	18	$	$
	Lead	SW-846 6010C	13	$	$
	Lead, Copper, and Zinc	SW-846 6010C	5	$	$

(1) See list of required target analytes in Attachment A. (2) Laboratory to indicate whether full scan or SIM mode will be used in order to meet LOQ goals.

TOTAL COST $

The laboratory must state if they are not DOD ELAP accredited for all of the methods and analytes requested. Clearly state the methods and or compounds that you are NOT accredited for (if any).

Can the laboratory provide sample pick-up on site? YES or NO (circle one)
If yes is there a charge and what is that charge?_____

Name of Laboratory_____

Signature_____

2/16/11
Revision 0
AF

ATTACHMENT A

REQUIRED TARGET ANALYTE LISTS AND PROJECT ACTION LIMITS

ATTACHMENT A

REQUIRED TARGET ANALYTE LISTS AND PROJECT ACTION LIMITS
CTO WE61, OFF-SHORE DERECKTOR SHIPYARD, SITE 19
NAVAL STATION NEWPORT, NEWPORT, RHODE ISLAND
DATA GAPS INVESTIGATION, CHEMICAL & PHYSICAL ANALYSES

Matrix: Sediment
Analytical Group: High Molecular Weight (HMW) PAHs, analyzed by SW-846, Method 8270D or 8270D SIM[1]

Analyte	CAS Number	PAL (µg/kg)	PAL Reference	Project LOQ Goal (µg/kg)	Lab Limits[2]		
					LOQ (µg/kg)	LOD (µg/kg)	DL (µg/kg)
HMW PAHs							
Benzo(a)anthracene	56-55-3	--	--	495			
Benzo(a)pyrene	50-32-8	539	RPRG	180			
Benzo(b)fluoranthene	205-99-2	--	--	495			
Benzo(g,h,i)perylene	191-24-2	--	--	495			
Benzo(k)fluoranthene	207-08-9	--	--	495			
Chrysene	218-01-9	--	--	495			
Dibenzo(a,h)anthracene	53-70-3	--	--	495			
Fluoranthene	206-44-0	--	--	495			
Indeno(1,2,3-cd)pyrene	193-39-5	--	--	495			
Pyrene	129-00-0	--	--	495			
Total HMWPAHs[3]	--	13903	RPRG	--	--	--	--

Matrix: Sediment
Analytical Group: PCBs, analyzed by SW-846, Method 8082A

Analyte	CAS Number	PAL (µg/kg)	PAL Reference	Project LOQ Goal (µg/kg)	Lab Limits[2]		
					LOQ (µg/kg)	LOD (µg/kg)	DL (µg/kg)
PCBs							
Aroclor-1016	12674-11-2	--	--	50			
Aroclor-1221	11104-28-2	--	--	50			
Aroclor-1232	11141-16-5	--	--	50			
Aroclor-1242	53469-21-9	--	--	50			
Aroclor-1248	12672-29-6	--	--	50			
Aroclor-1254	11097-69-1	--	--	50			
Aroclor-1260	11096-82-5	--	--	50			
Total PCBs[4]	--	1060	RPRG	--	--	--	--

Notes:
1. Laboratory to identify whether full scan or SIM mode will be used to meet LOQ goals.
2. If the Project LOQ Goal cannot be met, laboratory shall enter its lowest limits technically achievable for the requested method.
3. TtNUS will calculate Total. Laboratory reports only individual PAHs.
4. TtNUS will calculate Total. Laboratory reports only individual Aroclors.

ATTACHMENT A

REQUIRED TARGET ANALYTE LISTS AND PROJECT ACTION LIMITS
CTO WE61, OFF-SHORE DERECKTOR SHIPYARD, SITE 19
NAVAL STATION NEWPORT, NEWPORT, RHODE ISLAND
DATA GAPS INVESTIGATION, CHEMICAL ANALYSES

Matrix: Sediment
Analytical Group: TOC, analyzed by Lloyd Kahn

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	LOQ Goal (mg/kg)	Lab Limits[1]		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
TOC	--	--	--	--			

Matrix: Sediment
Analytical Group: Metals, analyzed by SW846, Method 6010C

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	LOQ Goal (mg/kg)	Lab Limits[1]		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Metals							
Copper	7440-50-8	74	RPRG	25			
Lead	7439-92-1	168	RPRG	56			
Zinc	7440-66-6	118	RPRG	39			

Notes:
1. If the Project LOQ Goal cannot be met, laboratory shall enter its lowest limits technically achievable for the requested method.

ATTACHMENT B
DATA PACKAGE DELIVERABLES REQUIREMENTS

DATA PACKAGE DELIVERABLE REQUIREMENTS

The laboratory is to provide a hard copy plus two compact disks (CDs) each containing a PDF file in the following format:

1. Table of Contents
2. Case Narrative
3. Chain-of-Custody
4. Data Summary Package (contains summary of all CLP or CLP-like Forms 1 through 15 per analytical fraction)
5. Analytical Fractions (e.g., VOA, SVOC, Metals, etc.)
   a. Results and QC Summary (summary of all CLP or CLP like Forms 1 through 15 for a particular analytical fraction)
   b. Raw Sample Data (includes all sample dilutions, sample re-analyses, QC samples, etc.)
   c. Calibration Data (includes all initial and continuing calibrations and initial calibration verfication)
   d. Miscellaneous (includes extraction/preparation forms, percent solids determination, IDLs, MDLs, etc.)

Each of the above sections should be bookmarked in the PDF for easy access.

Summary Form Requirements for PDF data package deliverable for non-CLP Methods:

The following summary forms are required as part of the data package deliverable for SW-846 6020/6010B/7000A series for metals:

Results Report - must present all information presented on CLP FORM I (ILM05.4).
Initial and Continuing Calibration Summary - must present all information presented on CLP FORM 2A (ILM05.4).
Low-Level Calibration Standard Summary – if applicable, must present all information presented on CLP FORM 2B (ILM05.4).
Blanks - must present all information presented on CLP FORM 3 (ILM05.4).
ICP Interference Check Sample Summary - must present all information presented on CLP FORM 4 (ILM05.4).
Matrix Spike Summary - must present all information presented on CLP FORM 5A (ILM05.4).
Post Digestion Spike - must present all information presented on CLP FORM 5B (ILM05.4).
Lab Duplicate Results - must present all information presented on CLP FORM 6 (ILM05.4).
LCS Summary - must present all information presented on CLP FORM 7 (ILM05.4).
MSA Summary (Method of Standard Addition) – if applicable, must present all information presented on CLP FORM 8 (ILM04.1).
ICP Serial Dilution Summary - must present all information presented on CLP FORM 8 (ILM05.4).
Detection Limits - must present all information presented on CLP FORM 9 (ILM05.4).
Linear Range – must present all information presented on CLP FORM 11 (ILM05.4).
Internal Standard Association (ICP-MS) –must present all information presented on CLP FORM 11 (ISM01.2). Alternatively, the laboratory may provide the information in the Narrative.
Prep Log - must present all information presented on CLP FORM 12 (ILM05.4).
Analysis Run Log - must present all information presented on CLP FORM 13 (ILM05.4).
ICP-MS Tune – must present all information presented on CLP FORM 14 (ISM01.2). Laboratory must also document the number of tune analysis integrations on this form or in the Narrative.
ICP/MS Internal Standard Relative Intensity Summary - must present all information presented on CLP FORM 15 (ILM05.4).

<u>Also must include</u>: Instrument Calibration Records, Chain-of-Custody Forms, and Case Narrative. The Narrative, forms, or raw data must indicate the number of replicate integrations for ICP-MS sample analysis. Also, the compounds associated with internal standards must be identified.

Summary Forms for GC/MS analysis of PAHs should be presented in a CLP-Like format. The following Summary Forms must be included:

Result Summary	One Sample per summary page. Presentation of analytical results for both method blanks and environmental samples, date of collection, preparation, and analysis. Environmental samples should be identified with the field identification numbers on the COCs.
Surrogate Recovery Form	Present all information contained on CLP Form II.
Summary of Matrix Spike/Matrix Spike Duplicate Recovery	Present all information contained on CLP Form III.
Instrument Performance Check Summary Form - Mass Spec Tuning Form	Present all information Contained on CLP Form V.
Initial Calibration Summary	Present all information contained CLP Form VI.
Continuing Calibration Summary	Present All Information contained on CLP Form VII.
Internal Standard Area and Retention Time Summary	Present all information contained CLP Form VIII.

Also must include: initial calibration verification summary forms and identification of the compounds associated with internal standards.

Summary Forms for GC analysis of PCBs should be presented in a CLP-Like format. The following Summary Forms must be included:

Result Summary	One Sample per summary page. Presentation of analytical results for both method blanks and environmental samples, date of collection, preparation, and analysis. Environmental samples should be identified with the field identification numbers on the COCs.
Surrogate Recovery Form	Present all information contained on CLP Form II for both Analytical Columns.
Summary of Matrix Spike/Matrix Spike Duplicate Recovery	Present all information contained on CLP Form III.
Summary of Initial Calibration of Single Component Analytes (if applicable	Present all information contained on CLP Form VI-PEST-2.
Summary of Calibration Verification	Present all information contained on CLP Form VII-PEST-1 and Form VII-PEST-2.
Analytical Sequence	Present all information contained on CLP Form VIII-PEST.
Identification Summary For Single Component Analytes	Present all information contained on CLP Form X PEST 1 and 2.

and for Multiple Component Analytes

Also must include: initial calibration verification summary forms.

ATTACHMENT C
ELECTRONIC DATA DELIVERABLE REQUIREMENTS

ELECTRONIC DATA FORMAT REQUIREMENTS

1.0 **INTRODUCTION**

The laboratory is to submit text-based tab delimited EDD files for each SDG using Tetra Tech's laboratory data checker explained below. The files must be in the format specified in this Attachment. Additional information such as laboratory name, project name, fractions included, project number, site name/number, laboratory contact person and any specific comments related to the EDD should be included in the comments section of the EDD Submittal page.

The RESULT for nondetects should be populated with the project-specific sample quantitation reporting limits (i.e., either the sample quantitation limit or method detection limit, as specified in Section 3.0 of this scope of work. Any corrections made to the hardcopy data must also be made to the electronic file. Appropriate qualifiers as identified by the analytical protocol must also be designated; laboratory QC non-compliance codes are not to be depicted.

Tetra Tech's electronic EDD format follows the ADAPT structure and requires the A1 and A3 files. The A2 file is only required if the project is using ADAPT; and, for non-ADAPT EDD submittals the A2 file may be omitted. The EDD consists of separate, tab-delimited ASCII text files. Each file corresponds to a database table. The tables are identified as the Analytical Results Table (A1) and Sample Analysis Table (A3). A separate set of text files must be created and submitted for each sample delivery group (SDG). The files must be identified to correspond to the (A1) table and the (A3) table. The file naming convention is: the Sample Delivery Group (SDG) followed by the table identifier (A1 or A3), followed by the ".txt" extension. The file names must not contain spaces or special characters. For example, the EDD file names for a laboratory-reporting batch identified as SDG001 would be as follows:

SDG001A1.txt
SDG001A3.txt

On certain projects Tetra Tech will utilize the ADAPT Electronic Data Validation software, which will require the laboratory to use the ADAPT electronic data deliverable checker software prior to submitting the files through Tetra Tech's laboratory data checker (this will be clearly specified in the Tetra Tech laboratory statement of work). The ADAPT checker software can be downloaded from Laboratory Data Consultants' web site: http://www.lab-data.com. For projects which Tetra Tech is using the ADAPT software, Tetra Tech will provide the laboratory with the project library. The laboratory is not permitted to modify the project library. ADAPT projects will require the laboratory to export all three checked files (A1, A2, and A3) from the ADAPT software and submit them through Tetra Tech's laboratory data checker. **ADAPT error logs generated must be included with the electronic PDF data validation package and cannot be submitted through the laboratory data checker.**

The values reported in the EDD text files must agree exactly with the final values reported on the PDF data package sample result summaries. The details of file naming conventions, data structure and data checker use are discussed below.

Analytical Results Table (A1 File)

The Analytical Results table contains analytical results and related information for target analytes in field samples and associated laboratory quality control samples (excluding calibrations and tunes). Field samples and laboratory method blanks must report a result record for each analyte reported within a method. Laboratory control samples (LCS and LCSD) and matrix spike samples (MS and MSD) must report a result record for every analyte specified as a spiked analyte in the laboratory statement of work. Table A1 in this document lists the field names and data type descriptions for the Analytical Results Table (A1).

Lab Instrument Table (A2 File)

A2 file is only required if the project is using ADAPT. In all other EDD submittals, the A2 file may be omitted. Laboratories should refer to the ADAPT User Guide for populating the A2 Table.

Sample Analysis Table (A3 File)

The Sample Analysis table contains information specific to field environmental samples and laboratory quality control analyses (excluding calibrations and tunes). A sample record must exist for each sample/method/matrix/analysis type combination. Table A3 in this document lists the field names and data type descriptions for the Sample Analysis Table (A3).

All electronic data deliverables are due within the same time established for the associated hardcopy data packages.

In addition, the laboratory QC officer must read and sign a copy of the Quality Assurance Review Form displayed on the next page of this Attachment. Electronic deliverables are not considered to be complete without the accompanying Quality Assurance Review Form.

I _____, as the designated Quality Assurance Officer, hereby attest that all electronic deliverables have been thoroughly reviewed and are in agreement with the associated hardcopy data. The enclosed electronic files have been reviewed for accuracy (including significant figures), completeness and format. The laboratory will be responsible for any labor time necessary to correct enclosed electronic deliverables that have been found to be in error. I can be reached at
(_____)_____if there are any questions or problems with the enclosed electronic deliverables.

Signature:_____ Title: _____Date: _____

2.0 EDD Field Properties

Tables A1 and A3 in this document specify the EDD field properties. Laboratories should refer to the ADAPT User Guide for populating the A2 Table. These include the field name, sequence order, field description, data type/length and reporting requirement for each field. Fields in the EDD **must** be sequenced according to the order that they appear below in Tables A1 and A3. For example, in the Analytical Results table (A1), the field "ClientSampleID" will always be the first piece of information to start every new line of data (or database record), followed by the field "LabAnalysisRefMethodID", "AnalysisType", etc.

When creating an EDD as a text file, use the ASCII character set in a file of lines terminated by a carriage return and line feed. No extra characters are allowed at the end of a line, after the carriage return and line feed. Enclose each data value with double quotes (text qualifier) and separate each field value with a **tab** character (tab delimiter). Data fields with no information (null) may be represented by two consecutive tabs. For example, in the Sample Analysis table, since the "Collected", "ShippingBatchID", and "Temperature" fields do not apply to laboratory generated QA/QC samples, the record for a Laboratory Control Sample by Method 8270C would be entered as follows. Note that the first two fields ("ProjectNumber" and "ProjectName") are omitted in this example.

…"LCSW100598" "AQ" "LCSW100598" "LCS" "8270C",…etc.

If a field is populated with less than the maximum allowed number of characters, do not pad the values with leading or trailing spaces. In the above example, although the "MatrixID" field can accommodate up to 10 characters, only 2 characters were entered in this field. **Do not include the delimiter (tab character) within any of the field values.** Example EDD files may be downloaded from the LEDD Checker application.

An example database shall be sent for review prior to the first electronic deliverable in the required .txt format. The example file will be examined for completeness and comments will be sent to the laboratory. Any questions regarding the electronic deliverable should be directed to LabSupport@tetratech.com

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
ClientSampleID	Client or contractor's identifier for a field sample If a sample is analyzed as a laboratory duplicate, matrix spike, or matrix spike duplicate, append suffixes DUP, MS and MSD respectively to the Client Sample ID	Text	25	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	with no intervening spaces or hyphens (i.e. MW01DUP, MW01MS, and MW01MSD). For Method Blanks, LCS, and LCSD enter the unique LaboratorySampleID into this field. Do not append suffixes to the ClientSampleID for dilutions, reanalyses, or re-extracts (the AnalysisType field is used for this distinction). For example, MW01DL and MW01RE are not allowed. Parent sample records must exist for each MS and MSD. If an MS/MSD is shared between two EDDs, records for the MS/MSD and its parent sample must exist in the Analytical Results table for both EDDs.			
LabAnalysisRefMethodID	Laboratory reference method ID. The method ID may be an EPA Method number or a Lab Identifier for a method such as a SOP Number, however; method ID is specified by the project. The method ID must be entered into the standard list.	Text	25	X
AnalysisType	Defines the analysis type (i.e., Dilution, Reanalysis, etc.). This field provides distinction for sample result records when multiple analyses are submitted for the same sample, method, and matrix; for example dilutions, re-analyses, and re-extracts.	Text	10	X
LabSampleID	Laboratory tracking number for field samples and lab generated QC samples such as method blank, LCS, and LCSD. There are no restrictions for the LabSampleID except for field length and that the LabSampleID must be distinct for a given field sample or lab QC sample and method.	Text	25	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	Suffixes may be applied to the LabSampleID to designate dilutions, reanalysis, etc.			
LabID	Identification of the laboratory performing the analyses.	Text	7	X
ClientAnalyteID	CAS Number or unique client identifier for an analyte or isotope. If a CAS Number is not available, use a unique identifier provided by the client or contractor. The ClientAnalyteID for a particular target analyte or isotope should be specified by the project and must exist in the standard value tables for Analytes. For the LCS, LCSD, MS, and MSD, it is only necessary to report the compounds designated as spikes in the library (and surrogates for organic methods.) For TICs from GC/MS analyses, enter the retention time in decimal minutes as the Client Analyte ID.	Text	12	X
AnalyteName	Chemical name for the analyte or isotope. The project specifies how an analyte or isotope is named. The analyte name must be associated to a ClientAnalyteID in the standard values table for Analytes (excluding compounds designated as TIC's).	Text	60	X
Result	Result value for the analyte or isotope. Entries must be numeric. For non-	Numeric (1)	20(6)	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	detects of target analytes or isotopes and spikes, do not enter "ND" or "0". Do not leave this field blank. If an analyte or spike was not detected, enter the associated value specified in Section 3.0 of this scope of work (e.g., LOD, SQL, PQL, etc.), corrected for dilution and percent moisture as applicable. Do not enter "0". A "0" result may be acceptable for surrogate or internal standard percent recoveries; however, it should not be reported for any target compound.			
ResultUnits	The units defining how the values in the Result, DetectionLimit, and ReportingLimit fields are expressed. For radiochemistry this also includes how the value in the Error field is expressed.	Text	10	X
LabQualifiers	A string of single letter result qualifiers assigned by the lab based on client-defined rules and values. <u>The "U" Lab Qualifier must be entered for all non-detects.</u> Other pertinent lab qualifiers may be entered with the "U" qualifier. Order is insignificant. Lab qualifiers other than those listed in the standard values table may be used. If so, these must be added to the standard value table in the application.	Text	7	Q
DetectionLimit	For radiochemistry methods, the minimum detectable activity for the isotope being measured. For all other methods: The minimum	Numeric (1)	10(6)	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	detection limit value for the analyte being measured. For surrogates, internal standards, etc. where detection limits are not applicable use the value -99.			
DetectionLimitType	Specifies the type of detection limit (i.e., MDA, MDL, IDL, etc.). If -99 is specified in the DetectionLimit field us the value NA.	Text	10	X
RetentionTime or Error	For radiochemistry methods only, enter the 2 Sigma Counting Errors. The units for error are entered in the ResultUnits field. For GC/MS methods only, enter the time expressed in decimal minutes between injection and detection for GC/MS TICs only For target analytes in all other methods, leave this field blank. Note: GC retention times are not evaluated at this time.	Text	5	T
AnalyteType	Defines the type of result, such as tracer, surrogate, spike, or target compound.	Text	7	X
PercentRecovery	For radiochemistry methods: The tracer yield, if applicable. For all other analytical methods: The percent recovery value of a spiked compound or surrogate. If the spike or surrogate was not recovered because of dilution, enter “DIL”. If a spike or surrogate was not recovered because of matrix interference, enter “INT”. If a spike or	Numeric (1)	5(3)	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	surrogate was not recovered because it was not added to the sample, enter “NS”.			
RelativePercentDifference	The relative percent difference (RPD) of two QC results, such as MS/MSD, LCS/LCSD, and Laboratory Duplicates. Report RPD in Laboratory Duplicate, LCSD, and MSD records only.	Numeric (1)	5(3)	X
ReportingLimit	Reporting limit value for the measured analyte or isotope Factor in the dilution factor and percent moisture correction, if applicable. The Reporting Limit for each analyte and matrix in a given method is specified in the project library or QAPP. For surrogates, internal standards, etc. where reporting limits are not applicable use the value -99.	Numeric (1)	10(6)	X
ReportingLimitType	Specifies the type of reporting limit (i.e., CRQL, PQL, SQL, RDL, etc). The Reporting Limit Type for each method and matrix is specified in the project library or QAPP. If -99 is specified in the ReportingLimit field us the value NA.	Text	10	X
ReportableResult	This field indicates whether or not the laboratory chooses an individual analyte or isotope result as reportable. Enter “YES” if the result is reportable. Enter “NO” if the result is not reportable. If only one analysis is submitted for a particular sample and method, enter “YES”	Text	3	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	for all target compounds (where Analyte Type = TRG). For GC/MS methods enter yes for tentatively identified compounds (where Analyte Type = TIC). If two or more analyses are submitted for a particular sample and method (i.e. initial analysis, reanalysis and/or dilutions), enter "YES" from only <u>one</u> of the analyses for each target compound. For example: a sample was run a second time at dilution because benzene exceeded the calibration range in the initial, undiluted analysis. All target analytes are reported in each analysis. For the initial analysis, (Analysis Type = RES), enter "NO" for benzene and enter "YES" for all other compounds. For the diluted analysis (Analysis Type = DL), enter "YES" for benzene and enter "NO" for all other compounds. For TICs (Analyte Type = TIC), if more than one analysis is submitted for a particular sample and method, choose only one of the analyses where Reportable Result = YES for <u>all</u> TICs. For example, a sample was run a second time because one or more target compounds exceeded the calibration range in the undiluted analysis. Choose a particular analysis and enter "YES" for all TICS. In the other analysis enter "NO" for all TICs. Note that it is not necessary to report the full target analyte list for the initial result, dilution, re-analysis, or re-extraction. However, each target analyte must be reported YES once and once only in the case of multiple analyses for a given sample, method, and			

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	matrix. In the case of organics, all surrogates must be reported for all analyses submitted for a given sample, method, and, matrix.			
SpkConcnAdded	The spike added. This value must be reported in the same units as the result. Where (SA) in the following equation: % Recovery = (SSA-SC)/SA x 100% where : SSA is the spiked sample concentration (amount) after spiking. SC is the sample concentration (amount) before spiking. SA is the the expected increase in sample concentration (amount) as a result of spiking. This value must incorporate all correction factors such as dilution factor and moisture content that are applied to the spiked sample when computing the spiked sample concentration or amount. Enter -99 where no spike was added.	Numeric (1)	10(6)	X
SpkParentSampleID	The sampleID of a sample (often called the original sample) that receives a spike aliquot to form a spiked sample such as a matrix spike. This is not the same as the ID of the spiked sample (such as a matrix spike) after spiking. The result for SpkParentSampleID and the result (i.e., SpkConcnAdded) for the spiked sample are used to compute percent recovery of the analyte.	Text	25	X
SamplePrepInitial	The initial sample preparation volume in liters (L) for aqueous samples or grams (g) for solid samples.	Numeric (1)	20(6)	

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
SamplePrepFinal	The final sample preparation volume in liters (L) for aqueous samples or grams (g) for solid samples.	Numeric (1)	20(6)	
LimitOfDetection	The smallest amount or concentration of a substance that must be present in a sample in order to be detected at a 99% confidence level. In other words, if a sample has a true concentration at the LOD, there is a minimum probability of 99% of reporting a "detection" (a measured value ≥ DL) and a 1% chance of reporting a non-detect (a false negative).	Numeric (1)	10(6)	N
Comment	Add any comments or additional information specific to the analyte test result data record.	Text	200	

X Required field.
Q Only required if laboratory has qualified the result.
T Only required for tentatively identified compounds by GC/MS.
(1) Field Length indicates decimal precision in parenthesis. For example, 5(2) = a total width of 5 numbers including a maximum of 2 decimal places.

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
ProjectNumber	Project number assigned by the client.	Text	30	X
ProjectName	Project name assigned by the client.	Text	90	X
ClientSampleID	Client or contractor's identifier for a field sample If a sample is analyzed as a laboratory duplicate, matrix spike, or matrix spike duplicate, append suffixes DUP, MS and MSD respectively to the Client Sample ID with no intervening spaces or hyphens (i.e. MW01DUP, MW01MS, and MW01MSD). For Laboratory QC samples (i.e. Method Blanks, LCS, and LCSD, etc.) enter the unique LaboratorySampleID into this field Do not append suffixes to the ClientSampleID for dilutions, reanalyses, or re-extracts (the Analysis_Type field is used for this distinction). For example, MW01DL and MW01RE are not allowed Parent sample records must exist for each MS and MSD. If an MS/MSD is shared between two EDDs, records for the MS/MSD and its parent sample must exist in the Sample Analysis table for both EDDs.	Text	25	X
Collected	Date and Time of sample collection. Refer to the date/time format at the end of this table. Leave this field blank for Method Blank, LCS, and LCSD. For Collected values that are not applicable use the value of 00/00/0000 00:00.	Date/ Time	16*	X
MatrixID	Sample matrix (i.e., AQ, SO, etc.)	Text	10	X
LabSampleID	Laboratory tracking number for field samples	Text	25	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	and lab generated QC samples such as method blank, LCS, and LCSD. There are no restrictions for the LabSampleID except field length and that the LabSampleID must be unique for a given field sample or lab QC sample and method.			
QCType	This record identifies the type of quality control sample QC (i.e., Duplicate, LCS, Method Blank, MS, or MSD). For regular environmental samples, populate this field with "NM".	Text	10	X
ShippingBatchID	Unique identifier assigned to a cooler or shipping container used to transport client or field samples. Links all samples to a cooler or shipping container. No value is required for method blanks, LCS, and LCSD.	Text	25	X
Temperature	Temperature (in centigrade degrees) of the sample as received. The storage refrigerator or room temperature should be reported (in centigrade degrees) for laboratory QC samples (i.e. method blanks, laboratory control standards). Use -99 if temperature is not available. This field is not required for radiochemistry methods.	Numeric (1)	10(6)	X
LabAnalysisRefMethodID	Laboratory reference method ID. The method ID may be an EPA Method number or laboratory identifier for a method such as a SOP number, however; values used for Laboratory Method IDs are specified by the	Text	25	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	project and must in the in standard value list for method IDs.			
PreparationType	Preparation Method Number (i.e., 3010A, 3510C, 3550C, 5030B, etc.) For analytical procedures that do not have a specific preparation method number, use “Gen Prep”.	Text	25	X
AnalysisType	Defines the type of analysis such as initial analysis, dilution, re-analysis, etc. This field provides distinction for sample records when multiple analyses are submitted for the same sample, method, and matrix, for example: dilutions, re-analyses, and re-extracts.	Text	10	X
Prepared	Refer to the date/time format at the end of this table. If no sample preparation is involved enter the analysis date and time in this field. Refer to the date/time format at the end of this table.	Date/ Time	16*	X
Analyzed	Date and time of sample analysis. Refer to the date and time format at the end of this table. For Analyzed values that are not applicable use the value of 00/00/0000 00:00.	Date/ Time	16*	X
LabID	Identification of the laboratory performing the analysis.	Text	7	X
QCLevel	The level of laboratory QC associated with the analysis reported in the EDD. If only the Analytical Results Table (A1) and the Sample Analysis Table (A3) information are submitted for the sample, enter “COA”. If the Laboratory Instrument Table (A2) information is also submitted for the sample, enter “COCAL”	Text	6	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
ResultBasis	Indicates whether results associated with this sample records are reported as wet or percent moisture corrected. Enter “WET” if results are not corrected for percent moisture. Enter “DRY” if percent moisture correction is applied to results. For aqueous samples, enter “WET”. For other matrices where basis is not applicable enter “NA”	Text	3	X
TotalOrDissolved	This field indicates if the results related to this sample record are reported as a total or dissolved fraction. If not applicable please report "NA"	Text	3	X
Dilution	Dilution of the sample aliquot. Enter “1” for method blanks, LCS, and LCSD, or if the field samples was analyzed without dilution.	Numeric (1)	10(6)	X
HandlingType	Indicates the type of leaching procedure, if applicable (i.e., SPLP, TCLP, WET). Enter “NA” if the sample analysis was <u>not</u> performed on a leachate.	Text	10	X
HandlingBatch	Unique laboratory identifier for a batch of samples prepared together in a leaching procedure (i.e., SPLP, TCLP, or WET preparation). The HandlingBatch links samples with leaching blanks. Enter “NA” if the sample analysis was <u>not</u> performed on a leachate.	Text	12	X
LeachateDate	Date and time of leaching procedure (i.e., date for SPLP, TCLP, or WET preparation). Refer to the date and time format at the end of this table.	Date /Time	16*	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	. For Analyzed values that are not applicable use the value of 00/00/0000 00:00			
Percent_Moisture	For soil and sediment samples, enter the percent of sample composed of water. For aqueous samples enter "100". For other matrices where Percent_Moisture is not applicable use a value of -99	Numeric (1)	10(6)	X
MethodBatch	Unique laboratory identifier for a batch of samples of similar matrices analyzed by one method and treated as a group for matrix spike, matrix spike duplicate, or laboratory duplicate association The method batch links the matrix spike and/or matrix spike duplicate or laboratory duplicates to associated samples. Note the MethodBatch association may coincide with the PreparationBatch association. The MethodBatch is specifically used to link the MS/MSD and/or DUP to associated samples.	Text	12	X
PreparationBatch	Unique laboratory identifier for a batch of samples prepared together for analysis by one method and treated as a group for method blank, LCS and LCSD association. The PreparationBatch links method blanks and laboratory control samples (blank spikes) to associated samples. Note, the PreparationBatch association may coincide with the MethodBatch association but the PreparationBatch specifically links the Method Blank and LCS to associated samples.	Text	12	X
RunBatch	<u>For all other methods</u> the RunBatch is the unique identifier for a batch of analyses performed on one instrument under the control	Text	12	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	of one initial calibration and initial calibration verification. The RunBatch links both the initial calibration and initial calibration verification to subsequently analyzed and associated continuing calibrations, field samples, and QC analyses. For GC/MS methods, the RunBatch also links a BFB or DFTPP tune. A distinct RunBatch must used with every new initial calibration within a method The value entered in this field links a particular sample/method/analysis type record to a set of associated initial calibration and initial calibration verification records from Table A2. If Table A2 is not submitted enter a value of 'NA" in this field.			
AnalysisBatch	For radiochemistry methods leave this field blank. For all other methods the AnalysisBatch is the unique identifier for a batch of analyses performed on one instrument and under the control of a continuing calibration or continuing calibration verification. The AnalysisBatch links the continuing calibration or calibration verification to subsequently analyzed and associated field sample and QC analyses. For GC/MS methods, the AnalysisBatch also links the BFB or DFTPP tune. A distinct AnalysisBatch must be used with every new continuing calibration or continuing calibration verification within a method The value entered in this field links a particular sample/method/analysis type record to a set of	Text	12	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	associated continuing calibration records in the Laboratory Instrument table.			
LabReportingBatch	Unique laboratory identifier for the EDD. This is equivalent to the sample delivery group, lab work number, login ID, etc. The LabReportingBatch links all records in the EDD reported as one group. The value entered in this field must be the same in all records.	Text	12	X
LabReceipt	Date and time the sample was received in the lab. A time value of 00:00 may be entered. Refer to the date/time format at the end of this table.	Date/ Time	16*	X
LabReported	Date and time hard copy reported delivered by the lab. A time value of 00:00 may be entered. Refer to the date/time format at the end of this table.	Date/ Time	16*	X
Comment	Add any comments or additional information specific to the sample analysis data record.	Text	200	

C Only required for regular samples, duplicates and MS/MSDs.
X Required field.
(1) Field Length indicates decimal precision in parenthesis. For example, 5(2) = a total width of 5 numbers including a maximum of 2 decimal places.
* Format Date and Time as MM/DD/YYYY hh:mm; where MM = two digit month, DD = two digit day, and YYYY = four digit year, hh = hour in 24 hour format, and mm = minutes.

3.0 Laboratory Data Checker

The Laboratory Data Checker is a web-based application that will review Laboratory Electronic Data Deliverables (LEDDs) for adherence to Tetra Tech's EDD format requirements. EDDs will be reviewed for elements such as missing data and/or columns of data, and compliance of the data within each column to the required data types/lengths.

Once an EDD passes through the checker with no errors, it must be submitted to Tetra Tech through the LEDD Checker application.

Access to the LEDD Checker application will be provided by an initial registration/approval process. An Information Systems Group (ISG) Administrator will approve requests for access**.** To access the site or begin the registration process, visit the ISG web site at http://isg.ttnus.com and select the "Laboratory Checker" link on the left of the home page. Registered users may access the checker immediately by logging in to the system using theircredentials. New users must select the "Register" button and provide all of the requested information**.**

After completing all fields on the registration form, select the "Submit" button to complete the request process. Upon verification by an ISG Administrator, an email notification will be sent verifying the user ID, password and account status. Forgotten passwords may be retrieved by using the "Forgot password?" link on the login page. Note that the email address that was provided for registration or password retrieval is the user ID and must be a valid e-mail address.

The general process for submitting EDD files through the LEDD Checker involves a 3-stage process that includes an upload stage, an error checking stage and a submittal stage.

Log into the LEDD Checker by typing your login credentials and select the "Login" button. The LEDD Checker home page provides a general overview of the checker functionality and EDD file format requirements. At the bottom of the home page, example EDDs are provided that may be viewed or downloaded. To download the files, right click on the link and select "Save target as" from the menu. Each LEDD Checker page includes a navigation bar with links to return to the home page or continue the checking and submittal process. Users should **NOT** use the back or forward buttons on the browser, instead use the links provided in the application to navigate through the site.

Detailed information regarding EDD preparation, formatting requirements and text file naming conventions are provided in the Electronic Data Format Requirements Section of the Laboratory SOW.

Begin the upload stage by selecting the "Upload/Check Files" link on the home page. Follow the steps on the upload page starting with the selection of the laboratory name that corresponds to your organization. If your organization is not listed, contact LabSupport@tetratech.com, and provide a full description of your organization name, contact information and include "Laboratory Contractor ID Request" in the subject line. An ISG Administrator will respond to the request via e-mail.

Load the appropriate A1, A2, or A3 target EDD files by clicking the "Browse" button next to each data table input box. A file browser dialog will appear allowing files to be selected from a local or network drive. After the EDD files are loaded, click the "Upload" button to complete the upload stage. Note that each table may be uploaded and checked separately; however, a minimum of the A1 and A3 files are required in order to submit the EDDs.

If the file upload was successful, the checking page will immediately load. Begin the checking stage by selecting the “Check Files” button. The LEDD Checker will begin validating the EDD files for compliance. Depending on file size and network activity the validation process may take several minutes. The progress should be displayed in the information bar at the bottom of the browser window. **Do not** select the “Check Files” button again or otherwise use the browser during this process. Other applications may be used; however, note that the LEDD Checker may not sit idle for more than 30 minutes. If the time is exceeded a new session must be started in a new browser window.

Any errors will be processed and returned on the error page. The following general errors may be returned.

- Column count / table structure errors – due to column header names being included, improper delimiter, extra tabs, extra or missing columns of data, spaces or other characters at the end of a row.
- Row and column value specific errors – may occur for one or more reasons including: data truncation, invalid date / time format, invalid decimal precision or field width exceedance, or if a value is not in a list of valid values or expected range.

If column count / table structure errors are encountered, the LEDD Checker will return an error and stop the checking process.

The EDDs will not be processed any further until the column errors are resolved. Text fields are validated for truncation. Date / Time fields are validated for truncation and format compliance. Numeric decimal fields are validated for truncation, character type compliance and decimal precision. All required fields are validated for null values or empty text strings (i.e. spaces). The LEDD Checker will return a list of all errors in and include a reference to the row number on which the error occurred. Note that consecutive EDD files may be loaded and checked, and submitted while logged in. However, no data may be submitted until all EDD files have passed through the LEDD Checker without errors. The list of errors may be printed by selecting the “Print this Page” button from the checker error page.

If the EDD files pass with no errors, the submittal page will immediately load. To complete the submittal stage, include the following information in the comment and additional information area of the form: laboratory name, laboratory contact person, project name, project number, site name/number, fractions included and any specific comments related to the EDD. Select the “Submit Files” button to continue the submittal process.

The submittal stage is not considered complete until a unique ticket key reference is returned in the browser window. The ticket key reference must be printed for record of submission and future reference. In addition, a copy of the ticket key reference must be included in the PDF data package.

D-2 ARI

ATTACHMENT NO. 2

STATEMENT OF WORK/PRICE TABLES

TECHNICAL SPECIFICATION FOR LABORATORY SERVICES
DERECKTOR SHIPYARD, SITE 19
NAVAL STATION NEWPORT, NEWPORT, RHODE ISLAND

COMPREHENSIVE LONG-TERM ENVIRONMENTAL ACTION - NAVY (CLEAN)
CONTRACT N62470-08-D-1001, CONTRACT TASK ORDER (CTO) NO. WE61

DATA GAPS INVESTIGATION – MARINE SEDIMENT
CHEMICAL ANALYSIS

1.0 INTRODUCTION

Tetra Tech NUS, Inc. (TtNUS) under CLEAN Contract N62470-08-D-1001, is procuring laboratory analytical services to support a Data Gaps Investigation at Naval Station Newport (NAVSTA), off-shore portions of Derecktor Shipyard, Site 19. The requested analysis is tributyltin (TBT)..

The laboratory performing this analysis must provide a copy of its Department of Defense (DOD) Environmental Laboratory Accreditation Program (ELAP) accreditation letter; the scope of the ELAP accreditation must include TBT in solid and aqueous matrices.

The responding laboratory must submit limits of quantitation (LOQs), limits of detection (LODs), and detection limits (DLs) for TBT for sediment by filling out the last three columns of the table in Attachment A and including the completed attachment with the bid response.

After subcontract award, the laboratory will be required to submit Standard Operating Procedures (SOPs) and relevant precision and accuracy limits for all preparation and analytical methods required under this scope of work. The laboratory will also be asked to complete Uniform Federal Policy (UFP) Sampling and Analysis Plan (SAP) Worksheets 19, 24, 25, and 28 for inclusion in the SAP. The SAP will be prepared according to the UFP for Quality Assurance Project Plans (March 2005) and will utilize the 37 UFP-SAP worksheets.

2.0 SAMPLE INFORMATION

The approximate number of samples to be submitted, the type of analyses to be conducted, and the analytical methods to be used are summarized in Table 1. This investigation includes analysis of marine sediment samples and associated aqueous rinsate blanks.

The sampling is scheduled for June/July, 2011. The exact starting date of sample collection will be communicated to the laboratory at least 2 weeks in advance.

The samples are expected to be of low or moderate contaminant concentration except that because the sediment samples will be marine sediment, they may contain high concentrations of sodium, calcium, and magnesium. The field crew will attempt to identify any potentially high concentration samples and mark as such on the chain-of-custody forms.

Field duplicate samples will be submitted to the laboratory with "blinded" identification. The field crew will designate one per twenty sediment samples for matrix spike/matrix spike duplicate (MS/MSD) analysis. Additional volumes of these samples will be provided as necessary.

Percent Moisture

For the sediment analysis, the laboratory must decant any standing water, homogenize the sample, and determine the percent moisture before sample analysis. The sample aliquots must be increased to compensate for the moisture content of the samples or the sample may be centrifuged to eliminate more water. **The project LOQ goals listed in Attachment A must be met regardless the moisture content of the sediment samples**.

If the percent moisture content is too high and the sediment samples contain noticeable organic material, further dewatering must be performed at the laboratory prior to sample analysis. This could be accomplished by freeze-drying under controlled conditions proven to recover the analysis-specific target compounds; centrifugation and decanting free water; low temperature oven drying (below 60°C); or other procedure proposed by the laboratory and approved by Tetra Tech.

TBT results must be reported as tin, in units of ug Sn/kg or ng Sn/g. **3.0 ANALYSIS/REPORTING INFORMATION**

One hard copy data package deliverable and two PDF CD copies must be submitted, in addition to the electronic data deliverables to be provided in the format described in Attachment C. The original chain-of-custody form received with the samples and signed by the laboratory sample custodian must be returned with the hard copy data package.

The analytical requirements and approximate number of samples to be submitted for analysis are detailed in Table 1. Analysis and reporting requirements addressed in the Department of Defense (DOD) Quality Systems Manual (April 2009) and the requested methods must be followed. Additionally, it is a requirement of TtNUS that the associated PDF and hard copy data packages for the TBT analysis must be in a CLP-modified format and must include the appropriate summary forms and raw data for all samples and laboratory quality control samples. The summary forms should include the method-specific quality control limits (recoveries, relative percent differences, relative standard deviations, and/or percent differences, etc.). Second-source initial calibration verification results must be reported on a summary form.

The TtNUS sample identification numbers **must be included** on the raw data and summary forms.

Additionally, each hard copy and PDF data package must contain a summary data package. This summary data package shall consist of only the summary forms (i.e., the CLP-like equivalent of Forms 1 through 8).

Attachment A details the required target analyte list and LOQ goals that must be met. The laboratory must submit its LOQs, LODs, and DLs for TBT for sediment by filling out the last three columns of the table in Attachment A and including the completed attachment with the bid response. If the LOQ goal for a target compound is not technically achievable, the laboratory should propose the lowest LOQ technically possible for the requested method.

Attachment B details the required summary forms for CLP-like data packages and requirements for organization/bookmarking of hard copy/PDF data packages.

Non-detected results must be reported down to the laboratory's LODs. Positive results above the DL but below the laboratory's LOQ must be reported as estimated values qualified with a "J". Sediment samples must be reported on a **dry-weight basis**.

The hard copy/PDF data package deliverable must contain a detailed case narrative for all analytical fractions. This case narrative must also include the Contract Task Order (CTO) number, the site name, and the TtNUS Project Manager's name. Data from all analytical runs (i.e., original, dilution, re-analysis) must be reported in the raw data and Form Is.

As part of the laboratory case narrative, it is required that the Laboratory Quality Assurance Manager sign an attestation statement verifying that all electronic diskette deliverables exactly match the data summary forms (i.e. Form Is).

Sample Delivery Group (SDG) and fractionally-specific text (TXT) files containing all environmental sample and field quality control blank analysis results must be generated in accordance with the requirements outlined in Attachment C of this specification.

Maximum holding time allowances, as defined in the following table, are to be strictly observed. Calculation of holding time is in calendar days and is to begin from the time of sample collection. The holding times are as follows:

Analyses	Preservation	Holding Time
Tributyltin	Aqueous: Cool to ≤ 6 ^{o}C	7 days to extraction, 40 days to analysis
	Sediment: Cool to ≤ 6 ^{o}C	14 days to extraction, 40 days to analysis

These holding times are based on 40 CFR 136, data validation criteria, and method specific requirements, and are measured from date of collection for samples preserved as requested in the analytical methods. The holding time criteria depicted apply to all analyses necessary to successfully determine the contaminant level contained in the sample. Hence, **the holding time criteria apply to any/all subsequent sample dilutions and re-analyses**.

The TtNUS Project Manager for this project is Mr. Stephen Parker and he must be contacted in the event of any laboratory problems that could impact project deadlines (i.e., late deliverables, technical problems in the lab that could lead to late deliverables.) To ensure good communication, it is required that the laboratory's appointed project manager contact Mr. Parker once a week for the entire project duration.

Contact information for Mr. Parker is as follows:

Tetra Tech NUS
250 Andover Street, Suite 200
Wilmington, MA 01887
Phone: 978-474-8434
Fax: 978-474-8499
Email: stephen.parker@tetratech.com

Technical, quality assurance, and data format concerns are to be directed to Ms. Lucy Guzman at 978-474-8416 or via e-mail lucy.guzman@tetratech.com. Ms. Guzman must be contacted and informed of any difficulties encountered during the conduct of the requested analyses.

Analytical data turnaround times are to be measured from receipt of each sample shipment. All hard copy/PDF (2 CDs) analytical data packages and associated electronic (TXT) deliverables are due within the turnaround term of 21 calendar days from receipt of the last sample in a Sample Delivery Group (SDG).

The SDGs shall contain no more than 20 samples. The frequency with which SDGs contain fewer than 20 samples should be minimal. The hard copy data packages, PDF files (CDs), and electronic deliverables must be received at the same time or the deliverable will be considered incomplete and payment deductions may be imposed.

The hardcopy analytical data package, one PDF (CD) copy of the analytical data package, and the original chain-of-custody form (received with the samples and signed by the laboratory sample custodian) shall be sent to Ms. Lucy Guzman.

The electronic (TXT) deliverables, one PDF (CD) copy of the analytical data package, and a copy of the chain-of-custody form, should be sent to Ms. Tobrena Sedlmyer. The contact information for Ms. Sedlmyer is as follows:

Tetra Tech NUS, Inc.
661 Andersen Drive, Foster Plaza 7
Pittsburgh, PA 15220-2745
Phone: 412-921-8582
Fax: 412-921-4040
e-mail: tobrena.sedlmyer@tetratech.com

4.0 PERIOD OF PERFORMANCE/BOTTLEWARE INFORMATION

All samples will be shipped to the laboratory via express carrier within 48 hours of collection. **The laboratory must be capable of receiving samples on Saturdays.** Please circle the Yes or No at the bottom of Table 1 which will indicate if the laboratory will provide courier service from the collection site in Newport, Rhode Island at no extra charge. The laboratory will be notified at least 2 weeks prior to sample collection.

Bottleware shipments will be coordinated by the field operation leader.

The laboratory shall provide all necessary sample containers **(plus approximately 10% extra for breakage)**. All sample containers must meet ICHEM series 300 cleanliness criteria (or equivalent), and documentation of certified cleanliness must be provided. The bottleware must be shipped to the designated location in Coleman-like coolers. Each cooler must include a “temperature blank” vial. **The laboratory must also provide any extra coolers needed for return shipment of samples to the laboratory for analysis.** The laboratory is also requested to provide a packing slip indicating the analytical parameters for which each container type is designated, sample labels, and chain-of-custody forms.

5.0 ADDITIONAL COMMENTS/CONTACTS

Within the laboratory, the internal transfer of samples, extracts, and digestates must be accomplished and documented as controlled custody transfers. The laboratory must submit the documentation that supports an unbroken chain of custody for samples, digestates and extracts from time of receipt or production in the laboratory until disposal.

The laboratory is to provide a minimum of 60 days storage of sample extracts and 60 days storage of intact leftover sample aliquots. **Additionally, the laboratory must store PDF data packages for 7 years.**

All analyses conducted under this subcontract assignment are to be performed at the solicited facility only. The laboratory is not permitted to lower-tier subcontract these analyses, or analyze these samples at a corporate facility other than the facility stipulated without prior notification and consent from the CLEAN Subcontracting Officer.

The unit cost for analysis is to include compensation for containers, preservatives, coolers, shipping costs, storage, disposal, and laboratory quality control analyses (such as matrix spike, matrix spike duplicate, laboratory duplicate, and laboratory control sample analyses.) These items are not to be billed as separate line items.

Contract concerns, and response to this solicitation, are to be directed to:

Ms. Meg Price
CLEAN Subcontracting Officer
Tetra Tech NUS, Inc.
234 Mall Boulevard, Suite 260
King of Prussia, PA 19406-1433
Phone: 610-382-1525
Fax: 610-491-9645
e-mail: meg.price@tetratech.com

Triplicate copies of invoices associated with the analyses contracted herein are to be submitted to the attention of the Accounting Supervisor:

Tetra Tech NUS, Inc.
661 Andersen Drive, Foster Plaza 7
Pittsburgh, PA 15220
Phone: 412-921-8506
Fax: 412-921-4040

Please confirm the laboratory's ability to perform the methodologies requested at the analyte quantitation limits indicated. Also confirm available laboratory capacity, and complete/confirm the costing information indicated in Table 1. All costing information must reflect the terms and conditions established herein.

TABLE 1
NUMBER OF SAMPLES/ANALYTICAL METHODS
CTO WE61, OFF-SHORE DERECKTOR SHIPYARD
NAVSTA NEWPORT, NEWPORT, RI
DATA GAPS INVESTIGATION, CHEMICAL ANALYSIS

Matrix	Parameter	Method	# Samples	Unit Price	Total Cost
Sediment	Tributyltin(1)	Krone, et al.(2)/ GC/MS SIM	380	$	$
Aqueous (rinsate blank)	Tributyltin(1)	Krone, et al.(2)/ GC/MS SIM	18	$	$

(1) Must be reported as tin, in units of ug Sn/kg or ng Sn/g. (2) Krone CA, Brown, DW, Burrows, DG, Chan, S-L, Varanasi, U (1989). Butyltins in sediment from marinas and waterways in Puget Sound, Washington State, U.S.A. Mar Poll Bull 20:528-31.

TOTAL COST $

The laboratory must state if they are not DOD ELAP accredited for TBT for solids and water.

Can the laboratory provide sample pick-up on site? YES or NO (circle one)
If yes is there a charge and what is that charge?_____

Name of Laboratory_____

Signature_____

ATTACHMENT A

REQUIRED TARGET ANALYTE LIST AND PROJECT ACTION LIMIT

ATTACHMENT A

REQUIRED TARGET ANALYTE LISTS AND PROJECT ACTION LIMITS
CTO WE61, OFF-SHORE DERECKTOR SHIPYARD, SITE 19
NAVAL STATION NEWPORT, NEWPORT, RHODE ISLAND
DATA GAPS INVESTIGATION, CHEMICAL ANALYSIS

Matrix: Sediment
Analyte: Tributyltin, analyzed by GC/ MS SIM

Analyte	CAS Number	PAL (µg/kg)	PAL Reference	Project LOQ Goal (µg/kg)	Lab Limits		
					LOQ (µg/kg)	LOD (µg/kg)	DL (µg/kg)
Tributyltin (as Sn[1])	36643-28-4	228	NOEC	20			

Note:
1. TBT must be reported as tin, in units of ug Sn/kg or ng Sn/g.

ATTACHMENT B
DATA PACKAGE DELIVERABLES REQUIREMENTS

DATA PACKAGE DELIVERABLE REQUIREMENTS

The laboratory is to provide a hard copy plus two compact disks (CDs) each containing a PDF file in the following format:

1. Table of Contents
2. Case Narrative
3. Chain-of-Custody
4. Data Summary Package (contains summary of all CLP or CLP-like Forms 1 through 15 per analytical fraction)
5. Analytical Fractions (e.g., VOA, SVOC, Metals, etc.)
   a. Results and QC Summary (summary of all CLP or CLP like Forms 1 through 15 for a particular analytical fraction)
   b. Raw Sample Data (includes all sample dilutions, sample re-analyses, QC samples, etc.)
   c. Calibration Data (includes all initial and continuing calibrations and initial calibration verfication)
   d. Miscellaneous (includes extraction/preparation forms, percent solids determination, IDLs, MDLs, etc.)

Each of the above sections should be bookmarked in the PDF for easy access.

Summary Forms for GC/MS analysis of Tributyltin should be presented in a CLP-Like format. The following Summary Forms must be included:

Result Summary	One Sample per summary page. Presentation of analytical results for both method blanks and environmental samples, date of collection, preparation, and analysis. Environmental samples should be identified with the field identification numbers on the COCs.
Surrogate Recovery Form	Present all information contained on CLP Form II.
Summary of Matrix Spike/Matrix Spike Duplicate Recovery	Present all information contained on CLP Form III.
Instrument Performance Check Summary Form - Mass Spec Tuning Form	Present all information Contained on CLP Form V.
Initial Calibration Summary	Present all information contained CLP Form VI.
Continuing Calibration Summary	Present All Information contained on CLP Form VII.
Internal Standard Area and Retention Time Summary	Present all information contained CLP Form VIII.

<u>Also must include</u>: initial calibration verification summary forms and identification of the compounds associated with internal standards.

ATTACHMENT C
ELECTRONIC DATA DELIVERABLE REQUIREMENTS

ELECTRONIC DATA FORMAT REQUIREMENTS

1.0 **INTRODUCTION**

The laboratory is to submit text-based tab delimited EDD files for each SDG using Tetra Tech's laboratory data checker explained below. The files must be in the format specified in this Attachment. Additional information such as laboratory name, project name, fractions included, project number, site name/number, laboratory contact person and any specific comments related to the EDD should be included in the comments section of the EDD Submittal page.

The RESULT for nondetects should be populated with the project-specific sample quantitation reporting limits (i.e., either the sample quantitation limit or method detection limit, as specified in Section 3.0 of this scope of work. Any corrections made to the hardcopy data must also be made to the electronic file. Appropriate qualifiers as identified by the analytical protocol must also be designated; laboratory QC non-compliance codes are not to be depicted.

Tetra Tech's electronic EDD format follows the ADAPT structure and requires the A1 and A3 files. The A2 file is only required if the project is using ADAPT; and, for non-ADAPT EDD submittals the A2 file may be omitted. The EDD consists of separate, tab-delimited ASCII text files. Each file corresponds to a database table. The tables are identified as the Analytical Results Table (A1) and Sample Analysis Table (A3). A separate set of text files must be created and submitted for each sample delivery group (SDG). The files must be identified to correspond to the (A1) table and the (A3) table. The file naming convention is: the Sample Delivery Group (SDG) followed by the table identifier (A1 or A3), followed by the ".txt" extension. The file names must not contain spaces or special characters. For example, the EDD file names for a laboratory-reporting batch identified as SDG001 would be as follows:

SDG001A1.txt
SDG001A3.txt

On certain projects Tetra Tech will utilize the ADAPT Electronic Data Validation software, which will require the laboratory to use the ADAPT electronic data deliverable checker software prior to submitting the files through Tetra Tech's laboratory data checker (this will be clearly specified in the Tetra Tech laboratory statement of work). The ADAPT checker software can be downloaded from Laboratory Data Consultants' web site: http://www.lab-data.com. For projects which Tetra Tech is using the ADAPT software, Tetra Tech will provide the laboratory with the project library. The laboratory is not permitted to modify the project library. ADAPT projects will require the laboratory to export all three checked files (A1, A2, and A3) from the ADAPT software and submit them through Tetra Tech's laboratory data checker. **ADAPT error logs generated must be included with the electronic PDF data validation package and cannot be submitted through the laboratory data checker.**

The values reported in the EDD text files must agree exactly with the final values reported on the PDF data package sample result summaries. The details of file naming conventions, data structure and data checker use are discussed below.

Analytical Results Table (A1 File)

The Analytical Results table contains analytical results and related information for target analytes in field samples and associated laboratory quality control samples (excluding calibrations and tunes). Field samples and laboratory method blanks must report a result record for each analyte reported within a method. Laboratory control samples (LCS and LCSD) and matrix spike samples (MS and MSD) must report a result record for every analyte specified as a spiked analyte in the laboratory statement of work. Table A1 in this document lists the field names and data type descriptions for the Analytical Results Table (A1).

Lab Instrument Table (A2 File)

A2 file is only required if the project is using ADAPT. In all other EDD submittals, the A2 file may be omitted. Laboratories should refer to the ADAPT User Guide for populating the A2 Table.

Sample Analysis Table (A3 File)

The Sample Analysis table contains information specific to field environmental samples and laboratory quality control analyses (excluding calibrations and tunes). A sample record must exist for each sample/method/matrix/analysis type combination. Table A3 in this document lists the field names and data type descriptions for the Sample Analysis Table (A3).

All electronic data deliverables are due within the same time established for the associated hardcopy data packages.

In addition, the laboratory QC officer must read and sign a copy of the Quality Assurance Review Form displayed on the next page of this Attachment. Electronic deliverables are not considered to be complete without the accompanying Quality Assurance Review Form.

I _____, as the designated Quality Assurance Officer, hereby attest that all electronic deliverables have been thoroughly reviewed and are in agreement with the associated hardcopy data. The enclosed electronic files have been reviewed for accuracy (including significant figures), completeness and format. The laboratory will be responsible for any labor time necessary to correct enclosed electronic deliverables that have been found to be in error. I can be reached at
(_____)_____if there are any questions or problems with the enclosed electronic deliverables.

Signature:_____ Title: _____Date: _____

2.0 EDD Field Properties

Tables A1 and A3 in this document specify the EDD field properties. Laboratories should refer to the ADAPT User Guide for populating the A2 Table. These include the field name, sequence order, field description, data type/length and reporting requirement for each field. Fields in the EDD **must** be sequenced according to the order that they appear below in Tables A1 and A3. For example, in the Analytical Results table (A1), the field "ClientSampleID" will always be the first piece of information to start every new line of data (or database record), followed by the field "LabAnalysisRefMethodID", "AnalysisType", etc.

When creating an EDD as a text file, use the ASCII character set in a file of lines terminated by a carriage return and line feed. No extra characters are allowed at the end of a line, after the carriage return and line feed. Enclose each data value with double quotes (text qualifier) and separate each field value with a **tab** character (tab delimiter). Data fields with no information (null) may be represented by two consecutive tabs. For example, in the Sample Analysis table, since the "Collected", "ShippingBatchID", and "Temperature" fields do not apply to laboratory generated QA/QC samples, the record for a Laboratory Control Sample by Method 8270C would be entered as follows. Note that the first two fields ("ProjectNumber" and "ProjectName") are omitted in this example.

…"LCSW100598" "AQ" "LCSW100598" "LCS" "8270C",…etc.

If a field is populated with less than the maximum allowed number of characters, do not pad the values with leading or trailing spaces. In the above example, although the "MatrixID" field can accommodate up to 10 characters, only 2 characters were entered in this field. **Do not include the delimiter (tab character) within any of the field values.** Example EDD files may be downloaded from the LEDD Checker application.

An example database shall be sent for review prior to the first electronic deliverable in the required .txt format. The example file will be examined for completeness and comments will be sent to the laboratory. Any questions regarding the electronic deliverable should be directed to LabSupport@tetratech.com

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
ClientSampleID	Client or contractor's identifier for a field sample If a sample is analyzed as a laboratory duplicate, matrix spike, or matrix spike duplicate, append suffixes DUP, MS and MSD respectively to the Client Sample ID	Text	25	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	with no intervening spaces or hyphens (i.e. MW01DUP, MW01MS, and MW01MSD). For Method Blanks, LCS, and LCSD enter the unique LaboratorySampleID into this field. Do not append suffixes to the ClientSampleID for dilutions, reanalyses, or re-extracts (the AnalysisType field is used for this distinction). For example, MW01DL and MW01RE are not allowed. Parent sample records must exist for each MS and MSD. If an MS/MSD is shared between two EDDs, records for the MS/MSD and its parent sample must exist in the Analytical Results table for both EDDs.			
LabAnalysisRefMethodID	Laboratory reference method ID. The method ID may be an EPA Method number or a Lab Identifier for a method such as a SOP Number, however; method ID is specified by the project. The method ID must be entered into the standard list.	Text	25	X
AnalysisType	Defines the analysis type (i.e., Dilution, Reanalysis, etc.). This field provides distinction for sample result records when multiple analyses are submitted for the same sample, method, and matrix; for example dilutions, re-analyses, and re-extracts.	Text	10	X
LabSampleID	Laboratory tracking number for field samples and lab generated QC samples such as method blank, LCS, and LCSD. There are no restrictions for the LabSampleID except for field length and that the LabSampleID must be distinct for a given field sample or lab QC sample and method.	Text	25	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	Suffixes may be applied to the LabSampleID to designate dilutions, reanalysis, etc.			
LabID	Identification of the laboratory performing the analyses.	Text	7	X
ClientAnalyteID	CAS Number or unique client identifier for an analyte or isotope. If a CAS Number is not available, use a unique identifier provided by the client or contractor. The ClientAnalyteID for a particular target analyte or isotope should be specified by the project and must exist in the standard value tables for Analytes. For the LCS, LCSD, MS, and MSD, it is only necessary to report the compounds designated as spikes in the library (and surrogates for organic methods.) For TICs from GC/MS analyses, enter the retention time in decimal minutes as the Client Analyte ID.	Text	12	X
AnalyteName	Chemical name for the analyte or isotope. The project specifies how an analyte or isotope is named. The analyte name must be associated to a ClientAnalyteID in the standard values table for Analytes (excluding compounds designated as TIC's).	Text	60	X
Result	Result value for the analyte or isotope. Entries must be numeric. For non-	Numeric (1)	20(6)	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	detects of target analytes or isotopes and spikes, do not enter "ND" or "0". Do not leave this field blank. If an analyte or spike was not detected, enter the associated value specified in Section 3.0 of this scope of work (e.g., LOD, SQL, PQL, etc.), corrected for dilution and percent moisture as applicable. Do not enter "0". A "0" result may be acceptable for surrogate or internal standard percent recoveries; however, it should not be reported for any target compound.			
ResultUnits	The units defining how the values in the Result, DetectionLimit, and ReportingLimit fields are expressed. For radiochemistry this also includes how the value in the Error field is expressed.	Text	10	X
LabQualifiers	A string of single letter result qualifiers assigned by the lab based on client-defined rules and values. <u>The "U" Lab Qualifier must be entered for all non-detects.</u> Other pertinent lab qualifiers may be entered with the "U" qualifier. Order is insignificant. Lab qualifiers other than those listed in the standard values table may be used. If so, these must be added to the standard value table in the application.	Text	7	Q
DetectionLimit	For radiochemistry methods, the minimum detectable activity for the isotope being measured. For all other methods: The minimum	Numeric (1)	10(6)	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	detection limit value for the analyte being measured. For surrogates, internal standards, etc. where detection limits are not applicable use the value -99.			
DetectionLimitType	Specifies the type of detection limit (i.e., MDA, MDL, IDL, etc.). If -99 is specified in the DetectionLimit field us the value NA.	Text	10	X
RetentionTime or Error	For radiochemistry methods only, enter the 2 Sigma Counting Errors. The units for error are entered in the ResultUnits field. For GC/MS methods only, enter the time expressed in decimal minutes between injection and detection for GC/MS TICs only For target analytes in all other methods, leave this field blank. Note: GC retention times are not evaluated at this time.	Text	5	T
AnalyteType	Defines the type of result, such as tracer, surrogate, spike, or target compound.	Text	7	X
PercentRecovery	For radiochemistry methods: The tracer yield, if applicable. For all other analytical methods: The percent recovery value of a spiked compound or surrogate. If the spike or surrogate was not recovered because of dilution, enter “DIL”. If a spike or surrogate was not recovered because of matrix interference, enter “INT”. If a spike or	Numeric (1)	5(3)	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	surrogate was not recovered because it was not added to the sample, enter “NS”.			
RelativePercentDifference	The relative percent difference (RPD) of two QC results, such as MS/MSD, LCS/LCSD, and Laboratory Duplicates. Report RPD in Laboratory Duplicate, LCSD, and MSD records only.	Numeric (1)	5(3)	X
ReportingLimit	Reporting limit value for the measured analyte or isotope Factor in the dilution factor and percent moisture correction, if applicable. The Reporting Limit for each analyte and matrix in a given method is specified in the project library or QAPP. For surrogates, internal standards, etc. where reporting limits are not applicable use the value -99.	Numeric (1)	10(6)	X
ReportingLimitType	Specifies the type of reporting limit (i.e., CRQL, PQL, SQL, RDL, etc). The Reporting Limit Type for each method and matrix is specified in the project library or QAPP. If -99 is specified in the ReportingLimit field us the value NA.	Text	10	X
ReportableResult	This field indicates whether or not the laboratory chooses an individual analyte or isotope result as reportable. Enter “YES” if the result is reportable. Enter “NO” if the result is not reportable. If only one analysis is submitted for a particular sample and method, enter “YES”	Text	3	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	for all target compounds (where Analyte Type = TRG). For GC/MS methods enter yes for tentatively identified compounds (where Analyte Type = TIC). If two or more analyses are submitted for a particular sample and method (i.e. initial analysis, reanalysis and/or dilutions), enter "YES" from only <u>one</u> of the analyses for each target compound. For example: a sample was run a second time at dilution because benzene exceeded the calibration range in the initial, undiluted analysis. All target analytes are reported in each analysis. For the initial analysis, (Analysis Type = RES), enter "NO" for benzene and enter "YES" for all other compounds. For the diluted analysis (Analysis Type = DL), enter "YES" for benzene and enter "NO" for all other compounds. For TICs (Analyte Type = TIC), if more than one analysis is submitted for a particular sample and method, choose only one of the analyses where Reportable Result = YES for <u>all</u> TICs. For example, a sample was run a second time because one or more target compounds exceeded the calibration range in the undiluted analysis. Choose a particular analysis and enter "YES" for all TICS. In the other analysis enter "NO" for all TICs. Note that it is not necessary to report the full target analyte list for the initial result, dilution, re-analysis, or re-extraction. However, each target analyte must be reported YES once and once only in the case of multiple analyses for a given sample, method, and			

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	matrix. In the case of organics, all surrogates must be reported for all analyses submitted for a given sample, method, and, matrix.			
SpkConcnAdded	The spike added. This value must be reported in the same units as the result. Where (SA) in the following equation: % Recovery = (SSA-SC)/SA x 100% where : SSA is the spiked sample concentration (amount) after spiking. SC is the sample concentration (amount) before spiking. SA is the the expected increase in sample concentration (amount) as a result of spiking. This value must incorporate all correction factors such as dilution factor and moisture content that are applied to the spiked sample when computing the spiked sample concentration or amount. Enter -99 where no spike was added.	Numeric (1)	10(6)	X
SpkParentSampleID	The sampleID of a sample (often called the original sample) that receives a spike aliquot to form a spiked sample such as a matrix spike. This is not the same as the ID of the spiked sample (such as a matrix spike) after spiking. The result for SpkParentSampleID and the result (i.e., SpkConcnAdded) for the spiked sample are used to compute percent recovery of the analyte.	Text	25	X
SamplePrepInitial	The initial sample preparation volume in liters (L) for aqueous samples or grams (g) for solid samples.	Numeric (1)	20(6)	

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
SamplePrepFinal	The final sample preparation volume in liters (L) for aqueous samples or grams (g) for solid samples.	Numeric (1)	20(6)	
LimitOfDetection	The smallest amount or concentration of a substance that must be present in a sample in order to be detected at a 99% confidence level. In other words, if a sample has a true concentration at the LOD, there is a minimum probability of 99% of reporting a "detection" (a measured value ≥ DL) and a 1% chance of reporting a non-detect (a false negative).	Numeric (1)	10(6)	N
Comment	Add any comments or additional information specific to the analyte test result data record.	Text	200	

X Required field.
Q Only required if laboratory has qualified the result.
T Only required for tentatively identified compounds by GC/MS.
(1) Field Length indicates decimal precision in parenthesis. For example, 5(2) = a total width of 5 numbers including a maximum of 2 decimal places.

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
ProjectNumber	Project number assigned by the client.	Text	30	X
ProjectName	Project name assigned by the client.	Text	90	X
ClientSampleID	Client or contractor's identifier for a field sample If a sample is analyzed as a laboratory duplicate, matrix spike, or matrix spike duplicate, append suffixes DUP, MS and MSD respectively to the Client Sample ID with no intervening spaces or hyphens (i.e. MW01DUP, MW01MS, and MW01MSD). For Laboratory QC samples (i.e. Method Blanks, LCS, and LCSD, etc.) enter the unique LaboratorySampleID into this field Do not append suffixes to the ClientSampleID for dilutions, reanalyses, or re-extracts (the Analysis_Type field is used for this distinction). For example, MW01DL and MW01RE are not allowed Parent sample records must exist for each MS and MSD. If an MS/MSD is shared between two EDDs, records for the MS/MSD and its parent sample must exist in the Sample Analysis table for both EDDs.	Text	25	X
Collected	Date and Time of sample collection. Refer to the date/time format at the end of this table. Leave this field blank for Method Blank, LCS, and LCSD. For Collected values that are not applicable use the value of 00/00/0000 00:00.	Date/ Time	16*	X
MatrixID	Sample matrix (i.e., AQ, SO, etc.)	Text	10	X
LabSampleID	Laboratory tracking number for field samples	Text	25	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	and lab generated QC samples such as method blank, LCS, and LCSD. There are no restrictions for the LabSampleID except field length and that the LabSampleID must be unique for a given field sample or lab QC sample and method.			
QCType	This record identifies the type of quality control sample QC (i.e., Duplicate, LCS, Method Blank, MS, or MSD). For regular environmental samples, populate this field with "NM".	Text	10	X
ShippingBatchID	Unique identifier assigned to a cooler or shipping container used to transport client or field samples. Links all samples to a cooler or shipping container. No value is required for method blanks, LCS, and LCSD.	Text	25	X
Temperature	Temperature (in centigrade degrees) of the sample as received. The storage refrigerator or room temperature should be reported (in centigrade degrees) for laboratory QC samples (i.e. method blanks, laboratory control standards). Use -99 if temperature is not available. This field is not required for radiochemistry methods.	Numeric (1)	10(6)	X
LabAnalysisRefMethodID	Laboratory reference method ID. The method ID may be an EPA Method number or laboratory identifier for a method such as a SOP number, however; values used for Laboratory Method IDs are specified by the	Text	25	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	project and must in the in standard value list for method IDs.			
PreparationType	Preparation Method Number (i.e., 3010A, 3510C, 3550C, 5030B, etc.) For analytical procedures that do not have a specific preparation method number, use “Gen Prep”.	Text	25	X
AnalysisType	Defines the type of analysis such as initial analysis, dilution, re-analysis, etc. This field provides distinction for sample records when multiple analyses are submitted for the same sample, method, and matrix, for example: dilutions, re-analyses, and re-extracts.	Text	10	X
Prepared	Refer to the date/time format at the end of this table. If no sample preparation is involved enter the analysis date and time in this field. Refer to the date/time format at the end of this table.	Date/ Time	16*	X
Analyzed	Date and time of sample analysis. Refer to the date and time format at the end of this table. For Analyzed values that are not applicable use the value of 00/00/0000 00:00.	Date/ Time	16*	X
LabID	Identification of the laboratory performing the analysis.	Text	7	X
QCLevel	The level of laboratory QC associated with the analysis reported in the EDD. If only the Analytical Results Table (A1) and the Sample Analysis Table (A3) information are submitted for the sample, enter “COA”. If the Laboratory Instrument Table (A2) information is also submitted for the sample, enter “COCAL”	Text	6	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
ResultBasis	Indicates whether results associated with this sample records are reported as wet or percent moisture corrected. Enter “WET” if results are not corrected for percent moisture. Enter “DRY” if percent moisture correction is applied to results. For aqueous samples, enter “WET”. For other matrices where basis is not applicable enter “NA”	Text	3	X
TotalOrDissolved	This field indicates if the results related to this sample record are reported as a total or dissolved fraction. If not applicable please report "NA"	Text	3	X
Dilution	Dilution of the sample aliquot. Enter “1” for method blanks, LCS, and LCSD, or if the field samples was analyzed without dilution.	Numeric (1)	10(6)	X
HandlingType	Indicates the type of leaching procedure, if applicable (i.e., SPLP, TCLP, WET). Enter “NA” if the sample analysis was <u>not</u> performed on a leachate.	Text	10	X
HandlingBatch	Unique laboratory identifier for a batch of samples prepared together in a leaching procedure (i.e., SPLP, TCLP, or WET preparation). The HandlingBatch links samples with leaching blanks. Enter “NA” if the sample analysis was <u>not</u> performed on a leachate.	Text	12	X
LeachateDate	Date and time of leaching procedure (i.e., date for SPLP, TCLP, or WET preparation). Refer to the date and time format at the end of this table.	Date /Time	16*	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	. For Analyzed values that are not applicable use the value of 00/00/0000 00:00			
Percent_Moisture	For soil and sediment samples, enter the percent of sample composed of water. For aqueous samples enter "100". For other matrices where Percent_Moisture is not applicable use a value of -99	Numeric (1)	10(6)	X
MethodBatch	Unique laboratory identifier for a batch of samples of similar matrices analyzed by one method and treated as a group for matrix spike, matrix spike duplicate, or laboratory duplicate association The method batch links the matrix spike and/or matrix spike duplicate or laboratory duplicates to associated samples. Note the MethodBatch association may coincide with the PreparationBatch association. The MethodBatch is specifically used to link the MS/MSD and/or DUP to associated samples.	Text	12	X
PreparationBatch	Unique laboratory identifier for a batch of samples prepared together for analysis by one method and treated as a group for method blank, LCS and LCSD association. The PreparationBatch links method blanks and laboratory control samples (blank spikes) to associated samples. Note, the PreparationBatch association may coincide with the MethodBatch association but the PreparationBatch specifically links the Method Blank and LCS to associated samples.	Text	12	X
RunBatch	<u>For all other methods</u> the RunBatch is the unique identifier for a batch of analyses performed on one instrument under the control	Text	12	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	of one initial calibration and initial calibration verification. The RunBatch links both the initial calibration and initial calibration verification to subsequently analyzed and associated continuing calibrations, field samples, and QC analyses. For GC/MS methods, the RunBatch also links a BFB or DFTPP tune. A distinct RunBatch must used with every new initial calibration within a method The value entered in this field links a particular sample/method/analysis type record to a set of associated initial calibration and initial calibration verification records from Table A2. If Table A2 is not submitted enter a value of 'NA" in this field.			
AnalysisBatch	For radiochemistry methods leave this field blank. For all other methods the AnalysisBatch is the unique identifier for a batch of analyses performed on one instrument and under the control of a continuing calibration or continuing calibration verification. The AnalysisBatch links the continuing calibration or calibration verification to subsequently analyzed and associated field sample and QC analyses. For GC/MS methods, the AnalysisBatch also links the BFB or DFTPP tune. A distinct AnalysisBatch must be used with every new continuing calibration or continuing calibration verification within a method The value entered in this field links a particular sample/method/analysis type record to a set of	Text	12	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	associated continuing calibration records in the Laboratory Instrument table.			
LabReportingBatch	Unique laboratory identifier for the EDD. This is equivalent to the sample delivery group, lab work number, login ID, etc. The LabReportingBatch links all records in the EDD reported as one group. The value entered in this field must be the same in all records.	Text	12	X
LabReceipt	Date and time the sample was received in the lab. A time value of 00:00 may be entered. Refer to the date/time format at the end of this table.	Date/ Time	16*	X
LabReported	Date and time hard copy reported delivered by the lab. A time value of 00:00 may be entered. Refer to the date/time format at the end of this table.	Date/ Time	16*	X
Comment	Add any comments or additional information specific to the sample analysis data record.	Text	200	

C Only required for regular samples, duplicates and MS/MSDs.
X Required field.
(1) Field Length indicates decimal precision in parenthesis. For example, 5(2) = a total width of 5 numbers including a maximum of 2 decimal places.
* Format Date and Time as MM/DD/YYYY hh:mm; where MM = two digit month, DD = two digit day, and YYYY = four digit year, hh = hour in 24 hour format, and mm = minutes.

3.0 Laboratory Data Checker

The Laboratory Data Checker is a web-based application that will review Laboratory Electronic Data Deliverables (LEDDs) for adherence to Tetra Tech's EDD format requirements. EDDs will be reviewed for elements such as missing data and/or columns of data, and compliance of the data within each column to the required data types/lengths.

Once an EDD passes through the checker with no errors, it must be submitted to Tetra Tech through the LEDD Checker application.

Access to the LEDD Checker application will be provided by an initial registration/approval process. An Information Systems Group (ISG) Administrator will approve requests for access**.** To access the site or begin the registration process, visit the ISG web site at http://isg.ttnus.com and select the "Laboratory Checker" link on the left of the home page. Registered users may access the checker immediately by logging in to the system using theircredentials. New users must select the "Register" button and provide all of the requested information**.**

After completing all fields on the registration form, select the "Submit" button to complete the request process. Upon verification by an ISG Administrator, an email notification will be sent verifying the user ID, password and account status. Forgotten passwords may be retrieved by using the "Forgot password?" link on the login page. Note that the email address that was provided for registration or password retrieval is the user ID and must be a valid e-mail address.

The general process for submitting EDD files through the LEDD Checker involves a 3-stage process that includes an upload stage, an error checking stage and a submittal stage.

Log into the LEDD Checker by typing your login credentials and select the "Login" button. The LEDD Checker home page provides a general overview of the checker functionality and EDD file format requirements. At the bottom of the home page, example EDDs are provided that may be viewed or downloaded. To download the files, right click on the link and select "Save target as" from the menu. Each LEDD Checker page includes a navigation bar with links to return to the home page or continue the checking and submittal process. Users should **NOT** use the back or forward buttons on the browser, instead use the links provided in the application to navigate through the site.

Detailed information regarding EDD preparation, formatting requirements and text file naming conventions are provided in the Electronic Data Format Requirements Section of the Laboratory SOW.

Begin the upload stage by selecting the "Upload/Check Files" link on the home page. Follow the steps on the upload page starting with the selection of the laboratory name that corresponds to your organization. If your organization is not listed, contact LabSupport@tetratech.com, and provide a full description of your organization name, contact information and include "Laboratory Contractor ID Request" in the subject line. An ISG Administrator will respond to the request via e-mail.

Load the appropriate A1, A2, or A3 target EDD files by clicking the "Browse" button next to each data table input box. A file browser dialog will appear allowing files to be selected from a local or network drive. After the EDD files are loaded, click the "Upload" button to complete the upload stage. Note that each table may be uploaded and checked separately; however, a minimum of the A1 and A3 files are required in order to submit the EDDs.

If the file upload was successful, the checking page will immediately load. Begin the checking stage by selecting the “Check Files” button. The LEDD Checker will begin validating the EDD files for compliance. Depending on file size and network activity the validation process may take several minutes. The progress should be displayed in the information bar at the bottom of the browser window. **Do not** select the “Check Files” button again or otherwise use the browser during this process. Other applications may be used; however, note that the LEDD Checker may not sit idle for more than 30 minutes. If the time is exceeded a new session must be started in a new browser window.

Any errors will be processed and returned on the error page. The following general errors may be returned.

- Column count / table structure errors – due to column header names being included, improper delimiter, extra tabs, extra or missing columns of data, spaces or other characters at the end of a row.
- Row and column value specific errors – may occur for one or more reasons including: data truncation, invalid date / time format, invalid decimal precision or field width exceedance, or if a value is not in a list of valid values or expected range.

If column count / table structure errors are encountered, the LEDD Checker will return an error and stop the checking process.

The EDDs will not be processed any further until the column errors are resolved. Text fields are validated for truncation. Date / Time fields are validated for truncation and format compliance. Numeric decimal fields are validated for truncation, character type compliance and decimal precision. All required fields are validated for null values or empty text strings (i.e. spaces). The LEDD Checker will return a list of all errors in and include a reference to the row number on which the error occurred. Note that consecutive EDD files may be loaded and checked, and submitted while logged in. However, no data may be submitted until all EDD files have passed through the LEDD Checker without errors. The list of errors may be printed by selecting the “Print this Page” button from the checker error page.

If the EDD files pass with no errors, the submittal page will immediately load. To complete the submittal stage, include the following information in the comment and additional information area of the form: laboratory name, laboratory contact person, project name, project number, site name/number, fractions included and any specific comments related to the EDD. Select the “Submit Files” button to continue the submittal process.

The submittal stage is not considered complete until a unique ticket key reference is returned in the browser window. The ticket key reference must be printed for record of submission and future reference. In addition, a copy of the ticket key reference must be included in the PDF data package.

D-3 GEL

ATTACHMENT NO. 2

STATEMENT OF WORK/PRICE TABLES

TECHNICAL SPECIFICATION FOR LABORATORY SERVICES
NAVSTA NEWPORT, RHODE ISLAND

COMPREHENSIVE LONG-TERM ENVIRONMENTAL ACTION - NAVY (CLEAN)
CONTRACT N62470-08-D-1001, CONTRACT TASK ORDER (CTO) NO. WE61

AGING OF SEDIMENT BEDDING LAYERS
RADIOLOGICAL ANALYSES

1.0 INTRODUCTION

Tetra Tech NUS, Inc. (TtNUS) under CLEAN Contract N62470-08-D-1001 is procuring a laboratory in support of an environmental investigation. Requested analyses include Lead-210.

After award, the laboratory will be required to submit the minimum detectable activities (MDAs) and the quantitation limit (QLs), Standard Operating Procedures (SOP) and all relevant precision and accuracy limits for the analyses required under this scope of work.

The responding laboratory must submit QLs and MDAs for all analyses and matrices requested by filling out the last two columns of Attachment A and including the completed attachment with the bid response. After award, the laboratory will be required to submit Standard Operating Procedures (SOPs) and relevant precision and accuracy limits for all preparation and analytical methods required under this scope of work. The laboratory will also be asked to complete Uniform Federal Policy (UFP) Sampling and Analysis Plan (SAP) Worksheets 19, and 23-28 for inclusion in the SAP. The SAP will be prepared according to the UFP for quality assurance project plans (March 2005) and utilize the 37 worksheets.

2.0 SAMPLE INFORMATION

Samples will be collected by Tetra Tech of marine sediment (soil) by use of coring devices, separated into target layers and provided to the laboratory in appropriate sample containers provided by the laboratory. The approximate number of soil samples to be submitted, the type of analysis to be conducted, and the analytical method to be used are summarized in Table 1.

Samples will be collected from July 25, 2011 to August 26, 2011.

Field duplicate samples will be submitted with "blinded" identification to the laboratory. The field crew will designate samples (one per twenty samples of like matrices) for matrix spike/matrix spike duplicate for the Lead-210 analyses; additional volumes of these samples will be provided as necessary.

All samples are expected to be of low or moderate contaminant concentration. The field crew will attempt to identify any potentially high concentration samples.

3.0 ANALYSIS/REPORTING INFORMATION

One hard copy data package deliverable and two PDF CD copies must be submitted, in addition to the electronic data deliverables in the format described in Attachment C. The original chain-of-custody form received with the samples and signed by the laboratory sample custodian must be returned with the hard copy data package.

The analytical requirements and approximate number of samples to be submitted for analyses are detailed in Table 1. Any analysis and reporting requirements addressed in the DOD Quality Systems Manual (version 4.1, April 2009) and the requested methods must be followed. The data packages must

be complete including raw data and CLP-equivalent summary forms for all field samples and laboratory control samples. The summary forms should include method specific quality control limits (percent recoveries, relative percent differences, relative standard deviations, and/or percent differences etc.).

Additionally, each data package must contain a summary data package. This summary data package shall consist of only the summary forms.

Attachment A is a table of analytes, action limits, and spaces for lab MDAs. Attachment B details the required summary forms for data packages and requirements for organization/bookmarking of PDF data packages.

The laboratory is to provide Minimum Detectable Activities (MDAs) for all radiochemical analyses. The MDAs are to be provided both as inserts to the hardcopy data packages and as a component of the electronic deliverables. MDAs must be derived in accordance with method-specific criteria.

The PDF data package deliverable must contain a detailed case narrative for all analytical fractions. This case narrative must also include the Contract Task Order (CTO) number, the site name, and the TtNUS Project Manager's name. Data from all analytical runs (i.e., original, dilution, re-analysis) must be reported. All soil and sediment matrix sample results shall be reported on an adjusted dry-weight basis.

As stipulated in the CLEAN Basic Ordering Agreement (BOA), Sample Delivery Group (SDG) and fractionally-specific text (TXT) files containing all environmental sample and field quality control blank analysis results must be generated in accordance with the requirements outlined in Attachment B.

As part of the laboratory case narrative, it is required that the Laboratory Quality Assurance Manager sign an attestation statement verifying that all electronic diskette deliverables exactly match the data summary forms (i.e. Form Is).

Maximum holding time allowances, as defined in the following table, are to be strictly observed. Calculation of holding time is in calendar days and is to begin from the time of collection. The holding times are as follows:

Analyses	**Holding Time**
Lead-210	6 months to analysis

The TtNUS Project Manager for this project is Mr. Stephen Parker and he must be contacted in the event of any laboratory problems that could impact project deadlines (i.e., late deliverables, technical problems in the lab that could lead to late deliverables.) To ensure good communication it is required that the laboratory's appointed project manager contact Mr. Parker once a week for the entire project duration.

Contact information for Mr. Parker is as follows:

250 Andover Street, Suite 200
Wilmington, MA 01887-1048
(978) 474-8400
Fax: (978) 474-8499
Email: stephen.parker@tetratech.com

Analytical data turnaround times are to be measured from receipt of each sample shipment. All PDF (2 copies/2 CDs), and associated electronic TXT deliverables are due to the TtNUS Sample Management Coordinator, Ms. Tobrena Sedlmyer, within the standard BOA turnaround term of 21 calendar days from receipt of the last sample in a Sample Delivery Group (SDG). Additionally, all SDGs must contain 20 samples. The frequency in which SDGs contain less than 20 samples should be minimal. All PDF data packages and electronic deliverables must be received at the same time or the deliverable will be considered incomplete and payment deductions may be imposed. Additionally the laboratory Project Manager must fax copies of chain of custody forms to Mrs. Sedlmyer as samples are received by the laboratory.

Contact information for Mrs. Sedlmyer follows:

Tetra Tech NUS, Inc.
Foster Plaza 7
661 Andersen Drive
Pittsburgh, PA 15220
Phone: 412-921-8582
Fax: 412-921-4040
e-mail: tobrena.sedlmyer@tetratech.com

4.0 PERIOD OF PERFORMANCE/BOTTLEWARE INFORMATION

All samples will be shipped to the laboratory via express carrier within 24-hours of collection. Please circle the Yes or No at the bottom of Table 1 which will indicate whether the laboratory will provide courier service at no extra charge.

Bottleware shipments will be coordinated by the field operation leader.

The laboratory is to provide all necessary sample containers **(plus approximately 10% extra for breakage)**. All sample containers must meet ICHEM series 300 cleanliness criteria (or equivalent), and documentation of certified cleanliness must be provided. All of the appropriate sample bottleware must be pre-preserved. The bottleware must be shipped to the designated location in Coleman-like coolers. **The laboratory must also provide any extra coolers needed for return shipment of sample to the laboratory for analysis.** The laboratory is also requested to provide a packing slip indicating the analytical parameters for which each container type is designated, sample labels, custody seals, and chain-of-custody forms. The laboratory must provide temperature blanks in every cooler.

The laboratory must provide Material Safety Data Sheets (MSDSs) for all preservatives sent with each bottleware shipment to the field. MSDSs must be representative of the chemicals provided as preservatives with regard to mixtures and/or purity of the chemicals. For example if a 35% sulfuric acid solution is the preservative, the MSDS provided should be for 35% sulfuric acid solution not 96% sulfuric acid.

5.0 ADDITIONAL COMMENTS/CONTACTS

Within a laboratory, internal transfers of samples, extracts, and digestates must be accomplished and documented as controlled custody transfers. The laboratory must maintain documentation that supports an unbroken chain of custody for samples, digestates and extracts from time of receipt or production in the laboratory until disposal.

The laboratory is to provide a minimum of 60 days storage of sample extracts and 60 days storage of intact sample aliquots, as stipulated in the BOA. **Additionally, the laboratory must store hardcopy and/or PDF data packages for 7 years.**

All analyses conducted under this subcontract assignment are to be performed at the solicited facility only. The laboratory is not permitted to lower-tier subcontract these analyses, or analyze these samples at a corporate facility other than the facility stipulated without prior notification and consent from the CLEAN Subcontracting Officer.

The unit cost for analysis is to include compensation for containers, preservatives, coolers, shipping costs, storage, disposal, and laboratory quality control analyses (such as matrix spike, matrix spike duplicate, laboratory duplicate, and laboratory control sample analyses.) These items are not to be billed as separate line items.

Technical, quality assurance, and data format concerns are to be directed to Ms. Kelly Carper at 412-921-7273 or via e-mail kelly.carper@tetratech.com. Ms. Carper must be contacted and informed of any difficulties encountered during the conduct of the requested analyses.

Questions regarding electronic diskette deliverable concerns are to be directed to Ms. Amy Thomson at 412-921-8182 or via e-mail amy.thomson@tetratech.com. Ms. Thomson must be contacted and informed of any difficulties encountered preparing the required electronic deliverables.

Contract concerns, and response to this solicitation, are to be directed to:

Ms. Meg Price
CLEAN Subcontracting Officer
Tetra Tech NUS, Inc.
234 Mall Boulevard, Suite 260
King of Prussia, PA 19406
Phone: 610-491-9688
Fax: 610-491-9645
e-mail: meg.price@tetratech.com

Triplicate copies of invoices associated with the analyses contracted herein are to be submitted to the attention of the Accounting Supervisor:

Tetra Tech NUS, Inc.
661 Andersen Drive, Foster Plaza 7
Pittsburgh, PA 15220
Phone: 412-921-8506
Fax: 412-921-4040

Please confirm the laboratory's ability to perform the methodologies requested at the analyte detection limits indicated. Also confirm available laboratory capacity, and complete/confirm the costing information indicated in Table 1. **All costing information must reflect the terms and conditions established by the 2010 CLEAN BOA.**

TABLE 1
NUMBER OF SAMPLES/ANALYTICAL METHODS
NAVSTA NEWPORT, RHODE ISLAND
CTO WE61

Media	Parameter	Analytical Method	Number of Samples	Unit Price	Total Price
Soil	Lead-210	alpha spectroscopy (DOE STL-RC-0210).	90	$	$
TOTAL COST					

Can the laboratory provide sample pick-up on site? YES or NO (circle one)
If yes is there an additional charge and if so what is that charge?_____

The laboratory must point out if they are not DOD ELAP accredited for all the methods and analytes requested. Clearly state the methods and or compounds that you are NOT accredited for (if any).

Name of Laboratory_____

Signature_____

ATTACHMENT A

Please specify units.

Analyte	CAS Number	Project Action Limit	Project Action Limit Reference[1]	Project Quantitation Limit Goal	Laboratory-specific	
					QLs	MDAs
Lead-210		----	-----	------		

ATTACHMENT B
Summary Form Requirements for PDF Deliverable and PDF Data Package Deliverables

PDF DATA PACKAGE DELIVERABLE REQUIREMENTS

The laboratory is to provide 2 compact disks (CDs) containing a PDF file in the following format:

1. Table of Contents
2. Case Narrative
3. Chain-of-Custody
4. Data Summary Package (contains summary of all CLP or CLP like Forms 1 through 14 per analytical fraction)
5. Analytical Fractions (VOA, SVOC, General Chemistry, etc.)
a. QC Summary (summary of all CLP or CLP like Forms 1 through 14 for a particular analytical fraction)
b. Raw Sample Data (includes all sample dilutions, sample re-analyses, QC samples, etc.)
c. Calibration Data (includes all initial and continuing calibrations)
d. Miscellaneous (includes extraction forms, IDLs, MDLs, etc.)

Each of the above sections should be bookmarked in the PDF for easy access.

Summary forms for radiological analyses should include the following:

- Form I - Presentation of sample activities and sample uncertainties (2 sigma). Analytical yields should also be presented.
- Summaries of Quality Control Data - Method blank results, laboratory control sample results, laboratory duplicates, and matrix spike results should be summarized in the same format as environmental samples.
- Instrument calibration summaries
- Results of known reference QC samples should be included if analyzed.
- Sample Preparation Summary
- Summaries of sample counts, detector efficiencies, chemical yields, background counts, and time should be provided when applicable.

ATTACHMENT C
ELECTRONIC DATA DELIVERABLE REQUIREMENTS

ELECTRONIC DATA FORMAT REQUIREMENTS

1.0 INTRODUCTION

The laboratory is to submit text-based tab delimited EDD files for each SDG using Tetra Tech's laboratory data checker explained below. The files must be in the format specified in this Attachment. Additional information such as laboratory name, project name, fractions included, project number, site name/number, laboratory contact person and any specific comments related to the EDD should be included in the comments section of the EDD Submittal page.

The RESULT for nondetects should be populated with the project-specific sample quantitation reporting limits (i.e., either the sample quantitation limit or method detection limit, as specified in Section 3.0 of this scope of work. Any corrections made to the hardcopy data must also be made to the electronic file. Appropriate qualifiers as identified by the analytical protocol must also be designated; laboratory QC non-compliance codes are not to be depicted.

Tetra Tech's electronic EDD format follows the ADAPT structure and requires the A1 and A3 files. The A2 file is only required if the project is using ADAPT; and, for non-ADAPT EDD submittals the A2 file may be omitted. The EDD consists of separate, tab-delimited ASCII text files. Each file corresponds to a database table. The tables are identified as the Analytical Results Table (A1) and Sample Analysis Table (A3). A separate set of text files must be created and submitted for each sample delivery group (SDG). The files must be identified to correspond to the (A1) table and the (A3) table. The file naming convention is: the Sample Delivery Group (SDG) followed by the table identifier (A1 or A3), followed by the ".txt" extension. The file names must not contain spaces or special characters. For example, the EDD file names for a laboratory-reporting batch identified as SDG001 would be as follows:

SDG001A1.txt
SDG001A3.txt

On certain projects Tetra Tech will utilize the ADAPT Electronic Data Validation software, which will require the laboratory to use the ADAPT electronic data deliverable checker software prior to submitting the files through Tetra Tech's laboratory data checker (this will be clearly specified in the Tetra Tech laboratory statement of work). The ADAPT checker software can be downloaded from Laboratory Data Consultants' web site: http://www.lab-data.com. For projects which Tetra Tech is using the ADAPT software, Tetra Tech will provide the laboratory with the project library. The laboratory is not permitted to modify the project library. ADAPT projects will require the laboratory to export all three checked files (A1, A2, and A3) from the ADAPT software and submit them through Tetra Tech's laboratory data checker. **ADAPT error logs generated must be included with the electronic PDF data validation package and cannot be submitted through the laboratory data checker.**

The values reported in the EDD text files must agree exactly with the final values reported on the PDF data package sample result summaries. The details of file naming conventions, data structure and data checker use are discussed below.

Analytical Results Table (A1 File)

The Analytical Results table contains analytical results and related information for target analytes in field samples and associated laboratory quality control samples (excluding calibrations and tunes). Field samples and laboratory method blanks must report a result record for each analyte reported within a method. Laboratory control samples (LCS and LCSD) and matrix spike samples (MS and MSD) must report a result record for every analyte specified as a spiked analyte in the laboratory statement of work. Table A1 in this document lists the field names and data type descriptions for the Analytical Results Table (A1).

Lab Instrument Table (A2 File)

A2 file is only required if the project is using ADAPT. In all other EDD submittals, the A2 file may be omitted. Laboratories should refer to the ADAPT User Guide for populating the A2 Table.

Sample Analysis Table (A3 File)

The Sample Analysis table contains information specific to field environmental samples and laboratory quality control analyses (excluding calibrations and tunes). A sample record must exist for each sample/method/matrix/analysis type combination. Table A3 in this document lists the field names and data type descriptions for the Sample Analysis Table (A3).

All electronic data deliverables are due within the same time established for the associated hardcopy data packages.

In addition, the laboratory QC officer must read and sign a copy of the Quality Assurance Review Form displayed on the next page of this Attachment. Electronic deliverables are not considered to be complete without the accompanying Quality Assurance Review Form.

I _____, as the designated Quality Assurance Officer, hereby attest that all electronic deliverables have been thoroughly reviewed and are in agreement with the associated hardcopy data. The enclosed electronic files have been reviewed for accuracy (including significant figures), completeness and format. The laboratory will be responsible for any labor time necessary to correct enclosed electronic deliverables that have been found to be in error. I can be reached at (____)_____if there are any questions or problems with the enclosed electronic deliverables.

Signature:_____ Title: _____Date: _____

2.0 EDD Field Properties

Tables A1 and A3 in this document specify the EDD field properties. Laboratories should refer to the ADAPT User Guide for populating the A2 Table. These include the field name, sequence order, field description, data type/length and reporting requirement for each field. Fields in the EDD **must** be sequenced according to the order that they appear below in Tables A1 and A3. For example, in the Analytical Results table (A1), the field "ClientSampleID" will always be the first piece of information to start every new line of data (or database record), followed by the field "LabAnalysisRefMethodID", "AnalysisType", etc.

When creating an EDD as a text file, use the ASCII character set in a file of lines terminated by a carriage return and line feed. No extra characters are allowed at the end of a line, after the carriage return and line feed. Enclose each data value with double quotes (text qualifier) and separate each field value with a **tab** character (tab delimiter). Data fields with no information (null) may be represented by two consecutive tabs. For example, in the Sample Analysis table, since the "Collected", "ShippingBatchID", and "Temperature" fields do not apply to laboratory generated QA/QC samples, the record for a Laboratory Control Sample by Method 8270C would be entered as follows. Note that the first two fields ("ProjectNumber" and "ProjectName") are omitted in this example.

…"LCSW100598" "AQ" "LCSW100598" "LCS" "8270C",…etc.

If a field is populated with less than the maximum allowed number of characters, do not pad the values with leading or trailing spaces. In the above example, although the "MatrixID" field can accommodate up to 10 characters, only 2 characters were entered in this field. **Do not include the delimiter (tab character) within any of the field values.** Example EDD files may be downloaded from the LEDD Checker application.

An example database shall be sent for review prior to the first electronic deliverable in the required .txt format. The example file will be examined for completeness and comments will be sent to the laboratory. Any questions regarding the electronic deliverable should be directed to LabSupport@tetratech.com

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
ClientSampleID	Client or contractor's identifier for a field sample If a sample is analyzed as a laboratory duplicate, matrix spike, or matrix spike duplicate, append suffixes DUP, MS and MSD respectively to the Client Sample ID with no intervening spaces or hyphens (i.e. MW01DUP, MW01MS, and MW01MSD). For Method Blanks, LCS, and LCSD enter the unique LaboratorySampleID into this field. Do not append suffixes to the ClientSampleID for dilutions, reanalyses, or re-extracts (the AnalysisType field is used for this distinction). For example, MW01DL and MW01RE are not allowed. Parent sample records must exist for each MS and MSD. If an MS/MSD is shared between two EDDs, records for the MS/MSD and its parent sample must exist in the Analytical Results table for both EDDs.	Text	25	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
LabAnalysisRefMethodID	Laboratory reference method ID. The method ID may be an EPA Method number or a Lab Identifier for a method such as a SOP Number, however; method ID is specified by the project. The method ID must be entered into the standard list.	Text	25	X
AnalysisType	Defines the analysis type (i.e., Dilution, Reanalysis, etc.). This field provides distinction for sample result records when multiple analyses are submitted for the same sample, method, and matrix; for example dilutions, re-analyses, and re-extracts.	Text	10	X
LabSampleID	Laboratory tracking number for field samples and lab generated QC samples such as method blank, LCS, and LCSD. There are no restrictions for the LabSampleID except for field length and that the LabSampleID must be distinct for a given field sample or lab QC sample and method. Suffixes may be applied to the LabSampleID to designate dilutions, reanalysis, etc.	Text	25	X
LabID	Identification of the laboratory performing the analyses.	Text	7	X
ClientAnalyteID	CAS Number or unique client identifier for an analyte or isotope. If a CAS Number is not available, use a unique identifier provided by the client or contractor. The ClientAnalyteID for a particular target analyte or isotope should be specified by the project and must exist in the standard value tables for Analytes. For the LCS, LCSD, MS, and MSD, it is only necessary to report the compounds designated as spikes in the library (and surrogates for organic methods.) For TICs from GC/MS analyses, enter the retention time in decimal minutes as the Client Analyte ID.	Text	12	X
AnalyteName	Chemical name for the analyte or isotope. The project specifies how an analyte or isotope is named. The analyte name must be associated to a	Text	60	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	ClientAnalyteID in the standard values table for Analytes (excluding compounds designated as TIC's).			
Result	Result value for the analyte or isotope. Entries must be numeric. For non-detects of target analytes or isotopes and spikes, do not enter "ND" or "0". Do not leave this field blank. If an analyte or spike was not detected, enter the associated value specified in Section 3.0 of this scope of work (e.g., LOD, SQL, PQL, etc.), corrected for dilution and percent moisture as applicable. Do not enter "0". A "0" result may be acceptable for surrogate or internal standard percent recoveries; however, it should not be reported for any target compound.	Numeric (1)	20(6)	X
ResultUnits	The units defining how the values in the Result, DetectionLimit, and ReportingLimit fields are expressed. For radiochemistry this also includes how the value in the Error field is expressed.	Text	10	X
LabQualifiers	A string of single letter result qualifiers assigned by the lab based on client-defined rules and values. The "U" Lab Qualifier must be entered for all non-detects. Other pertinent lab qualifiers may be entered with the "U" qualifier. Order is insignificant. Lab qualifiers other than those listed in the standard values table may be used. If so, these must be added to the standard value table in the application.	Text	7	Q
DetectionLimit	For radiochemistry methods, the minimum detectable activity for the isotope being measured. For all other methods: The minimum detection limit value for the analyte being measured. For surrogates, internal standards, etc. where detection limits are not applicable use the value -99.	Numeric (1)	10(6)	X
DetectionLimitType	Specifies the type of detection limit (i.e., MDA, MDL, IDL, etc.). If -99 is specified in the DetectionLimit field us the	Text	10	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	value NA.			
RetentionTime or Error	For radiochemistry methods only, enter the 2 Sigma Counting Errors. The units for error are entered in the ResultUnits field. For GC/MS methods only, enter the time expressed in decimal minutes between injection and detection for GC/MS TICs only For target analytes in all other methods, leave this field blank. Note: GC retention times are not evaluated at this time.	Text	5	T
AnalyteType	Defines the type of result, such as tracer, surrogate, spike, or target compound.	Text	7	X
PercentRecovery	For radiochemistry methods: The tracer yield, if applicable. For all other analytical methods: The percent recovery value of a spiked compound or surrogate. If the spike or surrogate was not recovered because of dilution, enter “DIL”. If a spike or surrogate was not recovered because of matrix interference, enter “INT”. If a spike or surrogate was not recovered because it was not added to the sample, enter “NS”.	Numeric (1)	5(3)	X
RelativePercentDifference	The relative percent difference (RPD) of two QC results, such as MS/MSD, LCS/LCSD, and Laboratory Duplicates. Report RPD in Laboratory Duplicate, LCSD, and MSD records only.	Numeric (1)	5(3)	X
ReportingLimit	Reporting limit value for the measured analyte or isotope Factor in the dilution factor and percent moisture correction, if applicable. The Reporting Limit for each analyte and matrix in a given method is specified in the project library or QAPP. For surrogates, internal standards, etc. where reporting limits are not applicable use the value -99.	Numeric (1)	10(6)	X
ReportingLimitType	Specifies the type of reporting limit (i.e., CRQL, PQL, SQL, RDL, etc). The Reporting Limit Type for each	Text	10	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	method and matrix is specified in the project library or QAPP. If -99 is specified in the ReportingLimit field us the value NA.			
ReportableResult	This field indicates whether or not the laboratory chooses an individual analyte or isotope result as reportable. Enter "YES" if the result is reportable. Enter "NO" if the result is not reportable. If only one analysis is submitted for a particular sample and method, enter "YES" for all target compounds (where Analyte Type = TRG). For GC/MS methods enter yes for tentatively identified compounds (where Analyte Type = TIC). If two or more analyses are submitted for a particular sample and method (i.e. initial analysis, reanalysis and/or dilutions), enter "YES" from only <u>one</u> of the analyses for each target compound. For example: a sample was run a second time at dilution because benzene exceeded the calibration range in the initial, undiluted analysis. All target analytes are reported in each analysis. For the initial analysis, (Analysis Type = RES), enter "NO" for benzene and enter "YES" for all other compounds. For the diluted analysis (Analysis Type = DL), enter "YES" for benzene and enter "NO" for all other compounds. For TICs (Analyte Type = TIC), if more than one analysis is submitted for a particular sample and method, choose only one of the analyses where Reportable Result = YES for <u>all</u> TICs. For example, a sample was run a second time because one or more target compounds exceeded the calibration range in the undiluted analysis. Choose a particular analysis and enter "YES" for all TICS. In the other analysis enter "NO" for all TICs. Note that it is not necessary to report the full target analyte list for the initial result, dilution, re-analysis, or re-extraction. However, each target analyte must be reported YES once and once only in the case of multiple analyses for a given sample, method, and matrix. In the case of organics, all surrogates must be reported for all analyses submitted for a given	Text	3	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	sample, method, and, matrix.			
SpkConcnAdded	The spike added. This value must be reported in the same units as the result. Where (SA) in the following equation: % Recovery = (SSA-SC)/SA x 100% where : SSA is the spiked sample concentration (amount) after spiking. SC is the sample concentration (amount) before spiking. SA is the the expected increase in sample concentration (amount) as a result of spiking. This value must incorporate all correction factors such as dilution factor and moisture content that are applied to the spiked sample when computing the spiked sample concentration or amount. Enter -99 where no spike was added.	Numeric (1)	10(6)	X
SpkParentSampleID	The sampleID of a sample (often called the original sample) that receives a spike aliquot to form a spiked sample such as a matrix spike. This is not the same as the ID of the spiked sample (such as a matrix spike) after spiking. The result for SpkParentSampleID and the result (i.e., SpkConcnAdded) for the spiked sample are used to compute percent recovery of the analyte.	Text	25	X
SamplePrepInitial	The initial sample preparation volume in liters (L) for aqueous samples or grams (g) for solid samples.	Numeric (1)	20(6)	
SamplePrepFinal	The final sample preparation volume in liters (L) for aqueous samples or grams (g) for solid samples.	Numeric (1)	20(6)	
LimitOfDetection	The smallest amount or concentration of a substance that must be present in a sample in order to be detected at a 99% confidence level. In other words, if a sample has a true concentration at the LOD, there is a minimum probability of 99% of reporting a "detection" (a measured value ≥ DL) and a 1% chance of reporting a non-detect (a false negative).	Numeric (1)	10(6)	N
Comment	Add any comments or additional information specific to the analyte test result data record.	Text	200	

X Required field.
Q Only required if laboratory has qualified the result.
T Only required for tentatively identified compounds by GC/MS.

(1) Field Length indicates decimal precision in parenthesis. For example, 5(2) = a total width of 5 numbers including a maximum of 2 decimal places.

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
ProjectNumber	Project number assigned by the client.	Text	30	X
ProjectName	Project name assigned by the client.	Text	90	X
ClientSampleID	Client or contractor's identifier for a field sample If a sample is analyzed as a laboratory duplicate, matrix spike, or matrix spike duplicate, append suffixes DUP, MS and MSD respectively to the Client Sample ID with no intervening spaces or hyphens (i.e. MW01DUP, MW01MS, and MW01MSD). For Laboratory QC samples (i.e. Method Blanks, LCS, and LCSD, etc.) enter the unique LaboratorySampleID into this field Do not append suffixes to the ClientSampleID for dilutions, reanalyses, or re-extracts (the Analysis_Type field is used for this distinction). For example, MW01DL and MW01RE are not allowed Parent sample records must exist for each MS and MSD. If an MS/MSD is shared between two EDDs, records for the MS/MSD and its parent sample must exist in the Sample Analysis table for both EDDs.	Text	25	X
Collected	Date and Time of sample collection. Refer to the date/time format at the end of this table. Leave this field blank for Method Blank, LCS, and LCSD. For Collected values that are not applicable use the value of 00/00/0000 00:00.	Date/ Time	16*	X
MatrixID	Sample matrix (i.e., AQ, SO, etc.)	Text	10	X
LabSampleID	Laboratory tracking number for field samples and lab generated QC samples such as method blank, LCS, and LCSD. There are no restrictions for the LabSampleID except field length and that the LabSampleID must be unique for a given field sample or lab QC sample and method.	Text	25	X
QCType	This record identifies the type of quality control sample QC (i.e., Duplicate, LCS, Method Blank, MS, or MSD). For regular environmental samples, populate this field with "NM".	Text	10	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
ShippingBatchID	Unique identifier assigned to a cooler or shipping container used to transport client or field samples. Links all samples to a cooler or shipping container. No value is required for method blanks, LCS, and LCSD.	Text	25	X
Temperature	Temperature (in centigrade degrees) of the sample as received. The storage refrigerator or room temperature should be reported (in centigrade degrees) for laboratory QC samples (i.e. method blanks, laboratory control standards). Use -99 if temperature is not available. This field is not required for radiochemistry methods.	Numeric (1)	10(6)	X
LabAnalysisRefMethodID	Laboratory reference method ID. The method ID may be an EPA Method number or laboratory identifier for a method such as a SOP number, however; values used for Laboratory Method IDs are specified by the project and must in the in standard value list for method IDs.	Text	25	X
PreparationType	Preparation Method Number (i.e., 3010A, 3510C, 3550C, 5030B, etc.) For analytical procedures that do not have a specific preparation method number, use “Gen Prep”.	Text	25	X
AnalysisType	Defines the type of analysis such as initial analysis, dilution, re-analysis, etc. This field provides distinction for sample records when multiple analyses are submitted for the same sample, method, and matrix, for example: dilutions, re-analyses, and re-extracts.	Text	10	X
Prepared	Refer to the date/time format at the end of this table. If no sample preparation is involved enter the analysis date and time in this field. Refer to the date/time format at the end of this table.	Date/ Time	16*	X
Analyzed	Date and time of sample analysis. Refer to the date and time format at the end of this table. For Analyzed values that are not applicable use the value of 00/00/0000 00:00.	Date/ Time	16*	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
LabID	Identification of the laboratory performing the analysis.	Text	7	X
QCLevel	The level of laboratory QC associated with the analysis reported in the EDD. If only the Analytical Results Table (A1) and the Sample Analysis Table (A3) information are submitted for the sample, enter “COA”. If the Laboratory Instrument Table (A2) information is also submitted for the sample, enter “COCAL”	Text	6	X
ResultBasis	Indicates whether results associated with this sample records are reported as wet or percent moisture corrected. Enter “WET” if results are not corrected for percent moisture. Enter “DRY” if percent moisture correction is applied to results. For aqueous samples, enter “WET”. For other matrices where basis is not applicable enter “NA”	Text	3	X
TotalOrDissolved	This field indicates if the results related to this sample record are reported as a total or dissolved fraction. If not applicable please report "NA"	Text	3	X
Dilution	Dilution of the sample aliquot. Enter “1” for method blanks, LCS, and LCSD, or if the field samples was analyzed without dilution.	Numeric (1)	10(6)	X
HandlingType	Indicates the type of leaching procedure, if applicable (i.e., SPLP, TCLP, WET). Enter “NA” if the sample analysis was <u>not</u> performed on a leachate.	Text	10	X
HandlingBatch	Unique laboratory identifier for a batch of samples prepared together in a leaching procedure (i.e., SPLP, TCLP, or WET preparation). The HandlingBatch links samples with leaching blanks. Enter “NA” if the sample analysis was <u>not</u> performed on a leachate.	Text	12	X
LeachateDate	Date and time of leaching procedure (i.e., date for SPLP, TCLP, or WET preparation). Refer to the date and time format at the end of this table. . For Analyzed values that are not applicable use the value of 00/00/0000 00:00	Date /Time	16*	X
Percent_Moisture	For soil and sediment samples, enter the percent of	Numeric (1)	10(6)	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	sample composed of water. For aqueous samples enter "100". For other matrices where Percent_Moisture is not applicable use a value of -99			
MethodBatch	Unique laboratory identifier for a batch of samples of similar matrices analyzed by one method and treated as a group for matrix spike, matrix spike duplicate, or laboratory duplicate association The method batch links the matrix spike and/or matrix spike duplicate or laboratory duplicates to associated samples. Note the MethodBatch association may coincide with the PreparationBatch association. The MethodBatch is specifically used to link the MS/MSD and/or DUP to associated samples.	Text	12	X
PreparationBatch	Unique laboratory identifier for a batch of samples prepared together for analysis by one method and treated as a group for method blank, LCS and LCSD association. The PreparationBatch links method blanks and laboratory control samples (blank spikes) to associated samples. Note, the PreparationBatch association may coincide with the MethodBatch association but the PreparationBatch specifically links the Method Blank and LCS to associated samples.	Text	12	X
RunBatch	<u>For all other methods</u> the RunBatch is the unique identifier for a batch of analyses performed on one instrument under the control of one initial calibration and initial calibration verification. The RunBatch links both the initial calibration and initial calibration verification to subsequently analyzed and associated continuing calibrations, field samples, and QC analyses. For GC/MS methods, the RunBatch also links a BFB or DFTPP tune. A distinct RunBatch must used with every new initial calibration within a method The value entered in this field links a particular sample/method/analysis type record to a set of associated initial calibration and initial calibration verification records from Table A2. If Table A2 is not submitted enter a value of 'NA" in this field.	Text	12	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
AnalysisBatch	For radiochemistry methods leave this field blank. For all other methods the AnalysisBatch is the unique identifier for a batch of analyses performed on one instrument and under the control of a continuing calibration or continuing calibration verification. The AnalysisBatch links the continuing calibration or calibration verification to subsequently analyzed and associated field sample and QC analyses. For GC/MS methods, the AnalysisBatch also links the BFB or DFTPP tune. A distinct AnalysisBatch must be used with every new continuing calibration or continuing calibration verification within a method The value entered in this field links a particular sample/method/analysis type record to a set of associated continuing calibration records in the Laboratory Instrument table.	Text	12	X
LabReportingBatch	Unique laboratory identifier for the EDD. This is equivalent to the sample delivery group, lab work number, login ID, etc. The LabReportingBatch links all records in the EDD reported as one group. The value entered in this field must be the same in all records.	Text	12	X
LabReceipt	Date and time the sample was received in the lab. A time value of 00:00 may be entered. Refer to the date/time format at the end of this table.	Date/ Time	16*	X
LabReported	Date and time hard copy reported delivered by the lab. A time value of 00:00 may be entered. Refer to the date/time format at the end of this table.	Date/ Time	16*	X
Comment	Add any comments or additional information specific to the sample analysis data record.	Text	200	

C Only required for regular samples, duplicates and MS/MSDs.
X Required field.
(1) Field Length indicates decimal precision in parenthesis. For example, 5(2) = a total width of 5 numbers including a maximum of 2 decimal places.
* Format Date and Time as MM/DD/YYYY hh:mm; where MM = two digit month, DD = two digit day, and YYYY = four digit year, hh = hour in 24 hour format, and mm = minutes.

3.0 Laboratory Data Checker

The Laboratory Data Checker is a web-based application that will review Laboratory Electronic Data Deliverables (LEDDs) for adherence to Tetra Tech's EDD format requirements. EDDs will be reviewed for elements such as missing data and/or columns of data, and compliance of the data within each column to the required data types/lengths. Once an EDD passes through the checker with no errors, it must be submitted to Tetra Tech through the LEDD Checker application.

Access to the LEDD Checker application will be provided by an initial registration/approval process. An Information Systems Group (ISG) Administrator will approve requests for access**.** To access the site or begin the registration process, visit the ISG web site at http://isg.ttnus.com and select the "Laboratory Checker" link on the left of the home page. Registered users may access the checker immediately by logging in to the system using theircredentials. New users must select the "Register" button and provide all of the requested information**.**

After completing all fields on the registration form, select the "Submit" button to complete the request process. Upon verification by an ISG Administrator, an email notification will be sent verifying the user ID, password and account status. Forgotten passwords may be retrieved by using the "Forgot password?" link on the login page. Note that the email address that was provided for registration or password retrieval is the user ID and must be a valid e-mail address.

The general process for submitting EDD files through the LEDD Checker involves a 3-stage process that includes an upload stage, an error checking stage and a submittal stage.

Log into the LEDD Checker by typing your login credentials and select the "Login" button. The LEDD Checker home page provides a general overview of the checker functionality and EDD file format requirements. At the bottom of the home page, example EDDs are provided that may be viewed or downloaded. To download the files, right click on the link and select "Save target as" from the menu. Each LEDD Checker page includes a navigation bar with links to return to the home page or continue the checking and submittal process. Users should **NOT** use the back or forward buttons on the browser, instead use the links provided in the application to navigate through the site.

Detailed information regarding EDD preparation, formatting requirements and text file naming conventions are provided in the Electronic Data Format Requirements Section of the Laboratory SOW.

Begin the upload stage by selecting the "Upload/Check Files" link on the home page. Follow the steps on the upload page starting with the selection of the laboratory name that corresponds to your organization. If your organization is not listed, contact LabSupport@tetratech.com, and provide a full description of your organization name, contact information and include "Laboratory Contractor ID Request" in the subject line. An ISG Administrator will respond to the request via e-mail.

Load the appropriate A1, A2, or A3 target EDD files by clicking the "Browse" button next to each data table input box. A file browser dialog will appear allowing files to be selected from a local or network drive. After the EDD files are loaded, click the "Upload" button to complete the upload stage. Note that each table may be uploaded and checked separately; however, a minimum of the A1 and A3 files are required in order to submit the EDDs.

If the file upload was successful, the checking page will immediately load. Begin the checking stage by selecting the "Check Files" button. The LEDD Checker will begin validating the EDD files for compliance. Depending on file size and network activity the validation process may take several minutes. The progress should be displayed in the information bar at the bottom of the browser window. **Do not** select the "Check Files" button again or otherwise use the browser during this process. Other applications may be used; however, note that the LEDD Checker may not sit idle

for more than 30 minutes. If the time is exceeded a new session must be started in a new browser window.

Any errors will be processed and returned on the error page. The following general errors may be returned.

- Column count / table structure errors – due to column header names being included, improper delimiter, extra tabs, extra or missing columns of data, spaces or other characters at the end of a row.
- Row and column value specific errors – may occur for one or more reasons including: data truncation, invalid date / time format, invalid decimal precision or field width exceedance, or if a value is not in a list of valid values or expected range.

If column count / table structure errors are encountered, the LEDD Checker will return an error and stop the checking process.

The EDDs will not be processed any further until the column errors are resolved. Text fields are validated for truncation. Date / Time fields are validated for truncation and format compliance. Numeric decimal fields are validated for truncation, character type compliance and decimal precision. All required fields are validated for null values or empty text strings (i.e. spaces). The LEDD Checker will return a list of all errors in and include a reference to the row number on which the error occurred. Note that consecutive EDD files may be loaded and checked, and submitted while logged in. However, no data may be submitted until all EDD files have passed through the LEDD Checker without errors. The list of errors may be printed by selecting the “Print this Page” button from the checker error page.

If the EDD files pass with no errors, the submittal page will immediately load. To complete the submittal stage, include the following information in the comment and additional information area of the form: laboratory name, laboratory contact person, project name, project number, site name/number, fractions included and any specific comments related to the EDD. Select the “Submit Files” button to continue the submittal process.

The submittal stage is not considered complete until a unique ticket key reference is returned in the browser window. The ticket key reference must be printed for record of submission and future reference. In addition, a copy of the ticket key reference must be included in the PDF data package.

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005

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ENVIRONMENTAL

Valid To: August 31, 2011 Certificate Number: 2567.01

In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with ISO IEC 17025:2005, the 2003 NELAC Chapter 5 Standard, 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)) accreditation is granted to this laboratory to perform the following radiochemical tests in various matrices, including soils, drinking water, wastewater, groundwater, fiber air filters, vegetation, animal tissues and milk.

	Preparation SOP	Analytical SOP
Alpha Spectrometry: Alpha: Am-241, Am-243, Cf-252, Cm-242, Cm-243/244, Cm-245/246, Np-237, Po-208, Po-209, Po-210, Pu-236, Pu-238, Pu-239/240, Pu-242, Pu-244, Th-228, Th-229, Th-230, Th-232, U-232, U-233/234, U-235/236, U-238	GL-RAD-A-011, GL-RAD-A-016, GL-RAD-A-032, GL-RAD-A-036, GL-RAD-A-038, GL-RAD-A-043, GL-RAD-A-045	GL-RAD-I-009
Radon Emanation: Ra-226	GL-RAD-A-008, GL-RAD-A-028	GL-RAD-I-007
Gamma Spectrometry: Gamma: 46 to 1836 keV, I-129, I-131, Ni-59	GL-RAD-A-006, GL-RAD-A-013, GL-RAD-A-022	GL-RAD-I-001
Kinetic Phosphorescence Analyzer Total Uranium	GL-RAD-A-023	GL-RAD-B-018

	Preparation SOP	Analytical SOP
Gas Flow Proportional Counting: Beta: Cl-36, I-131, Pb-210, Ra-228, Sr-89, Sr-90, Total Radium	GL-RAD-A-004, GL-RAD-A-009, GL-RAD-A-010, GL-RAD-A-017, GL-RAD-A-018, GL-RAD-A-029, GL-RAD-A-030, GL-RAD-A-033	GL-RAD-I-006, GL-RAD-I-015, GL-RAD-I-016
Gross Alpha/Gross Beta:	GL-RAD-A-001, GL-RAD-A-001B, GL-RAD-A-001C	GL-RAD-I-006, GL-RAD-I-015, GL-RAD-I-016
48 hour Gross Alpha	GL-RAD-A-047	GL-RAD-I-006, GL-RAD-I-015, GL-RAD-I-016
Liquid Scintillation Spectrometry: Beta: C-14, Ca-45, Fe-55, H-3, Ni-63, P-32, Pm-147, Pu-241, S-35, Se-79, Tc-99 Alpha: Rn-222	GL-RAD-A-002, GL-RAD-A-003, GL-RAD-A-005, GL-RAD-A-007, GL-RAD-A-019, GL-RAD-A-020, GL-RAD-A-022, GL-RAD-A-031, GL-RAD-A-035, GL-RAD-A-040, GL-RAD-A-048, GL-RAD-A-049, GL-RAD-A-050	GL-RAD-I-004, GL-RAD-I-014, GL-RAD-I-017
ICP-MS Uranium Isotopes Tc-99	 GL-MA-E-008 GL-RAD-A-005	 GL-MA-E-014 GL-RAD-B-034

Additionally, in recognition of the successful completion of the A2LA evaluation process, including an assessment of the laboratory's compliance with ISO IEC 17025:2005, the 2003 NELAC Chapter 5 Standard, 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), accreditation is granted to this laboratory to perform recognized EPA, Standard Methods for the Examination of Water and Wastewater, ASTM, Department of Energy (DOE), California and Connecticut test methods using the following testing technologies and in the analyte categories identified below:

Testing Technologies

Atomic Absorption/ICP-AES Spectrometry, ICP/MS, Gas Chromatography, Gas Chromatography/Mass Spectrometry, Gravimetry, High Performance Liquid Chromatography, Ion Chromatography, Methylene Blue Active Substances, Misc.- Electronic Probes (pH, O_2), Oxygen Demand, Hazardous Waste Characteristics Tests, Spectrophotometry (Visible), Spectrophotometry (Automated), IR Spectrometry, Titrimetry, Total Organic Carbon, Total Organic Halide, Turbidity, Liquid Chromatography/Mass Spectrometer/Mass Spectrometer and Various Radiochemistry Techniques

Parameter/Analyte	Nonpotable Water	Solid Hazardous Waste (Liquids and Solids)
Metals		
Aluminum	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Antimony	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Arsenic	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Barium	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Beryllium	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Boron	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Cadmium	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Calcium	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Chromium	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Cobalt	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Copper	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Iron	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Lead	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Lithium	EPA 6020/6020A	EPA 6020/6020A
Magnesium	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Manganese	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Mercury	EPA 1631E/7470/7470A/245.1	EPA 7470/7470A/7471A/7471B
Molybdenum	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Nickel	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Phosphorous	EPA 6020/6020A	EPA 6010B/6010C/6020/6020A
Potassium	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Selenium	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Silicon[1]	EPA 6010B/6010C modified	EPA 6010B/6010C modified
Silica as SiO2	EPA 6010B/6010C	EPA 6010B/6010C
Silver	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Sodium	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Strontium	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Thallium	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Tin	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Titanium	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Tungsten	----------	EPA 6020/6020A
Vanadium	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Zinc	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
General Chemistry		
Acidity	SM 2310 B/EPA 305.1	----------
Adsorbable Organic Halogens (AOX)	EPA 1650	----------

Parameter/Analyte	Nonpotable Water	Solid Hazardous Waste (Liquids and Solids)
Alkalinity	SM 2320B/EPA 310.1	----------
Ammenable Cyanide	EPA 9012A/9012B/EPA 335.1	EPA 9012A/9012B
Ammonia Nitrogen	EPA 350.1	----------
Biochemical oxygen demand	SM 5210 B/EPA 405.1	----------
Bromide	EPA 9056A/EPA 300.0	EPA 9056A[3]
Carbonaceous BOD	SM 5210 B	----------
Chemical Oxygen Demand (COD)	EPA 410.4	----------
Chloride	EPA 9056A/EPA 300.0	EPA 9056A[3]
Chlorine (residual)	SM 4500Cl-G/EPA 330.5	----------
Chromium VI	EPA 7196A/SM 3500Cr-B	EPA 7196A
Color	SM 2120B/EPA 110.2	----------
Corrosivity toward Steel	----------	EPA 1110/1110A
Cyanide	EPA 9012A/9012B/335.3/335.4	EPA 9012A/9012B
Density	----------	ASTM D 5057
Extractable Organic Halides (EOX)	----------	EPA 9023
Filterable residue	SM 2540C	----------
Fluoride	EPA 9056A/EPA 300.0	EPA 9056A[3]
Ignitability	EPA 1010/1020A/1020B	EPA 1010/1020A/1020B
Hardness	SM 2340B/SM2340C/ EPA 130.2	----------
Kjeldahl Nitrogen	EPA 351.2	----------
MBAS/Surfactants	SM 5540C/EPA 425.1	----------
Moisture Determination	----------	ASTM D-2216 (M)
Nitrate (as N)	EPA 9056A/EPA 300.0	EPA 9056A[3]
Nitrate-nitrite (as N)	EPA 9056A/EPA 300.0	EPA 9056A[3]
Nitrite (as N)	EPA 9056A/EPA 300.0	EPA 9056A[3]
Nonfilterable residue	SM 2540D	----------
Oil & Grease	EPA 1664A	EPA 1664A
Organic Nitrogen	TKN – Ammonia EPA 351.2 – EPA 350.1	----------
Orthophosphate (as P)	EPA 9056A/EPA 300.0	EPA 9056A[3]
Paint Filter Liquids Test	----------	EPA 9095A/9095B
Perchlorate	EPA 314.0/6850	EPA 6850
pH	SM 4500-H^+ B/ EPA 9040B /9040C/9041A/ EPA 150.1	EPA 9040B/9040C/9045C/9045D
Reactive Cyanide	Sec 7.3.3 SW846	Sec 7.3.3 SW846
Reactive Sulfide	Sec 7.3.4 SW846	Sec 7.3.4 SW846
Residue-Volatile	SM 2540E/EPA 160.4	----------
Residue-Settleable	SM 2540F	----------
Specific conductance	EPA 9050A/EPA 120.1	----------
Sulfate	EPA 9056A/EPA 300.0	EPA 9056A[3]
Sulfite	SM 4500-SO3 B	----------
Sulfide	EPA 9030B/9034	EPA 9030B/9034
Temperature	EPA 170.1	EPA 170.1
Total, fixed, and volatile residue	SM 2540G	----------
Total Nitrate-Nitrite	EPA 353.2	----------

Parameter/Analyte	Nonpotable Water	Solid Hazardous Waste (Liquids and Solids)
Total Organic Carbon (TOC)	EPA 9060/9060A/ SM 5310D/415.1	EPA 9060/9060A[2]
Total Organic Halides (TOX)	EPA 9020B	EPA 9020B[2]
Total Petroleum Hydrocarbons	EPA 1664A	EPA 1664A
Total Phenolics	EPA 9066/EPA 420.4	----------
Total Phosphorous	EPA 365.4	----------
Total residue	SM 2540B	----------
Turbidity	EPA 180.1/SM 2130	EPA 180.1/SM 2130
Organic Analytes		
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8011/8260B	EPA 8260B
1,2 Dibromoethane (EDB)	EPA 8011/8260B	EPA 8260B
Purgeable Organics (Volatiles)		
Acetone	EPA 8260B	EPA 8260B
Acetonitrile	EPA 8260B	EPA 8260B
Acrolein (Propenal)	EPA 8260B	EPA 8260B
Acrylonitrile	EPA 8260B	EPA 8260B
Allyl Chloride	EPA 8260B	EPA 8260B
Benzene	EPA 8260B	EPA 8260B
Benzyl chloride	EPA 8260B	EPA 8260B
Bromobenzene	EPA 8260B	EPA 8260B
Bromochloromethane	EPA 8260B	EPA 8260B
Bromodichloromethane	EPA 8260B	EPA 8260B
Bromoform	EPA 8260B	EPA 8260B
Bromomethane	EPA 8260B	EPA 8260B
2-Butanone (Methyl Ethyl Ketone)	EPA 8015B/8015C/8260B	EPA 8260B
n-Butyl alcohol	EPA 8015B/8015C/8260B	EPA 8260B
n-Butylbenzene	EPA 8260B	EPA 8260B
Sec-Butylbenzene	EPA 8260B	EPA 8260B
Tert-Butylbenzene	EPA 8260B	EPA 8260B
Carbon disulfide	EPA 8260B	EPA 8260B
Carbon tetrachloride	EPA 8260B	EPA 8260B
Chlorobenzene	EPA 8260B	EPA 8260B
Chloroethane	EPA 8260B	EPA 8260B
2-Chloroethyl vinyl ether	EPA 8260B	EPA 8260B
Chloroform	EPA 8260B	EPA 8260B
Chloromethane	EPA 8260B	EPA 8260B
Chloroprene	EPA 8260B	EPA 8260B
2-Chlorotoluene	EPA 8260B	EPA 8260B
4-Chlorotoluene	EPA 8260B	EPA 8260B
Dibromochloromethane	EPA 8260B	EPA 8260B
Dibromomethane	EPA 8260B	EPA 8260B
1,2-Dichlorobenzene	EPA 8260/8270C/8270D	EPA 8260/8270C/8270D
1,3-Dichlorobenzene	EPA 8260/8270C/8270D	EPA 8260/8270C/8270D
1,4-Dichlorobenzene	EPA 8260/8270C/8270D	EPA 8260/8270C/8270D

Parameter/Analyte	Nonpotable Water	Solid Hazardous Waste (Liquids and Solids)
Dichlorodifluoromethane	EPA 8260B	EPA 8260B
1,1-Dichloroethane	EPA 8260B	EPA 8260B
1,2-Dichloroethane	EPA 8260B	EPA 8260B
1,1-Dichloroethene	EPA 8260B	EPA 8260B
cis-1,2-Dichloroethene	EPA 8260B	EPA 8260B
trans-1,2-Dichloroethene	EPA 8260B	EPA 8260B
1,2-Dichloropropane	EPA 8260B	EPA 8260B
1,3-Dichloropropane	EPA 8260B	EPA 8260B
2,2-Dichloropropane	EPA 8260B	EPA 8260B
1,1-Dichloropropene	EPA 8260B	EPA 8260B
cis-1,3-Dichloropropene	EPA 8260B	EPA 8260B
trans-1,3-Dichloropropene	EPA 8260B	EPA 8260B
cis-1,4-Dichloro-2-butene	EPA 8260B	EPA 8260B
trans-1,4-Dichloro-2-butene	EPA 8260B	EPA 8260B
Diethyl ether	EPA 8260B	EPA 8260B
1,4-Dioxane	EPA 8260B	EPA 8260B
Ethyl Acetate	EPA 8015B/8015C/8260B	EPA 8015B/8015C/8260B
Ethyl Benzene	EPA 8260B	EPA 8260B
Ethyl methacrylate	EPA 8260B	EPA 8260B
2-Hexanone	EPA 8260B	EPA 8260B
Hexachlorobutadiene	EPA 8260/8270C/8270D	EPA 8260/8270C/8270D
Isopropylbenzene	EPA 8260B	EPA 8260B
4-Isopropyltoluene	EPA 8260B	EPA 8260B
Iodomethane	EPA 8260B	EPA 8260B
Isobutyl Alcohol	EPA 8015B/8015C/8260B	EPA 8260B
Methacrylonitrile	EPA 8260B	EPA 8260B
Methylene chloride	EPA 8260B	EPA 8260B
Methyl methyacrylate	EPA 8260B	EPA 8260B
4-Methyl-2-pentanone	EPA 8260B	EPA 8260B
Methyl tert butyl ether (MTBE)	EPA 8260B	EPA 8260B
Naphthalene	EPA 8260B/8270C/8270D/8310	EPA 8260B/8270C/8270D/8310
2-Nitropropane	EPA 8260B	EPA 8260B
n-Propylbenzene	EPA 8260B	EPA 8260B
Pentachloroethane	EPA 8260B	EPA 8260B
Propionitrile	EPA 8260B	EPA 8260B
Styrene	EPA 8260B	EPA 8260B
1,1,1,2-Tetrachloroethane	EPA 8260B	EPA 8260B
1,1,2,2-Tetrachloroethane	EPA 8260B	EPA 8260B
Tetrachloroethene	EPA 8260B	EPA 8260B
Toluene	EPA 8260B	EPA 8260B
1,1,1-Trichloroethane	EPA 8260B	EPA 8260B
1,1,2-Trichloroethane	EPA 8260B	EPA 8260B
Trichloroethene	EPA 8260B	EPA 8260B
Trichlorofluoromethane	EPA 8260B	EPA 8260B
1,2,3-Trichlorobenzene	EPA 8260B	EPA 8260B
1,2,3-Trichloropropane	EPA 8260B	EPA 8260B
1,2,4-Trichlorobenzene	EPA 8260B/8270C/8270D	EPA 8260B/8270C/8270D
1,1,2-Trichloro-1,2,2-trifluoroethane	EPA 8260B	----------

Parameter/Analyte	Nonpotable Water	Solid Hazardous Waste (Liquids and Solids)
1,2,4-Trimethylbenzene	EPA 8260B	EPA 8260B
1,3,5-Trimethylbenzene	EPA 8260B	EPA 8260B
Trihalomethanes	EPA 8260B	EPA 8260B
Vinyl acetate	EPA 8260B	EPA 8260B
Vinyl chloride	EPA 8260B	EPA 8260B
Xylenes, total	EPA 8260B	EPA 8260B
o-Xylene	EPA 8260B	EPA 8260B
m+p-Xylene	EPA 8260B	EPA 8260B
Semivolatile Compounds		
Acenaphthene	EPA 8270C/8270D/8310	EPA 8270C/8270D/8310
Acenaphthylene	EPA 8270C/8270D/8310	EPA 8270C/8270D/8310
Acetophenone	EPA 8270C/8270D	EPA 8270C/8270D
2-Acetylaminofluorene	EPA 8270C/8270D	EPA 8270C/8270D
4-Aminobiphenyl	EPA 8270C/8270D	EPA 8270C/8270D
Aniline	EPA 8270C/8270D	EPA 8270C/8270D
Anthracene	EPA 8270C/8270D/8310	EPA 8270C/8270D/8310
Aramite	EPA 8270C/8270D	EPA 8270C/8270D
Atrazine	EPA 8270C/8270D	EPA 8270C/8270D
Benzidine	EPA 8270C/8270D	EPA 8270C/8270D
Benzoic acid	EPA 8270C/8270D	EPA 8270C/8270D
Benzo (a) anthracene	EPA 8270C/8270D/8310	EPA 8270C/8270D/8310
Benzo (b) fluoranthene	EPA 8270C/8270D/8310	EPA 8270C/8270D/8310
Benzo (k) fluoranthene	EPA 8270C/8270D/8310	EPA 8270C/8270D/8310
Benzo (ghi) perylene	EPA 8270C/8270D/8310	EPA 8270C/8270D/8310
Benzo (a) pyrene	EPA 8270C/8270D/8310	EPA 8270C/8270D/8310
p-Benzoquinone	EPA 8270C/8270D	EPA 8270C/8270D
Benzyl alcohol	EPA 8270C/8270D	EPA 8270C/8270D
Bis (2-chloroethoxy) methane	EPA 8270C/8270D	EPA 8270C/8270D
Bis (2-chloroethyl) ether	EPA 8270C/8270D	EPA 8270C/8270D
Bis (2-chloroisopropyl) ether	EPA 8270C/8270D	EPA 8270C/8270D
Bis (2-ethylhexyl) phthalate	EPA 8270C/8270D	EPA 8270C/8270D
4-Bromophenyl phenyl ether	EPA 8270C/8270D	EPA 8270C/8270D
Butyl benzyl phthalate	EPA 8270C/8270D	EPA 8270C/8270D
Carbazole	EPA 8270C/8270D	EPA 8270C/8270D
4-Chloroaniline	EPA 8270C/8270D	EPA 8270C/8270D
Chlorobenzilate	EPA 8270C/8270D	EPA 8270C/8270D
4-Chloro-3-methylphenol	EPA 8270C/8270D	EPA 8270C/8270D
2-Chloronaphthalene	EPA 8270C/8270D	EPA 8270C/8270D
2-Chlorophenol	EPA 8270C/8270D	EPA 8270C/8270D
4-Chlorophenyl phenyl ether	EPA 8270C/8270D	EPA 8270C/8270D
Chrysene	EPA 8270C/8270D/8310	EPA 8270C/8270D/8310
n-Decane	EPA 625	----------
Diallate	EPA 8270C/8270D	EPA 8270C/8270D
Dibenzo (a,h) anthracene	EPA 8270C/8270D/8310	EPA 8270C/8270D/8310
Dibenzofuran	EPA 8270C/8270D	EPA 8270C/8270D
Dibenzo (a,e) pyrene	EPA 8270C/8270D	EPA 8270C/8270D
1,2-Dichlorobenzene	EPA 8260B/8270C/8270D	EPA 8260B/8270C/8270D
1,3-Dichlorobenzene	EPA 8260B/8270C/8270D	EPA 8260B/8270C/8270D
1,4-Dichlorobenzene	EPA 8260B/8270C/8270D	EPA 8260B/8270C/8270D

Parameter/Analyte	Nonpotable Water	Solid Hazardous Waste (Liquids and Solids)
3,3'-Dichlorobenzidine	EPA 8270C/8270D	EPA 8270C/8270D
2,4-Dichlorophenol	EPA 8270C/8270D	EPA 8270C/8270D
2,6-Dichlorophenol	EPA 8270C/8270D	EPA 8270C/8270D
3,3'-Dimethylbenzidine	EPA 8270C/8270D	EPA 8270C/8270D
Diethyl phthalate	EPA 8270C/8270D	EPA 8270C/8270D
Dimethoate	EPA 8270C/8270D	EPA 8270C/8270D
1,3-Dinitrobenzene	EPA 8270C/8270D/8330A/8330B	EPA 8270C/8270D/8330A/8330B
1,4-Dinitrobenzene	EPA 8270C/8270D	EPA 8270C/8270D
Disulfoton	EPA 8270C/8270D	EPA 8270C/8270D
p-Dimethylaminoazobenzene	EPA 8270C/8270D	EPA 8270C/8270D
7,12-Dimethylbenz(a)anthracene	EPA 8270C/8270D	EPA 8270C/8270D
Alpha-,alpha-Dimethylphenethylamine	EPA 8270C/8270D	EPA 8270C/8270D
2,4-Dimethylphenol	EPA 8270C/8270D	EPA 8270C/8270D
Dimethyl phthalate	EPA 8270C/8270D	EPA 8270C/8270D
Di-n-butyl phthalate	EPA 8270C/8270D	EPA 8270C/8270D
Di-n-octyl phthalate	EPA 8270C/8270D	EPA 8270C/8270D
2,4-Dinitrophenol	EPA 8270C/8270D	EPA 8270C/8270D
2,4-Dinitrotoluene	EPA 8270/8330A/8330B	EPA 8270/8330A/8330B
2,6-Dinitrotoluene	EPA 8270/8330A/8330B	EPA 8270/8330A/8330B
Diphenylamine	EPA 8270C/8270D	EPA 8270C/8270D
1,2-Diphenylhydrazine	EPA 8270C/8270D	EPA 8270C/8270D
Ethyl methanesulfonate	EPA 8270C/8270D	EPA 8270C/8270D
Famphur	EPA 8270C/8270D	EPA 8270C/8270D
Fluoroanthene	EPA 8270C/8270D/8310	EPA 8270C/8270D/8310
Fluorene	EPA 8270C/8270D/8310	EPA 8270C/8270D/8310
Hexachlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D
Hexachlorobutadiene	EPA 8260B/8270C/8270D	EPA 8260B/8270C/8270D
Hexachlorophene	EPA 8270C/8270D	EPA 8270C/8270D
Hexachloropropene	EPA 8270C/8270D	EPA 8270C/8270D
Hexachlorocyclopentadiene	EPA 8270C/8270D	EPA 8270C/8270D
Hexachloroethane	EPA 8270C/8270D	EPA 8270C/8270D
Indeno (1,2,3-cd) pyrene	EPA 8270C/8270D/8310	EPA 8270C/8270D/8310
Isodrin	EPA 8270C/8270D	EPA 8270C/8270D
Isophorone	EPA 8270C/8270D	EPA 8270C/8270D
Isosafrole	EPA 8270C/8270D	EPA 8270C/8270D
Kepone	EPA 8270C/8270D	EPA 8270C/8270D
Methapyrilene	EPA 8270C/8270D	EPA 8270C/8270D
3-Methylcholanthrene	EPA 8270C/8270D	EPA 8270C/8270D
2-Methyl-4,6-Dinitrophenol	EPA 8270C/8270D	EPA 8270C/8270D
Methyl methanesulfonate	EPA 8270C/8270D	EPA 8270C/8270D
1-Methylnaphthalene	EPA 8270C/8270D	EPA 8270C/8270D
2-Methylnaphthalene	EPA 8270C/8270D	EPA 8270C/8270D
Methyl Parathion	EPA 8270C/8270D	EPA 8270C/8270D
2-Methylphenol (o-cresol)	EPA 8270C/8270D	EPA 8270C/8270D
3/4-Methylphenols(m/p cresols)	EPA 8270C/8270D	EPA 8270C/8270D
Naphthalene	EPA 8260B/8270C/8270D/8310	EPA8260B/8270C/8270D/8310
1,4-Naphthoquinone	EPA 8270C/8270D	----------

Parameter/Analyte	Nonpotable Water	Solid Hazardous Waste (Liquids and Solids)
1-Naphthylamine	EPA 8270C/8270D	EPA 8270C/8270D
2-Naphthylamine	EPA 8270C/8270D	EPA 8270C/8270D
2-Nitroaniline	EPA 8270C/8270D	EPA 8270C/8270D
3-Nitroaniline	EPA 8270C/8270D	EPA 8270C/8270D
4-Nitroaniline	EPA 8270C/8270D	EPA 8270C/8270D
Nitrobenzene	EPA 8270C/8270D/8330A/8330B	EPA 8270C/8270D/8330A/8330B
5-Nitro-o-toluidine	EPA 8270C/8270D	EPA 8270C/8270D
2-Nitrophenol	EPA 8270C/8270D	EPA 8270C/8270D
4-Nitrophenol	EPA 8270C/8270D	EPA 8270C/8270D
Nitroquinoline-1-oxide	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodietheylamine	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodimethylamine	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodi-n-butylamine	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodi-n-propylamine	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodiphenylamine	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodimethylethylamine	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosomorpholine	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosopiperidine	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosopyrrolidine	EPA 8270C/8270D	EPA 8270C/8270D
n-Octadecane	----------	EPA 8270C/8270D
o,o,o-Triethyl phosphorothioate	EPA 8270C/8270D	EPA 8270C/8270D
o-Toluidine	EPA 8270C/8270D	EPA 8270C/8270D
Parathion, ethyl	EPA 8270C/8270D	EPA 8270C/8270D
Pentachlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D
Pentachloronitrobenzene	EPA 8270C/8270D	EPA 8270C/8270D
Pentachlorophenol	EPA 8270C/8270D/8151A	EPA 8270C/8270D/8151A
Phenacetin	EPA 8270C/8270D	EPA 8270C/8270D
Phenanthrene	EPA 8270C/8270D/8310	EPA 8270C/8270D/8310
Phenol	EPA 8270C/8270D	EPA 8270C/8270D
1,4-Phenylenediamine	EPA 8270C/8270D	----------
Phorate	EPA 8270C/8270D	EPA 8270C/8270D
2-Picoline (2-Methylpyridine)	EPA 8270C/8270D	EPA 8270C/8270D
Pronamide (Kerb)	EPA 8270C/8270D	EPA 8270C/8270D
Pyrene	EPA 8270C/8270D/8310	EPA 8270C/8270D/8310
Pyridine	EPA 8270C/8270D	EPA 8270C/8270D
Safrole	EPA 8270C/8270D	EPA 8270C/8270D
Sulfotepp	EPA 8270C/8270D	EPA 8270C/8270D
1,2,4,5-Tetrachlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D
2,3,4,6-Tetrachlorophenol	EPA 8270C/8270D	EPA 8270C/8270D
Thionazin (Zinophos)	EPA 8270C/8270D	EPA 8270C/8270D
1,2,4-Trichlorobenzene	EPA 8260B/8270C/8270D	EPA 8260B/8270C/8270D
2,4,5-Trichlorophenol	EPA 8270C/8270D	EPA 8270C/8270D
2,4,6-Trichlorophenol	EPA 8270C/8270D	EPA 8270C/8270D
1,3,5-Trinitrobenzene	EPA 8270C/8270D/8330A/8330B	EPA 8270C/8270D/8330A/8330B
Pesticides & PCBs		
Aldrin	EPA 8081A/8081B	EPA 8081A/8081B
alpha-BHC	EPA 8081A/8081B	EPA 8081A/8081B
alpha-Chlordane	EPA 8081A/8081B	EPA 8081A/8081B

Parameter/Analyte	Nonpotable Water	Solid Hazardous Waste (Liquids and Solids)
beta-BHC	EPA 8081A/8081B	EPA 8081A/8081B
Chlordane (technical)	EPA 8081A/8081B	EPA 8081A/8081B
delta-BHC	EPA 8081A/8081B	EPA 8081A/8081B
gamma-BHC	EPA 8081A/8081B	EPA 8081A/8081B
gamma-Chlordane	EPA 8081A/8081B	EPA 8081A/8081B
4,4'-DDD	EPA 8081A/8081B	EPA 8081A/8081B
4,4'-DDE	EPA 8081A/8081B	EPA 8081A/8081B
4,4',-DDT	EPA 8081A/8081B	EPA 8081A/8081B
Dieldrin	EPA 8081A/8081B	EPA 8081A/8081B
Endosulfan I	EPA 8081A/8081B	EPA 8081A/8081B
Endosulfan II	EPA 8081A/8081B	EPA 8081A/8081B
Endonsulfan sulfate	EPA 8081A/8081B	EPA 8081A/8081B
Endrin	EPA 8081A/8081B	EPA 8081A/8081B
Endrin aldehyde	EPA 8081A/8081B	EPA 8081A/8081B
Endrin ketone	EPA 8081A/8081B	EPA 8081A/8081B
Heptachlor	EPA 8081A/8081B	EPA 8081A/8081B
Heptachlor epoxide	EPA 8081A/8081B	EPA 8081A/8081B
Methoxychlor	EPA 8081A/8081B	EPA 8081A/8081B
Toxaphene	EPA 8081A/8081B	EPA 8081A/8081B
PCB-1016 (Aroclor)	EPA 8082/8082A	EPA 8082/8082A
PCB-1221	EPA 8082/8082A	EPA 8082/8082A
PCB-1232	EPA 8082/8082A	EPA 8082/8082A
PCB-1242	EPA 8082/8082A	EPA 8082/8082A
PCB-1248	EPA 8082/8082A	EPA 8082/8082A
PCB-1254	EPA 8082/8082A	EPA 8082/8082A
PCB-1260	EPA 8082/8082A	EPA 8082/8082A
PCB-1262	EPA 8082/8082A	EPA 8082/8082A
PCB-1268	EPA 8082/8082A	EPA 8082/8082A
Total Aroclors	EPA 8082/8082A	EPA 8082/8082A
FID Compounds		
Ethyl acetate	EPA 8015B/8015C/8260B	EPA 8015B/8015C/8260B
Ethylene Glycol	EPA 8015B/8015C	EPA 8015B/8015C
Isobutyl Alcohol	EPA 8015B/8015C/8260B	EPA 8260B
Isopropyl Alcohol (2-Propanol)	EPA 8015B/8015C	----------
Methanol	EPA 8015B/8015C	EPA 8015B/8015C
Diesel Range Organics (DRO)	EPA 8015B/8015C/CA-LUFT/ CT-ETPH	EPA 8015B/8015C/CA-LUFT/ CT-ETPH
Gas Range Organics (GRO)	EPA 8015B/8015C/CA-LUFT	EPA 8015B/8015C/CA-LUFT
Herbicides		
2,4-D	EPA 8151A	EPA 8151A
2,4-DB	EPA 8151A	EPA 8151A
Dalapon	EPA 8151A	EPA 8151A
Dicamba	EPA 8151A	EPA 8151A
Dichloroprop	EPA 8151A	EPA 8151A
Dinoseb	EPA 8151A	EPA 8151A
MCPA	EPA 8151A	EPA 8151A
MCPP	EPA 8151A	EPA 8151A

Parameter/Analyte	Nonpotable Water	Solid Hazardous Waste (Liquids and Solids)
2,4,5-T	EPA 8151A	EPA 8151A
2,4,5-TP (Silvex)	EPA 8151A	EPA 8151A
Pentachlorophenol	EPA 8151A	EPA 8151A
Nitrosamines, Nitroaromatics		
1,3-Dinitrobenzene	EPA 8270C/8270D/8330A/8330B	EPA 8270C/8270D/8330A/8330B
2,4-Dinitrotoluene	EPA 8270C/8270D/8330A/8330B	EPA 8270C/8270D/8330A/8330B
2,6-Dinitrotoluene	EPA 8270C/8270D/8330A/8330B	EPA 8270C/8270D/8330A/8330B
2,4,6-Trinitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B
2-Amino-4,6-Dinitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B
2-Nitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B
3-Nitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B
3,5-Dinitroaniline		EPA 8330B (Solids Only)
4-Amino-2,6-Dinitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B
4-Nitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B
Nitrobenzene	EPA 8270C/8270D/8330A/8330B	EPA 8270C/8270D/8330A/8330B
Nitroglycerine	EPA 8330A/8330B	EPA 8330A/8330B
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	EPA 8330A/8330B	EPA 8330A/8330B
Pentaerythritoltetranitrate (PETN)	EPA 8330A/8330B	EPA 8330A/8330B
hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	EPA 8330A/8330B	EPA 8330A/8330B
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	EPA 8330A/8330B	EPA 8330A/8330B
Radiochemistry		
Barium 133	DOE 4.5.2.3	DOE 4.5.2.3
Cesium 134	DOE 4.5.2.3/EPA 901.1	DOE 4.5.2.3
Cesium 137	DOE 4.5.2.3/EPA 901.1	DOE 4.5.2.3
Cobalt-60	DOE 4.5.2.3/EPA 901.1	DOE 4.5.2.3
Gamma Emitters	DOE 4.5.2.3/EPA 901.1	DOE 4.5.2.3
Gross Alpha	EPA 900.0/9310	EPA 9310
Gross Beta	EPA 900.0/9310	EPA 9310
Radioactive Iodine	DOE 4.5.2.3/EPA 901.1/902.0	DOE 4.5.2.3
Radium-226	EPA 903.1/DOE Ra-04	DOE Ra-04
Radium-228	EPA 904.0/9320/DOE 4.5.2.3	DOE 4.5.2.3/EPA9320
Total Radium	EPA 9315	EPA 9315
Radon	SM7500 Rn-B	----------
Strontium-89	EPA 905.0	DOE Sr-01
Strontium-90	EPA 905.0/DOE Sr-02	DOE Sr-02
Thorium	DOE 4.5.5	DOE 4.5.5
Tritium	EPA 906.0	----------
Uranium	ASTM D5174-02/D5174-97/DOE U-02/EPA 6020/6020A	DOE U-02/EPA 6020/6020A
Zinc-65	EPA 901.1/DOE 4.5.2.3	DOE 4.5.2.3
Preparatory and Clean-up Methods		

Parameter/Analyte	Nonpotable Water	Solid Hazardous Waste (Liquids and Solids)
Toxicity Characteristic Leaching Procedure (Inorganics, Extractable Organics, Volatile Organics)	----------	EPA 1311
Synthetic Preparation Leaching Procedure	----------	EPA 1312
Waste Extraction Test (W.E.T.)	----------	CCR Chapter 11, Article 5, Appendix II
Anion Preparation	----------	EPA 9056A[3]
Cyanide Distillation	EPA 9010B/9010C	EPA 9010B/9010C[3]
Sulfide Distillation	EPA 9030B	EPA 9030B
Metals Digestion	EPA 200.2, 3005A, 3010A	EPA 3050B
Alkaline Digestion for Hexavalent Chromium	----------	EPA 3060A
Bomb Preparation for Solid Waste	----------	EPA 5050
Mercury Preparation	EPA 7470/7470A	EPA 7471A/7471B
Separatory Funnel Liquid-Liquid Extraction	EPA 3510C	----------
Continuous Liquid-Liquid Extraction	EPA 3520C	----------
Solid Phase Extraction	EPA 3535A	----------
Automated Soxhlet Extraction	----------	EPA 3541
Ultrasonic Extraction	----------	EPA 3550C
Waste Dilution	----------	EPA 3580A
Waste Dilution for Volatile Organics	----------	EPA 3585
Purge and Trap for Volatile Organics	EPA 5030A/5030B/5030C	EPA 5035/5035A
Alumina Clean-up	----------	EPA 3610B/3611B
Florisil Clean-up	----------	EPA 3620B/3620C
Silica Gel Clean-up	----------	EPA 3630C
Gel Permeation Clean-up	----------	EPA 3640A
Sulfur Clean-up	----------	EPA 3660B
Sulfuric Acid/Permanganate Clean-up	----------	EPA 3665A

Additionally, in recognition of the successful completion of the A2LA evaluation process (including an assessment of the laboratory's compliance with the 2003 NELAC Chapter 5 Requirements), accreditation is granted to this laboratory to perform the following bioassay analyses on bone, tissue, urine, fecal, and nasal swabs.

	Preparation SOP	Analytical SOP
Bioassy Analysis		

	Preparation SOP	Analytical SOP
Bioassy Analysis		
Alpha Spectrometry: Alpha: Am-241, Cm-242, Cm-243/244, Cm 245/246,Cf-252, Np-237, Po-208, Po- 209, Po-210, Pu-236, Pu-238, Pu-239/240, Pu-242, Pu-244, Th-228, Th-229, Th-230, Th-232, U-232, U-233/234, U-235/236, U-238	GL-RAD-B-001, GL-RAD-B-002, GL-RAD-B-003, GL-RAD-B-010, GL-RAD-B-012, GL-RAD-B-013, GL-RAD-B-017	GL-RAD-B-009
Liquid Scintillation Spectrometry: C-14, Gross Alpha, H-3, Ni-63, Pu-241, Tc-99	GL-RAD-B-001, GL-RAD-B-008, GL-RAD-B-011, GL-RAD-B-012, GL-RAD-B-013, GL-RAD-B-016, GL-RAD-B-020, GL-RAD-B-023	GL-RAD-I-004, GL-RAD-I-014, GL-RAD-I-017
Gas Flow Proportional Counting: Beta: Sr-90	GL-RAD-B-001	GL-RAD-I-006, GL-RAD-I-015, GL-RAD-I-016
Gross Alpha/Gross Beta:	GL-RAD-B-022	GL-RAD-I-006
Kinetic Phosphorescence Analyzer Total Uranium	GL-RAD-B-019	GL-RAD-B-018
Radon Emanation: Ra-226	GL-RAD-B-002	GL-RAD-I-007
Refractometer Specific Gravity	GL-RAD-B-027	GL-RAD-B-027
ICP-MS Uranium Isotopes	GL-RAD-B-035	GL-RAD-B-027
Gamma Spectrometry: Gamma: Ni-59, 46 to 1836 keV	GL-RAD-B-020, GL-RAD-A-013	GL-RAD-I-001

Finally, accreditation is also granted to this laboratory to perform the following tests on children's toys:

Chemical

Lead in Paint by ICP	16 CFR part 1303 (using GL-MA-E-009 and GL-MA-E-013)

1 - Calculated from silica determination

2 – Applicable only to liquid 'Solid Hazardous Waste', where liquids may include aqueous, non-aqueous, and oily wastes. Solids may include soils, sediments, sludges, tissues, filters and any matrix deemed non-liquid.

3 – The referenced method is modified to include a simple prep for non-aqueous and/or solid matrix samples.

The American Association for Laboratory Accreditation

World Class Accreditation

Accredited DoD ELAP Laboratory

A2LA has accredited

GEL LABORATORIES, LLC

Charleston, SC

for technical competence in the field of

Environmental Testing

In recognition of the successful completion of the A2LA evaluation process that includes an assessment of the laboratory's compliance with ISO/IEC 17025:2005, the 2003 NELAC Chapter 5 Standard, and the requirements of the Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (QSM v4.1); accreditation is granted to this laboratory to perform recognized EPA methods as defined on the associated A2LA Environmental Scope of Accreditation. This accreditation demonstrates technical competence for this defined scope and the operation of a laboratory quality management system (*refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009*).

AMERICAN ASSOCIATION FOR LABORATORY ACCREDITATION
CORPORATE SEAL 1978
DISTRICT OF COLUMBIA
A2LA

Presented this 23rd day of October 2009.

President & CEO
For the Accreditation Council
Certificate Number 2567.01
Valid to August 31, 2011
REVISED June 22, 2011

For the tests or types of tests to which this accreditation applies, please refer to the laboratory's Environmental Scope of Accreditation.

APPENDIX E

Analytical SOPs and Certifications

E-1 Katahdin and Subcontractors

ASTM Soil Calculations

Date	REV #	TAB	Revision	Changes Made by:
2/15/10	Draft	ALL	First draft of spreadsheet; includes Drying, Results, Fractions and Reports tabs	K. Najuch

Sample ID	Date/Time	Weigh Boat	Initial Weigh Boat + Sample Weight	Date/Time	1st Weigh Boat + Sample Weight	Date/Time	2nd Weigh Boat + Sample Weight	Date/Time	3rd Weigh Boat + Sample Weight
1	2/11/10 11:00	6.98	42.85	2/11/10 1:40	41.9	2/11/10 4:00	41.98		
2	2/11/10 11:00	6.95	59.04	2/11/10 1:40	54.47	2/11/10 4:00	53.48	2/12/2010 6:00	54.67
3	2/11/10 11:00	6.95	52.24	2/11/10 1:40	50.76	2/11/10 4:00	50.74		
4	2/11/10 11:00	6.95	40.06	2/11/10 1:40	38.64	2/11/10 4:00	38.67		
5	2/11/10 11:00	6.91	38.42	2/11/10 1:40	37.39	2/11/10 4:00	37.39		
6	2/11/10 11:00	6.91	70.94	2/11/10 1:40	69.37	2/11/10 4:00	69.32		
7	2/11/10 11:00	6.96	39.55	2/11/10 1:40	37.51	2/11/10 4:00	37.51		
8	2/11/10 11:00	6.96	53.17	2/11/10 1:40	51.46	2/11/10 4:00	51.46		
9	2/11/10 11:00	6.95	51.48	2/11/10 1:40	50.59	2/11/10 4:00	50.59		
Blank	2/11/10 11:00	6.93	59.36	2/11/10 1:40	56.47	2/11/10 4:00	56.47		

Analysis Information				Raw Weights							
Sample ID	Asbestos Type	Analyst	Date	Weigh Boat plus Sample	Weigh Boat	Empty receiving Pan	Empty 106 micron sieve	Empty 2 mm Sieve	2mm sieve plus sample	106 micron sieve plus sample	Receiving Pan plus sample
1	Chrysotile	JCN	2/12/2010	41.98	6.98	382.32	504.36	493.29	503.06	525.34	387.02
2	Chrysotile	JCN	2/12/2010	54.67	6.95	479.80	504.88	443.44	463.54	523.85	490.12
3	Chrysotile	JCN	2/12/2010	50.74	6.95	382.26	504.44	495.37	506.76	521.74	397.85
4	Chrysotile	JCN	2/12/2010	38.67	6.95	479.76	505.03	445.13	447.50	523.78	490.71
5	Chrysotile	JCN	2/12/2010	37.39	6.91	382.68	504.53	494.31	495.35	518.00	399.60
5	Amosite	JCN	2/12/2010	37.39	6.91	382.68	504.53	494.31	495.35	518.00	399.60
5	Total	JCN	2/12/2010	37.39	6.91	382.68	504.53	494.31	495.35	518.00	399.60
6	Chrysotile	JCN	2/12/2010	69.32	6.91	479.92	505.02	444.05	462.98	529.61	499.77
7	Chrysotile	JCN	2/12/2010	37.51	6.96	383.72	504.52	493.91	498.18	523.68	390.93
8	Chrysotile	JCN	2/12/2010	51.46	6.96	480.69	504.94	443.93	468.96	521.58	484.15
8	Amosite	JCN	2/12/2010	51.46	6.96	480.69	504.94	443.93	468.96	521.58	484.15
8	Total	JCN	2/12/2010	51.46	6.96	480.69	504.94	443.93	468.96	521.58	484.15
9	Chrysotile	JCN	2/12/2010	50.59	6.95	382.90	504.61	494.70	505.39	525.75	394.88
9	Amosite	JCN	2/12/2010	50.59	6.95	382.90	504.61	494.70	505.39	525.75	394.88
9	Total	JCN	2/12/2010	50.59	6.95	382.90	504.61	494.70	505.39	525.75	394.88
Blank	None Detected	JCN	2/12/2010	56.47	6.93	480.31	505.12	445.23	446.47	552.84	480.78

Calculated Weights						Analytical Sensitivity (% Asbestos)				PLM Results (% Asbestos)							
Initial sample weight	Weight Course fraction	Weight Medium fraction	Weight Fine Fraction	Total weight of sieved fractions	% Loss in sieving	Coarse Fraction	Medium Fraction	Fine Fraction	Total	Coarse Fraction		Medium Fraction		Fine Fraction (Pt Ct)		Final Calculated Asbestos Percent	
35	9.77	20.98	4.7	35.45	-1.29	1.00	1.00	0.25	0.90	<	1.00	<	1.00		NAD	<	0.90
47.72	20.10	18.97	10.32	49.39	-3.50	1.00	1.00	0.25	0.84	<	1.00	<	1.00		NAD	<	0.84
43.79	11.39	17.3	15.59	44.28	-1.12	1.00	1.00	0.25	0.74		NAD	<	1.00		NAD	<	0.74
31.72	2.37	18.75	10.95	32.07	-1.10	1.00	1.00	0.25	0.74		NAD	<	1.00		NAD	<	0.74
30.48	1.04	13.47	16.92	31.43	-3.12	1.00	1.00	0.25	0.60		NAD	<	1.00		NAD	<	0.60
30.48	1.04	13.47	16.92	31.43	-3.12	1.00	1.00	0.25	0.60		NAD	<	1.00		NAD	<	0.60
30.48	1.04	13.47	16.92	31.43	-3.12	1.00	1.00	0.25	0.60		NAD	<	1.00		NAD	<	0.60
62.41	18.93	24.59	19.85	63.37	-1.54	1.00	1.00	0.25	0.77	<	1.00	<	1.00		NAD	<	0.77
30.55	4.27	19.16	7.21	30.64	-0.29	1.00	1.00	0.25	0.82		NAD	<	1.00		NAD	<	0.82
44.5	25.03	16.64	3.46	45.13	-1.42	1.00	1.00	0.25	0.94	<	1.00	<	1.00		NAD	<	0.94
44.5	25.03	16.64	3.46	45.13	-1.42	1.00	1.00	0.25	0.94	<	1.00	<	1.00		NAD	<	0.94
44.5	25.03	16.64	3.46	45.13	-1.42	1.00	1.00	0.25	0.94	<	1.00	<	1.00		NAD	<	0.94
43.64	10.69	21.14	11.98	43.81	-0.39	1.00	1.00	0.25	0.79	<	1.00	<	1.00		NAD	<	0.79
43.64	10.69	21.14	11.98	43.81	-0.39	1.00	1.00	0.25	0.79	<	1.00	<	1.00		NAD	<	0.79
43.64	10.69	21.14	11.98	43.81	-0.39	1.00	1.00	0.25	0.79	<	1.00	<	1.00		NAD	<	0.79
49.54	1.24	47.72	0.47	49.43	0.22	1.00	1.00	0.25	0.99		NAD		NAD		NAD		NAD

TEM Results	Filtration (Structures/µg)			
Drop Mount Detect/Non Detect	# of Structures	Analytical Sensitivity	Concentration	
		#DIV/0!	<	#DIV/0!
		#DIV/0!	<	#DIV/0!
		#DIV/0!	<	#DIV/0!
		#DIV/0!	<	#DIV/0!
		#DIV/0!	<	#DIV/0!
		#DIV/0!	<	#DIV/0!
		#DIV/0!	<	#DIV/0!
		#DIV/0!	<	#DIV/0!
		#DIV/0!	<	#DIV/0!
		#DIV/0!	<	#DIV/0!
		#DIV/0!	<	#DIV/0!
		#DIV/0!	<	#DIV/0!
		#DIV/0!	<	#DIV/0!
		#DIV/0!	<	#DIV/0!
		#DIV/0!	<	#DIV/0!
		#DIV/0!	<	#DIV/0!

				# of Positive
	Analysis Information			**Suspect ACM**
Sample	**Asbesots**			**ACM Materials**
ID	**Type**	**Analyst**	**Date**	**Catagorized**
1	Chrysotile	JCN	2/12/2010	0
2	Chrysotile	JCN	2/12/2010	0
3	Chrysotile	JCN	2/12/2010	0
4	Chrysotile	JCN	2/12/2010	0
5	Chrysotile	JCN	2/12/2010	0
5	Amosite	JCN	2/12/2010	0
5	Total	JCN	2/12/2010	
6	Chrysotile	JCN	2/12/2010	0
7	Chrysotile	JCN	2/12/2010	0
8	Chrysotile	JCN	2/12/2010	0
8	Amosite	JCN	2/12/2010	0
8	Total	JCN	2/12/2010	
9	Chrysotile	JCN	2/12/2010	0
9	Amosite	JCN	2/12/2010	0
9	Total	JCN	2/12/2010	
Blank	None Detected	JCN	2/12/2010	0

		% Unconsolidated Asbestos in		Weight (g) of Unconsolidated
	Total Weight	Remaining Soil		Asbestos in
	(g) Asbestos in	via Stereoscope		Remaining Soil
	Suspect ACM	(optional)		(optional)
	0.0000	<0.1	<	0.0098
	0.0000	<0.1	<	0.0201
	0.0000	ND	<	0.0000
	0.0000	ND	<	0.0000
	0.0000	ND	<	0.0000
	0.0000	ND	<	0.0000
<	0.0000		<	0.0000
	0.0000	<0.1	<	0.0189
	0.0000	ND	<	0.0000
	0.0000	<0.1	<	0.0250
	0.0000	<0.1	<	0.0250
<	0.0000		<	0.0501
	0.0000	0.5		0.0535
	0.0000	0.3		0.0321
<	0.0000			0.0855
	0.0000	ND	<	0.0000

Coarse Fraction - Suspect Material Info

Material:	#1	Material:	#2	Material:	#3
Weight (g)	% asbestos	Weight (g)	% asbestos	Weight (g)	% asbestos

Material:	#4	Material:	#5	Material:	#6	Material:
Weight (g)	% asbestos	Weight (g)	% asbestos	Weight (g)	% asbestos	Weight (g)

#7	Material:	#8	Material:	#9
% asbestos	Weight (g)	% asbestos	Weight (g)	% asbestos

Weighted Asbestos Totals by Number of Suspect ACM Materials

in grams

Material:	#10						
		Including the number of Suspect ACM Material Typ					
Weight (g)	% asbestos	1	2	3	4	5	6
		0	0	0	0	0	0
		0	0	0	0	0	0
		0	0	0	0	0	0
		0	0	0	0	0	0
		0	0	0	0	0	0
		0	0	0	0	0	0
		0	0	0	0	0	0
		0	0	0	0	0	0
		0	0	0	0	0	0
		0	0	0	0	0	0
		0	0	0	0	0	0
		0	0	0	0	0	0
		0	0	0	0	0	0
		0	0	0	0	0	0
		0	0	0	0	0	0
		0	0	0	0	0	0
		0	0	0	0	0	0

				Weights of suspect ACM Catagorized in grams						
es in Question				Material Type						
7	8	9	10	1	2	3	4	5	6	7
0	0	0	0	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0	0	0

				Is a Total Analysis Line
8	9	10		
0	0	0		0
0	0	0		0
0	0	0		0
0	0	0		0
0	0	0		0
0	0	0		0
0	0	0		1
0	0	0		0
0	0	0		0
0	0	0		0
0	0	0		0
0	0	0		1
0	0	0		0
0	0	0		0
0	0	0		1
0	0	0		0
0	0	0		0

Tabulation Calcs									
Asbestos (g) per	Asbestos (g) Material	Asbestos (g)	Asbestps (g) St.					Less than Ckecker	
Material	Sample Total	Stereoscope	Sample Total	1	2	3	4	5	6
0	0	0.00977	0.00977	1	1	1	1	1	1
0	0	0.0201	0.0201	1	1	1	1	1	1
0	0	0	0	1	1	1	1	1	1
0	0	0	0	1	1	1	1	1	1
0	0	0	0	1	1	1	1	1	1
0	0	0	0	1	1	1	1	1	1
0	0	0	0	1	1	1	1	1	1
0	0	0.01893	0.01893	1	1	1	1	1	1
0	0	0	0	1	1	1	1	1	1
0	0	0.02503	0.02503	1	1	1	1	1	1
0	0	0.02503	0.05006	1	1	1	1	1	1
0	0	0	0.05006	1	1	1	1	1	1
0	0	0.05345	0.05345	1	1	1	1	1	1
0	0	0.03207	0.08552	1	1	1	1	1	1
0	0	0	0.08552	1	1	1	1	1	1
0	0	0	0	1	1	1	1	1	1
0	0	#VALUE!	#VALUE!	1	1	1	1	1	1

7	8	9	10	UCA		current	1	2
1	1	1	1	0			1	1
1	1	1	1	0			1	1
1	1	1	1	0			1	1
1	1	1	1	0			1	1
1	1	1	1	0			1	1
1	1	1	1	0			1	1
1	1	1	1	1		To	1	1
1	1	1	1	0			1	1
1	1	1	1	0			1	1
1	1	1	1	0			1	1
1	1	1	1	0			1	1
1	1	1	1	1		To	1	1
1	1	1	1	1			1	1
1	1	1	1	1			1	1
1	1	1	1	1		To	1	1
1	1	1	1	0			1	1
1	1	1	1	1			1	1

Less than Tubalation Calculators										
material veriety previous					Stereo estimate - previous					
3	4	5	6	current	1	2	3	4	5	6
1	1	1			1	1	1	1	1	
1	1	1	1		1	1	1	1	1	1
1	1	1	1		1	1	1	1	1	1
1	1	1	1		1	1	1	1	1	1
1	1	1	1		1	1	1	1	1	1
1	1	1	1		1	1	1	1	1	1
1	1	1	1	To	1	1	1	1	1	1
1	1	1	1		1	1	1	1	1	1
1	1	1	1		1	1	1	1	1	1
1	1	1	1		1	1	1	1	1	1
1	1	1	1		1	1	1	1	1	1
1	1	1	1	To	1	1	1	1	1	1
1	1	1	1		1	1	1	1	1	1
1	1	1	1		0	1	1	1	1	1
1	1	1	1	To	0	0	1	1	1	1
1	1	1	1		1	1	1	1	1	1
1	1	1	1		1	1	1	1	1	1

Sample ID	Analysis Information Asbesots Type	Analyst	Date	# of Positive Suspect ACM Materials Catagorized	
1	Chrysotile	JCN	2/12/2010	0	
2	Chrysotile	JCN	2/12/2010	0	
3	Chrysotile	JCN	2/12/2010	0	
4	Chrysotile	JCN	2/12/2010	0	
5	Chrysotile	JCN	2/12/2010	0	
5	Amosite	JCN	2/12/2010	0	
5	Total	JCN	2/12/2010		<
6	Chrysotile	JCN	2/12/2010	0	
7	Chrysotile	JCN	2/12/2010	0	
8	Chrysotile	JCN	2/12/2010	0	
8	Amosite	JCN	2/12/2010	0	
8	Total	JCN	2/12/2010		<
9	Chrysotile	JCN	2/12/2010	0	
9	Amosite	JCN	2/12/2010	0	
9	Total	JCN	2/12/2010		<
Blank	None Detected	JCN	2/12/2010	0	

Total Weight (g) Asbestos in Suspect ACM	% Unconsolidated Asbestos in Remaining Soil via Stereoscope (optional)		Weight (g) of Unconsolidated Asbestos in Remaining Soil (optional)	Material: Weight (g)
0.0000	<0.1	<	0.0210	
0.0000	<0.1	<	0.0190	
0.0000	<0.1	<	0.0173	
0.0000	<0.1	<	0.0188	
0.0000	<0.1	<	0.0135	
0.0000	<0.1	<	0.0135	
0.0000		<	0.0269	
0.0000	<0.1	<	0.0246	
0.0000	<0.1	<	0.0192	
0.0000	<0.1	<	0.0166	
0.0000	<0.1	<	0.0166	
0.0000		<	0.0333	
0.0000	0.1		0.0211	
0.0000	<0.1	<	0.0211	
0.0000			0.0423	
0.0000	None Detected	<	0.0000	

Medium Fraction - Suspect Material Info

#1	Material: #2		Material: #3	
% asbestos	Weight (g)	% asbestos	Weight (g)	% asbestos

Material: #4		Material: #5		Material:
Weight (g)	% asbestos	Weight (g)	% asbestos	Weight (g)

#6	Material: #7		Material: #8	
% asbestos	Weight (g)	% asbestos	Weight (g)	% asbestos

Material: #9		Material: #10		
Weight (g)	% asbestos	Weight (g)	% asbestos	1
				0
				0
				0
				0
				0
				0
				0
				0
				0
				0
				0
				0
				0
				0
				0
				0
				0
				0

Weighted Asbestos Totals by Number of Suspect ACM Materials
in grams

Including the number of Suspect ACM Material Types in Question

2	3	4	5	6	7	8
0	0	0	0	0	0	0
0	0	0	0	0	0	0
0	0	0	0	0	0	0
0	0	0	0	0	0	0
0	0	0	0	0	0	0
0	0	0	0	0	0	0
0	0	0	0	0	0	0
0	0	0	0	0	0	0
0	0	0	0	0	0	0
0	0	0	0	0	0	0
0	0	0	0	0	0	0
0	0	0	0	0	0	0
0	0	0	0	0	0	0
0	0	0	0	0	0	0
0	0	0	0	0	0	0
0	0	0	0	0	0	0
0	0	0	0	0	0	0
0	0	0	0	0	0	0

		Weights of suspect in gr Materia
9	10	1	2	3	4	5
0	0	0	0	0	0	0
0	0	0	0	0	0	0
0	0	0	0	0	0	0
0	0	0	0	0	0	0
0	0	0	0	0	0	0
0	0	0	0	0	0	0
0	0	0	0	0	0	0
0	0	0	0	0	0	0
0	0	0	0	0	0	0
0	0	0	0	0	0	0
0	0	0	0	0	0	0
0	0	0	0	0	0	0
0	0	0	0	0	0	0
0	0	0	0	0	0	0
0	0	0	0	0	0	0
0	0	0	0	0	0	0
0	0	0	0	0	0	0

t ACM Catagorized
ams

al Type					Is a Total Analysis Line
6	7	8	9	10	
0	0	0	0	0	0
0	0	0	0	0	0
0	0	0	0	0	0
0	0	0	0	0	0
0	0	0	0	0	0
0	0	0	0	0	0
0	0	0	0	0	1
0	0	0	0	0	0
0	0	0	0	0	0
0	0	0	0	0	0
0	0	0	0	0	0
0	0	0	0	0	1
0	0	0	0	0	0
0	0	0	0	0	0
0	0	0	0	0	1
0	0	0	0	0	0
0	0	0	0	0	0

	Tabulation Calcs			
Asbestos (g) per Material	Asbestos (g) Material Sample Total	Asbestos (g) Stereoscope	Asbestps (g) St. Sample Total	1
0	0	0.02098	0.02098	1
0	0	0.01897	0.01897	1
0	0	0.0173	0.0173	1
0	0	0.01875	0.01875	1
0	0	0.01347	0.01347	1
0	0	0.01347	0.02694	1
0	0	0	0.02694	1
0	0	0.02459	0.02459	1
0	0	0.01916	0.01916	1
0	0	0.01664	0.01664	1
0	0	0.01664	0.03328	1
0	0	0	0.03328	1
0	0	0.02114	0.02114	1
0	0	0.02114	0.04228	1
0	0	0	0.04228	1
0	0	0	0	1
0	0	#VALUE!	#VALUE!	1

Less than Ckecker

2	3	4	5	6	7	8	9	10
1	1	1	1	1	1	1	1	1
1	1	1	1	1	1	1	1	1
1	1	1	1	1	1	1	1	1
1	1	1	1	1	1	1	1	1
1	1	1	1	1	1	1	1	1
1	1	1	1	1	1	1	1	1
1	1	1	1	1	1	1	1	1
1	1	1	1	1	1	1	1	1
1	1	1	1	1	1	1	1	1
1	1	1	1	1	1	1	1	1
1	1	1	1	1	1	1	1	1
1	1	1	1	1	1	1	1	1
1	1	1	1	1	1	1	1	1
1	1	1	1	1	1	1	1	1
1	1	1	1	1	1	1	1	1
1	1	1	1	1	1	1	1	1
1	1	1	1	1	1	1	1	1

				material veriety previous		
UCA	current	1	2	3	4	5
0		1	1	1	1	1
0		1	1	1	1	1
0		1	1	1	1	1
0		1	1	1	1	1
0		1	1	1	1	1
0		1	1	1	1	1
1	To	1	1	1	1	1
0		1	1	1	1	1
0		1	1	1	1	1
0		1	1	1	1	1
0		1	1	1	1	1
1	To	1	1	1	1	1
1		1	1	1	1	1
0		1	1	1	1	1
1	To	1	1	1	1	1
0		1	1	1	1	1
1		1	1	1	1	1

Less than Tubalation Calculators						
				Stereo estimate - previous		
6	current	1	2	3	4	5
		1	1	1	1	1
1		1	1	1	1	1
1		1	1	1	1	1
1		1	1	1	1	1
1		1	1	1	1	1
1		1	1	1	1	1
1	To	1	1	1	1	1
1		1	1	1	1	1
1		1	1	1	1	1
1		1	1	1	1	1
1		1	1	1	1	1
1	To	1	1	1	1	1
1		1	1	1	1	1
1		0	1	1	1	1
1	To	1	0	1	1	1
1		1	1	1	1	1
1		1	1	1	1	1

6

1
1
1
1
1
1
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1
1
1
1
1

Sample ID	Analysis Information Asbesots Type	Analyst	Date	Point Count EPA 400 Results % Asbestos
1	Chrysotile	JCN	2/12/2010	ND
2	Chrysotile	JCN	2/12/2010	ND
3	Chrysotile	JCN	2/12/2010	ND
4	Chrysotile	JCN	2/12/2010	ND
5	Chrysotile	JCN	2/12/2010	ND
5	Amosite	JCN	2/12/2010	ND
5	Total	JCN	2/12/2010	
6	Chrysotile	JCN	2/12/2010	ND
7	Chrysotile	JCN	2/12/2010	ND
8	Chrysotile	JCN	2/12/2010	ND
8	Amosite	JCN	2/12/2010	ND
8	Total	JCN	2/12/2010	
9	Chrysotile	JCN	2/12/2010	ND
9	Amosite	JCN	2/12/2010	ND
9	Total	JCN	2/12/2010	
Blank	None Detected	JCN	2/12/2010	ND

Total Weight (g) Asbestos in Suspect ACM	Tabulation Calcs		
	Is a Total Analysis Line	PT CT	Pt Ct sum
NAD	0	0	0
NAD	0	0	0
NAD	0	0	0
NAD	0	0	0
NAD	0	0	0
NAD	0	0	0
NAD	1	0	0
NAD	0	0	0
NAD	0	0	0
NAD	0	0	0
NAD	0	0	0
NAD	1	0	0
NAD	0	0	0
NAD	0	0	0
NAD	1	0	0
NAD	0	0	0
	0	0	0

				Less than Tubalation Calculators		
				previous		
Pt Ct tot	current	1	2	3	4	5
0	1	0	0	0	0	0
0	1	0	0	0	0	0
0	1	0	0	0	0	0
0	1	0	0	0	0	0
0	1	0	0	0	0	0
0	1	1	0	0	0	0
0.25	To	1	1	0	0	0
0	1	0	0	0	0	0
0	1	0	0	0	0	0
0	1	0	0	0	0	0
0	1	1	0	0	0	0
0.25	To	1	1	0	0	0
0	1	0	0	0	0	0
0	1	1	0	0	0	0
0.25	To	1	1	0	0	0
0	1	0	0	0	0	0
0	1	0	0	0	0	0

Number of Components Y=1 N=0

6	Tot	1	2	3	4	5	6	7	Tot
	0	0	0	0	0	0			0
0	0	0	0	0	0	0	0		0
0	0	0	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0	0
0	1	1	0	0	0	0	0	0	1
0	2	1	1	0	0	0	0	0	2
0	0	0	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0	0
0	1	1	0	0	0	0	0	0	1
0	2	1	1	0	0	0	0	0	2
0	0	0	0	0	0	0	0	0	0
0	1	1	0	0	0	0	0	0	1
0	2	1	1	0	0	0	0	0	2
0	0	0	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0	0

ND Tubalation Calculators

			previous				
current	1	2	3	4	5	6	Tot
1	0	0	0	0	0		0
1	0	0	0	0	0	0	0
1	0	0	0	0	0	0	0
1	0	0	0	0	0	0	0
1	0	0	0	0	0	0	0
1	1	0	0	0	0	0	1
To	1	1	0	0	0	0	2
1	0	0	0	0	0	0	0
1	0	0	0	0	0	0	0
1	0	0	0	0	0	0	0
1	1	0	0	0	0	0	1
To	1	1	0	0	0	0	2
1	0	0	0	0	0	0	0
1	1	0	0	0	0	0	1
To	1	1	0	0	0	0	2
1	0	0	0	0	0	0	0
1	0	0	0	0	0	0	0

Sample ID	Analysis Information Asbesots Type	Analyst	Date	Number of Total Structures Counted
1	Chrysotile	JCN	2/12/2010	
2	Chrysotile	JCN	2/12/2010	
3	Chrysotile	JCN	2/12/2010	
4	Chrysotile	JCN	2/12/2010	
5	Chrysotile	JCN	2/12/2010	
5	Amosite	JCN	2/12/2010	
5	Total	JCN	2/12/2010	
6	Chrysotile	JCN	2/12/2010	
7	Chrysotile	JCN	2/12/2010	
8	Chrysotile	JCN	2/12/2010	
8	Amosite	JCN	2/12/2010	
8	Total	JCN	2/12/2010	
9	Chrysotile	JCN	2/12/2010	
9	Amosite	JCN	2/12/2010	
9	Total	JCN	2/12/2010	
Blank	None Detected	JCN	2/12/2010	

Analytical Sensitivity Structures per µg	Concentration Structures per µg	Weight of Sample in Suspension (mg)	Total Volume of Suspension (ml)
#DIV/0!	< #DIV/0!		100.00
#DIV/0!	< #DIV/0!		100.00
#DIV/0!	< #DIV/0!		100.00
#DIV/0!	< #DIV/0!		100.00
#DIV/0!	< #DIV/0!		100.00
#DIV/0!	< #DIV/0!		
#DIV/0!	< #DIV/0!		
#DIV/0!	< #DIV/0!		
#DIV/0!	< #DIV/0!		
#DIV/0!	< #DIV/0!		
#DIV/0!	< #DIV/0!		
#DIV/0!	< #DIV/0!		
#DIV/0!	< #DIV/0!		
#DIV/0!	< #DIV/0!		
#DIV/0!	< #DIV/0!		
#DIV/0!	< #DIV/0!		

Volume Filtered (ml)	G.O.'s	GOA (mm^2)	EFA (mm^2)
10			
10			
10			
10			
1			

Tabulation Calcs

Structrues (tot)	Is a Total Analysis Line	Structures	Total sample Structures
0	0	0	0
0	0	0	0
0	0	0	0
0	0	0	0
0	0	0	0
0	0	0	0
0	1	0	0
0	0	0	0
0	0	0	0
0	0	0	0
0	0	0	0
0	1	0	0
0	0	0	0
0	0	0	0
0	1	0	0
0	0	0	0
0	0	0	0

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270

Prepared By: Casarin/Millett/Galasyn Date: 2/97

Approved By:

Group Supervisor: [signature] Date: 011201

Operations Manager: [signature] Date: 1/15/01

QA Officer: Deborah J. Nadeau Date: 1·23·01

General Manager: [signature] Date: 1/16/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
03 8270C	Format changes, added pollution prevention, changes to calibration section, new limits, added instrument. Other minor changes throughout.	DN	1·23·01	1·23·01
04 8270C	Updated sections 7.5.1.1 and 7.6.1 to be compliant with South Carolina requirements.	DN	5·22·01	5·22·01
05 8270C	Added statistical limits for LCS + MS/MSD recoveries. Modified corrective actions for these limits. Added 5973 instrument.	DN	5·21·02	5·21·02
06 8270C	major reorg. of sect.'s 4,5,6,7 and Tables added definitions and information for new processing system. added wording to sect. 8 minor changes throughout	LAD	02/05	02/05
07 8270C	Sect. 5.3.2.1 changed 200 ug/ml to 1500 ug/ml Sect. 7.2 changed Instrument "K" to Instr. "R" Sect. 7.5.1 updated table ~~Sect.~~ Table 5: minor edits	LAD	04/06	04/06

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
08 8270C	Added ICV Criteria to Definitions, 7.5.22 and Table 1 Added RT window clarification to 7.5.2.3 Added Allowable Exceedances Criteria to 8.4 and Table 1 Removed Grande Mean Option from 7.5.2.1 Updated Std. Prep in 5.3.2.1 and 7.5.1	LAD	~~05/07~~ 07/07	05/07 07/07
09 8270C	Reworded Correlation coefficient criteria Added new instrument (G). Updated Section 10. Fixed grammatical errors.	LAD	02/08	02/08
10 8270C	Minor change to Section 5.3.2.1 - regarding the prep of the ICV.	LAD	04/09	04/09
11 8270C	Updated throughout to address new calibration levels and DoD QSM Ver. 4.1 compliance	LAD	08/09	08/09
12 8270C	Added references. Added Table 2 - "DoD QSM QC Requirements" (F-Table)	LAD	04/10	04/10

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270.

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document SOP CA-204-12, titled "Analysis of Semivolatile Organic Compounds by Capillary Column GC/MS: SW 846 Method 8270".

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP CA-204-12, titled "Analysis of Semivolatile Organic Compounds by Capillary Column GC/MS: SW 846 Method 8270".

Recipient: _____ Date: _____

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270.

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services, Inc. laboratory personnel to prepare and analyze water and soil sample extracts for semivolatile organics by EPA SW-846 Method 8270, current revision.

In order to maintain consistency in data quality, this SOP consolidates all aspects of the analyses in one working document, to be revised as necessary.

1.1 Definitions:

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, laboratory reagent grade water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount; a calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution, which is different from the stock used to prepare standards.

INDEPENDANT CALIBRATION STANDARD: A solution prepared from a stock standard solution independent of the standard that is used to calibrate the instrument. Analyzed immediately after calibration,

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution, which is used to calibrate the instrument response with respect to analyte concentration.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative

percent difference between the samples is calculated and used to assess analytical precision.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

STOCK STANDARD SOLUTION: A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

QUICKFORMS: A laboratory reporting software for Target and Target DB. The QuickForms report module for Target is preconfigured with generalized forms and US EPA CLP report forms and disk deliverables, which can be customized.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of semivolatile organic compounds by EPA Method 8270. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, "Personnel Training & Documentation of Capability," current revision.

It is the responsibility of all Katahdin technical personnel involved in analysis of semivolatiles by Method 8270 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with the Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory.

Each analyst should know the location of a respirator and all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

After analysis, autosampler vials containing sample extracts in methylene chloride are returned to the SVOA hood, and the contents transferred to a labeled waste container. The contents of this container are disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD

The process involves the extraction of semivolatiles from a sample using an appropriate solvent followed by clean up steps (where applicable) and concentration of the extract (refer to Katahdin SOP CA-502, "Preparation Of Aqueous Samples For Extractable Semivolatile Analysis", SOP CA-512, "Preparation Of Sediment/Soil Samples By Sonication Using Method 3550 For Subsequent Extractable Semi-Volatiles Analysis" and SOP CA-526, "Preparation Of Sediment/Soil Samples By Soxhlet Extraction Using Method 3540 For

Subsequent Extractable Semivolatile Analysis"). An aliquot of the final extract is injected into the gas chromatograph for compound separation by capillary column, followed by the electron impact mass spectrometer for identification and quantitation.

Target and surrogate compounds are identified and compared to the mass spectra obtained from the analysis of standard solutions containing the same compounds. A relative response factor is established for each target compound and surrogate against an internal standard during the most recent initial or continuing calibrations. The identified compound is then quantitated using the relative response factor, the amount of internal standard in the sample, the initial volume of sample, and any other factors, such as dilutions.

3.0 INTERFERENCES

Interfering contamination may occur when a sample containing low concentrations of SVOCs is analyzed immediately after a sample containing high concentrations of SVOCs. Any samples that have suspected carryover must be reanalyzed.

4.0 APPARATUS AND MATERIALS

4.1 GC: Hewlett Packard 5890 and/or 6890

4.2 Mass Spectrometers (MS): HP5973, HP5972 and/or HP5970

4.3 Helium: Carrier gas for routine applications. All carrier gas lines must be constructed from stainless steel or copper tubing; non-polytetrafluoroethylene (non-PTFE) thread sealant or flow controllers with rubber component are not to be used.

4.4 Autosamplers: HP 7673As and HP 7683s

4.5 Hamilton syringes: 2.00 uL to 10 mL

4.6 Volumetric glassware: Grade A or equivalent

4.7 Columns: DB-5MS 30m, 0.25mm I.D., 25um film thickness, columns (J&W Scientific) or equivalent.

4.8 Acquisition System: The acquisition system must be interfaced to the MS and allow continuous acquisition of data throughout the duration of the chromatographic program. It must permit, at a minimum, the output of time vs. intensity (peak height or peak area). Hewlett Packard Chemstation or equivalent.

4.9 Data System: The Target software is used for processing data and generating forms.

4.10 1.8 mL vials with 350uL inserts

4.11 Crimp tops with Teflon lined septa

5.0 REAGENTS

5.1 J.T. Baker Ultra Resi-Analyzed methylene chloride (or equivalent)

5.2 Purge and trap grade methanol

5.3 Standards: Stock standards and working standards are received and recorded in accordance with SOP CA-106 "Standard Preparation and Documentation".

5.3.1 The expiration date for all standards is one year from date of opening the ampule. If the manufacturer's expiration date is before this one year date, the manufacturer's expiration must be followed. New standards must be opened if degradation is observed.

5.3.2 Secondary dilution standards

The standards are prepared on an as needed basis (but not greater than every 6 months) and stored in screw cap amber bottles with Teflon liners in the BNA standards freezer between uses. Standards prepared from various stock solutions must always use the first expiration date of any of the solutions used for preparation.

5.3.2.1 Calibration Mix – Prepare a standard stock mix that contains those compounds commonly considered 8270 and those compounds commonly considered Appendix IX compounds. The compound dinoseb should <u>not</u> be added to this stock as it is only available in methanol. This will be added separately to each calibration level. Use Table 4 as a guide. The stock should be prepared at 125 ug/mL.

5.3.2.2 Independent Calibration Verification (ICV) Standard – From a source other than that used to make the calibration standards, prepare separate standards mixes (A and B) such that Standard Mix A contains those compounds commonly considered 8270 and Standard B Mix contains those compounds commonly considered Appendix IX compounds. Use Table 4 as a guide. Each stock should be prepared at 100 ug/mL.

5.3.2.3 DFTPP Solution – Prepare standard in methylene chloride containing DFTPP, Pentachlorophenol, Benzidine and DDT at a final concentration of 25 ug/mL.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

All semivolatile sample extracts should be refrigerated until analysis. Extracts must be analyzed within forty days following the date of extraction.

7.0 PROCEDURES

7.1 NAMING AND CODING CONVENTIONS FOR ANALYTICAL STANDARDS – Used in accordance with SOP CA-106 "Standard Preparation and Documentation".

7.2 COMPUTER (DATA SYSTEM) CONVENTIONS -

Conventions for all instruments are as follows:

Sub-Directory for data acquisition and storage: C:\HPCHEM\1\DATA

Tune file: DFTPP.U

Method files: L8270CXX.M (all samples and standards)

Where:
XX = the calibration number in chronological order
L = instrument ID (R, U, or G)

DFTPP tuning acquisition: DFTPP390.M

NOTE: All acquisition parameters must be identical for L8270CXX.M and DFTPP390. M.

Data Files: L_ _ _ _.D, where _ _ _ _ is a number in chronological order from 0001 to 9999 and L is the instrument ID (R, U, or G). This file also contains the Quantitation output file.

Data Files for DFTPP: LD_ _ _.D, where _ _ _ is a number in chronological order from 001 to 999 and L is the instrument ID (R, U, or G).

7.3 INSTRUMENT SPECIFIC PROCEDURES

It is the policy of the GC/MS group that all data be acquired in the batch mode. The following items must be checked prior to data acquisition in the batch mode:

- Ensure that the proper sequence and tune files are being used.
- Check the autosampler syringe (Is it clean? Does the plunger move freely? etc.), its alignment and make sure the solvent rinse vial is full. Ensure that the knurled nut holding the top of the syringe plunger is tight.

- Look at the batch to be analyzed and check the following:
  - Make sure that the data files are in numerical order with no duplication and that the method file is the same as that used for ICAL or Continuing Calibration analysis.
  - Bottle numbers match with the numbers on the autosampler tray.

After the batch has been deemed free of errors, start the batch by using the "Position and run" command under the SEQUENCE menu in MSTop.

7.4 INSTRUMENT TUNING - Prior to the analysis of any calibration standards, blanks or samples, the GC/MS system must be shown to meet the mass spectral key ion and ion abundance criteria for decafluorotriphenylphosphine (DFTPP) tabulated below. Pentachlorophenol, benzidine and DDT are also present in this standard.

Mass	Criteria
51	30.0 to 60.0 percent of mass 198
68	less than 2.0 percent of mass 69
69	present
70	less than 2.0 percent of mass 69
127	40.0-60.0 percent of mass 198
197	less than 1.0 percent of mass 198
198	base peak, 100 percent relative abundance
199	5.0-9.0 percent of mass 198
275	10.0-30.0 percent of mass 198
365	greater than 1.00 percent of mass 198
441	present, but less than mass 443
442	greater than 40.0 percent of mass 198
443	17.0-23.0 percent of mass 442

All ion abundances must be normalized to m/z 198, the nominal base peak.

The following are the GC/MS operating conditions for injection of DFTPP.

Initial column temperature hold	140°C for 3 minutes
Column temperature program	140-275°C at 15 degrees/minute
Final column temperature hold	275°C
Injection port temperature	280°C
Transfer line/source temperature	285°C
Injector - splitless, valve time	0.18 minutes
EPC	inlet B
Constant flow	ON
Constant flow pressure	10psi
Constant flow temperature	30°C

Vacuum comp.	ON
Run time	10-12 minutes
Scan start time	5.0 minutes
Sample volume	2.0 uL of 25 ng/uL DFTPP solution
Carrier gas	helium at approximately 60 mL/minute
Mass range	35 to 500 amu
Number of A/D samples	4
GC Peak threshold	500 counts
Threshold	10 counts

Set up the run on the Enviroquant system using "Edit Sample Log Table". For a more detailed explanation of the Enviroquant software, consult the appropriate manual, Department Manager, or senior chemist within the GC/MS group.

When the DFTPP has concluded, the run must be evaluated to determine if sample analysis can proceed. The chromatography and the ion ratios must be examined. The DFTPP run is processed using the current algorithms in the Target software.

If the results indicate the system does not meet acceptance criteria, the GC/MS must be manually tuned. Once the manual tune procedure is completed, DFTPP must be re-injected and reevaluated. If the instrument still does not meet criteria, notify your Department Manager. Under no circumstances should calibration proceed if the instrument DFTPP is not in criteria.

The DFTPP tuning standard should also be used to assess the column performance and injection port inertness. Calculate the degradation of DDT to DDE and DDD; it should not exceed 20%. Benzidine and pentachlorophenol should be present at their normal responses, with no evidence of peak tailing. For clients requiring DOD criteria, the tailing factors for these two compounds should not exceed 2.

In order to document the performance of benzidine, pentachlorophenol and DDT, the following procedure must be followed. At the PC, which operates the instrument, load the method TUNETAIL.M into the ENVDA screen. Go into the quant drop down menu and select *calculate/generate report*. When that finishes, select *Qedit quant result.* Each compound can now be evaluated. Double click on benzidine and select *ChromEval* and then *Evaluate tailing*. Follow the instructions given on the screen to evaluate tailing. Send the report to the printer. Repeat the procedure for pentachlorophenol. Repeat the procedure for DDT, selecting *Evaluate degradation*. Follow the instructions given on the screen and then send the report to the printer. The report should be filed with the tune raw data.

The DFTPP solution must be analyzed once at the beginning of each twelve hour period during which standards and/or samples are analyzed. The 12 hour time period for GC/MS system begins at the moment of injection of the DFTPP analysis. The time period ends after twelve hours has elapsed according to the system clock. The last injection must be accomplished prior to the expiration of 12 hours;

conceivably, the run-time of an injection could end after the twelve hours.

7.5 INSTRUMENT CALIBRATION

7.5.1 Initial Calibration for Method 8270

Prior to the analysis of samples and required method blanks, and after the instrument DFTPP tuning criteria have been met, the GC/MS system must be calibrated. The calibration consists of a six point curve. The calibration levels are 10, 25, 50, 75, 100 and 125 ng/uL Calibration is done to determine instrument sensitivity and the linearity of GC/MS response for the semivolatile target and surrogate compounds.

Final conc. (ng/uL)	SVOA Stock Soln Added (uL)	1000 ug/mL dinoseb Standard (uL)	$MeCl_2$ Added (uL)	Final Vol (uL)	IS Added (uL)
10	16	2	182	200	2
25	40	5	155	200	2
50	80	10	110	200	2
75	120	15	65	200	2
100	160	20	20	200	2
125	100	0	0	100	1

If additional compound mixtures are added, the volume of $MeCl_2$ is adjusted to maintain a final volume of 200 or 100 uL. A 100 uL aliquot of each of the standards above is spiked as above with 4000 ng/uL Internal Standard stock and analyzed.

Internal Standards
1,4-Dichlorobenzene-d4
Naphthalene-d8
Acenaphthene-d10
Phenanthrene-d10
Chrysene-d12
Perylene-d12

The GC/MS operating conditions for the calibration standards injections are the same as for the DFTPP with the following exceptions:

Column Temperature Program	40°C for 3 minutes to 300°C at 10°/minute
Final Column Temperature hold	300°C
Run Time	34-36 minutes
Scan Start Time	1.8 minutes (time may vary dependent upon column length)
Injection volume	1 uL

The conditions are set up in the method files L8270CXX.M.

After analysis of the six calibration points, they must be processed and evaluated for adherence to QC criteria. Minimum requirements of ID files are the use of specific quantitation ions and quantitating a specific set of targets and surrogates with a set internal standard. These requirements are found in Tables 3 and 5.

7.5.2 Initial Calibration Criteria

Relative response factors (RRFs) must be calculated and evaluated for each target compound and surrogate. The RRF is defined as follows:

$$RRF = \frac{Ax}{A_{IS}} \times \frac{C_{IS}}{Cx}$$

where: Ax = area of the primary ion for the target compound
A_{IS} = area of the primary ion for the corresponding istd
C_{IS} = concentration of the istd (ng/uL)
C_x = concentration of the target compound

After the calibration points have been quantitated, update the calibration curve points using the Target data processing software to generate the RRF's and %RSD's for all analytes. If information is needed concerning the use of these programs, consult the Department Manager or a senior chemist within the group.

Response factor criteria have been established for the calibration of the semivolatile target and surrogate compounds. These criteria must be met in order for the calibration curve to be considered valid. The percent RSD for each calibration check compound (CCC) must be less than or equal to 30 percent. There are thirteen calibration check compounds: Acenaphthene, 1,4-Dichlorobenzene, Hexachlorobutadiene, N-Nitrosodiphenylamine, Di-n-octylphthalate, Fluoranthene, Benzo(a)pyrene, 4-Chloro-3-Methylphenol, 2,4-Dichlorophenol, 2-Nitrophenol, Phenol, Pentachlorophenol and 2,4,6-Trichlorophenol. This is also applicable to clients that request DOD criteria.

There are also four system performance check compounds (SPCCs). They have no maximum percent RSD but they must meet a minimum RRF criterion of 0.050. The four SPCCs are N-Nitroso-di-n-propylamine, Hexachlorocyclopentadiene, 2,4-Dinitrophenol and 4-Nitrophenol. The SPCCs are used to check the stability of both the standard and the instrument. This is also applicable to clients that request DOD criteria.

Achieving CCC and SPCC criteria is not a substitute for performing a calibration for all target analytes.

7.5.2.1 Linearity of Target Analytes (This is also applicable to clients that request DOD criteria.)

If the RSD of any target analyte is 15% or less, then the response factor is presumed to be constant over the calibration range, and the average response factor may be used for quantitation.

If the RSD of any target analyte exceeds 15%, then a calibration option outlined in section 7.0 of method 8000 will need to be employed. Please note that some options may not be allowable for certain states, federal programs, or clients. South Carolina does not allow option 1 or option 3 for compliance work originating in their state.

Option 1 (Section 7.5.2 of method 8000 - Rev. 2, 12/96), is a linear regression of instrument response versus the standard concentration. The correlation coefficient (r) for each target analyte and surrogate must be greater than or equal to 0.995. For linear models, Target reports r^2. This is calculated by either calculating r and squaring the result or by calculating the coefficient of determination. For a linear calibration, the equation for either is the same. The value for r^2 must greater than or equal to 0.990.

Option 2 (Section 7.5.3 of method 8000 - Rev. 2, 12/96), is a non-linear calibration model not to exceed a third order (seven calibration points required) polynomial. The lab would use a quadratic model or second order polynomial. The use of a quadratic model requires six calibration points. In order for the quadratic model to be acceptable, the coefficient of determination must be greater than or equal to 0.99.

Internal standard (IS) responses and retention times in all standards must be evaluated immediately after data acquisition; if the RT for any IS changes by more than 0.50 minutes from the latest daily calibration standard, corrections must be made to the chromatographic system. If the extracted ion current profile (EICP) area for any IS changes by more than a factor of two (-50% to +100%), corrective action must be performed.

Each GC/MS system must be calibrated following system corrective action, including ion source cleaning or repair and column removal or replacement.
If time remains in the clock after meeting the initial calibration acceptance criteria, samples may be analyzed. The calibration must be verified each twelve hour time period (time period starts from the moment of the DFTPP injection) for Method 8270. The SSTD050 in

the curve may be used as the calibration verification standard as long as it meets the calibration verification acceptance criteria. All sample results must be quantitated using the initial calibration response factors.

7.5.2.2 Immediately following calibration an Independent Calibration Verification Standard must be analyzed. For clients requiring DOD criteria, all project analytes must be within +/- 20% of true value.

7.5.2.3 Retention Time Windows

Retention time windows are set at the midpoint standard of the calibration curve, following every ICAL. When a CV is analyzed (and not an ICAL), the retention time windows of the daily CV must be within 30 seconds of the midpoint calibration standard of the most recent ICAL. The samples analyzed following the daily CV must have retention times within 30 seconds of those for the daily CV. Each successive daily CV must be compared to the most recent ICAL midpoint standard.

7.5.3 Continuing Calibration

A calibration verification check standard must be performed once every twelve hours immediately following analysis of the tuning compound DFTPP. This check contains all target compounds and surrogates at a concentration of 50 ng/uL.
After quantitation of the 50 ng/uL continuing calibration check, response factors must be calculated and compared to the average response factors in the initial calibration. The Target program calculates the calibration check response factors and compares them to the average RFs in the calibration curve by calculating percent differences. The method 8270 CCC's must have a % difference of +/- 20%D in order to be considered in criteria. The method 8270 SPCC's must meet a minimum RRF criterion of 0.050 in order to be considered in criteria. These conditions must be met before method blank and/or sample analysis can begin. For clients requiring DOD criteria, all project analytes and surrogates must be within +/- 20%.

The area for the internal standards in the calibration verification must be within a factor of two (-50% to 100%) from the mid-point standard of the most recent initial calibration. This is listed in the ISTD monitor report.
If the calibration verification does not meet criteria, corrective action must be taken. Depending on the situation, corrective action may be as follows:

- Re-analyze the 50 ng/uL continuing calibration check.
- Change the septum; clean the injection port; install a clean, silanized

quartz liner; cut off a small portion (1" to 3") of the front end of the capillary column (this is usually performed when acid RFs are low and/or chromatography is poor).

- Analyze a new initial calibration curve.

The last option, the generation of a new initial calibration curve, is usually chosen when percent difference are >30%. In these instances, there is little or no chance of a continuing calibration reanalysis meeting criteria. If there is any doubt concerning which corrective action to undertake, consult the Department Manager or a senior chemist within the group.

If the calibration verification does meet the criteria specified above then analysis may proceed using initial calibration response factors.

7.6 SAMPLE ANALYSIS

Sample extracts may be analyzed only after the GC/MS system has met tuning criteria, initial calibration and continuing calibration requirements. Ensure that the same instrument conditions are being used for tuning, calibration and sample analysis
by reviewing the GC parameters using the "Edit entire method" option under the Method menu in MSTOP. Note that you can not edit a method if the instrument is running.

Extracts are stored in the refrigerator in the organics extraction laboratory at 4°C ±2°C. Remove them from the refrigerator and place them in the GC/MS laboratory semivolatile hood when ready for analysis.

Prepare a 1.8 mL clear glass vial (crimp top) with a disposable insert (350 uL). Add 100 uL of sample extract and 1.0 uL of the 4000 ng/uL IS stock to the vial and then cap. This gives a 40 ng/uL final concentration for the internal standard compounds. The samples are topped with Teflon lined crimp top caps.

7.7 FINAL DATA PACKAGE

7.7.1 Initial Data Review (IDR)

The initial data review is accomplished by the analyst who analyzed the samples and is a review of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed sample. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed:

- Surrogate Recoveries

- Internal Standard Area Stability
- Method Blank Acceptance
- Chromatography
- Target Compound Detection/Quantitation/Review for false positives
- Laboratory Control Sample Recoveries
- Matrix Spike/Matrix Spike Duplicate Recoveries

The analyst must evaluate all data using the QA Acceptance Criteria table found within this SOP (Table 1). This table gives acceptance criteria and corrective actions for criteria that are not met. In addition to evaluating QC elements, the chromatography and quantitation of target analytes must be reviewed. During this review, the analyst checks the integration of each individual peak. The hardcopy has false positives crossed out so they can be reviewed for appropriateness by the Department Manager.

7.7.2 Chromatography

The chromatography should be examined for the presence or absence of any ghost peaks and can also be used as an indication of whether or not matrix interferences might be affecting surrogate recoveries and/or Istd area recoveries. Whether or not the chromatography is acceptable is a judgment call on the part of the analyst and should be used in conjunction with other monitored QC (e.g. surrogate recoveries) to determine the necessity of reanalyzing.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), an "m" qualifier will automatically be printed on the quantitation report summary. All manual integrations are initialed, dated and given a code which describes the reason for the manual integration.

This manual integration package must then be submitted to the Department Manager or his/her designee, who will review each manual integration.
For specific procedures on how to manually integrate, refer to Katahdin SOP QA-812, "Manual Integration", current revision.

7.7.3 Target Compound Detection/Quantitation
The semivolatile ID files have been set up to err on the side of false positives; that is, to identify and quantitate peaks as target compounds that may not

necessarily be valid hits. It is the responsibility of the GC/MS analyst to use his/her technical judgment to determine if the identification of a target compound is correct or not.

If any target concentration exceeds the upper limit, a dilution must be made and analyzed. The dilution chosen should keep the concentration of the largest target compound hit in the upper half of the initial calibration range. LCS and MS/MSD samples need not be diluted to get spiked analytes within the calibration range.

The requirements for qualitative verification by comparison of mass spectra are as follows:

- All ions present in the standard mass spectra at a relative intensity > 10% must be present in the sample spectrum.
- The relative intensities of primary and secondary ions must agree within ±20% between the standard and sample spectra.
- Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst.

If a compound cannot be verified by all three criteria above, but, in the technical judgment of the mass spectral interpretation specialist, the identification is correct, then the laboratory shall report that compound on the Form 1 as a valid hit.

The GC/MS laboratory initial data review must be completed within twelve hours of batch completion; in the majority of instances, the initial review should be accomplished at the beginning of a work shift for the previous set of analyses.

7.7.3.1 Tentatively Identified Compounds (TIC)

TIC's may be requested by certain clients for samples. Refer current Katahdin to SOP CA-207 "GC/MS Library Search and Quantitation.

7.7.4 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into QuickForms. Depending on the QC label requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any

errors. When completed, the package is sent to the department manager for final review. A complete review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

7.8 Injection Port Liner Cleaning And Silanizing Procedure

7.8.1 Remove the rubber o-ring from the liner and place the liner in a large Erlenmeyer flask.

7.8.2 In the hood, pour nitric acid into the flask until the liner is covered. Place the flask on a hotplate and boil for 2-3 hours.

7.8.3 Let cool; drain nitric acid and thoroughly flush the liner with water.

7.8.4 Bake briefly in the muffle oven until liner is dry and cool to room temperature.

7.8.5 Place the liner in a beaker, fill with Sylon and let it soak for at least two hours.

7.8.6 Take out the liner and rinse it thoroughly with toluene.

7.8.7 Rinse the liner thoroughly with purge and trap grade methanol.
7.8.8 Bake the liner in the muffle oven for a minimum of three hours.

7.9 Instrument Maintenance

Instrument preventative maintenance is performed on a semi-annual basis by GC/MS chemists. This maintenance includes a thorough inspection and cleaning of all parts, including changing rough and turbopump oils. GC/MS analysts perform other maintenance on an as-needed basis. Typically, routine maintenance involves clipping off the front end of the DB-5MS column, replacing the injection port septum, and installing a freshly silanized quartz liner after sample analysis.

All maintenance must be documented in the instrument-specific maintenance log, whether it is routine or not. The Department Manager must authorize any maintenance over and above a routine source cleaning.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may

be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 Method Blank Criteria

A method blank is defined as a volume of a clean reference material (laboratory reagent grade water for water samples, baked organic-free sand for soil/sediment matrices) that is carried through the entire analytical procedure. One method blank must be extracted with each group of samples of a similar matrix and must be analyzed on the GC/MS system that was used to analyze the samples.

An acceptable method blank must contain less than or equal to the PQL of any target compound. For clients requiring DOD criteria, no analytes detected at > ½ PQL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit.

If the method blank exceeds these contamination levels, the analytical system is considered out of control and corrective action must be taken before sample analysis.

Reanalysis of the blank is the first step of the corrective action; if that does not solve the problem, a Katahdin Corrective Action Report (CAR) will be initiated.

Corrective action will be specified after consultation including the Department Manager, Operations Manager, and QA Officer.

8.2 Surrogate Recoveries

There are six surrogates, which can be divided as follows:

- B/N - Nitrobenzene-d5, 2-Fluorobiphenyl and Terphenyl-d14

- Acid - Phenol-d5, 2-Fluorophenol and 2,4,6-Tribromophenol

The surrogates have laboratory derived statistical limits that are updated on an annual basis and are available in the QA office. For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

If specifications are not met, the sample (or blank) should be reanalyzed. If specifications are met in the reanalysis, this reanalysis should only be submitted. If surrogate specifications are not met in the sample or method blank reanalysis, a Corrective Action Report (CAR) should be initiated. Corrective action will be specified after consultation including the Department Manager and Operations Manager.

For further information regarding the acceptance of surrogate recoveries, consult the Department Manager.

8.3 Internal Standard Responses

Internal standard responses and retention times (RT) in all samples and blanks must be evaluated as part of the technical data review. The method files have been set up to only detect compounds that fall within a set RT window. For Method 8270 analysis, if the extracted ion current profile (EICP) area for any internal standard changes by more than a factor of two (-50% to +100%) as compared to the daily continuing calibration standard, reanalysis must occur. If the reanalysis meets criteria, only the in-criteria run should be reported. If the reanalysis is still out-of-criteria, both analyses should be included in the sample package set.

MS/MSD samples that do not meet the EICP area criteria above do not have to be reanalyzed.

8.4 Laboratory Control Sample (LCS)

An LCS must be performed for each group of samples of a similar matrix, for the following, whichever is more frequent:

- Every 20 samples of a similar matrix or similar concentration, or
- Every batch of samples extracted.

Statistical limits are compiled annually for LCS recoveries (archived in QA office). Statistical limits are only calculated when at least 20 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the Department Manager, Laboratory Operations Manager and Quality Assurance Officer. Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts", current revision.

The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 60-140% or 70-130% may be used for some projects or states (i.e. South Carolina). For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

The LCS recoveries for all analytes are evaluated. All of the compounds of interest must fall within the established statistical limits with the following sporadic exceedance allowances.

Number of Analytes	Number of Allowable Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
<11	0

If less than the number of allowable exceedances fail the statistical limits, no corrective action is needed. If greater than the number of allowable exceedances fail the statistical limits, corrective action may be taken. Corrective actions may vary with each situation. However, in the case where the failures are high and the samples are non-detect for those compounds, then no corrective action is required. Otherwise, corrective action may involve reanalysis or recalibration. The specific corrective actions taken will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time.

Please note that for compounds with only nominal limits (i.e. insufficient data points were available to generate statistical limits), no corrective action is required for out-of-criteria recoveries until enough data points are established to generate statistical limits.

8.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Criteria

Matrix Spike and Matrix Spike Duplicates must be extracted and analyzed for each group of up to 20 samples of a similar matrix or similar concentration. In the event insufficient sample volume is available an LCS/LCS Duplicate is extracted and analyzed in place of the MS/MSD.

Statistical limits are compiled annually for MS/MSD recoveries for a short list of the spiked compounds. Nominal limits of 60-140% are used for all other compounds. Generally, corrective action is only taken for the short list of the spiked compounds. The specific corrective actions will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control

indicators and/or the ability to reanalyze a sample within holding time. For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

8.6 QC Requirements

Refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. Table 1 criteria are intended to be guidelines for analysts. The table does not over all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all of the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of Method 8270 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods." SW-846, 3rd edition, Final Updates I, II, IIA, IIB, III, IIIA, and IIIB, Nov 2004, Method 8270C.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270.

Katahdin SOP CA QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

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TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270.

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Check of mass spectral ion intensities using DFTPP	Prior to initial calibration and calibration verification	Refer to the criteria listed in Section 7.4	Retune instrument, and verify
Six-point initial calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF ≥0.05; RSD ≤30 for RFs of the CCCs; Average %RSD < 15% for all compounds. Refer to section 7.5.2.1 also.	Repeat calibration if criterion is not met
Independent calibration verification	Once after Initial calibration	± 20 % D	1) Reanalyze standard 2) Reprep standard 3) Reprep standard from fresh stock.
Continuing calibration verification	Once per each 12 hours, prior to sample analysis	CCCs ≤ 20%D; SPCCs RF ≥0.05	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification
ISs	Immediately after or during data acquisition of calibration check standard	Retention time ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning
Demonstration of ability to generate acceptable accuracy and precision	Once per analyst and annually there after.	All recoveries within method QC acceptance limits.	Recalculate results; locate and fix problem; reextract/reanalyze P&A study for those analytes that did not meet criteria
Method blank	One per prep batch	No analytes detected > PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e.If the blank results are above the PQL, report samples that are <PQL or > 10X the blank result. Reprep a blank and the remaining samples.

TABLE 1, (cont.)

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
LCS for all analytes	One LCS per prep batch	Statistically derived from lab data or nominal limits depending on the project. Refer to QA records for statistical limits. Nominal limits are used as default limits. See also section 8.4 of this SOP for more information on allowable exceedances.	(1) Evaluate the samples and associated QC: i.e.If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS rec. is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.
Surrogate spike	Every sample, control, standard, and method blank	current statistical limits	(1) Check chromatogram for interference; if found, flag data (2) If not found, check instrument performance; if problem is found, correct and reanalyze (3) If still out reextract and analyze sample (4) If reanalysis is out, flag data
MS/MSD	One MS/MSD per every 20 samples	Statistically derived from lab data or nominal limits depending on the project. Refer to QA records for statistical limits and section 8.5 of this SOP.	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable reprep the samples and QC.
MDL study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TABLE 2

DoD QSM QC REQUIREMENTS

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-specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria.	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	Refer to current revision of SOP QA-806				
LOQ establishment and verification	Refer to current revision of SOP QA-806				
Tuning	Prior to ICAL and at the beginning of each 12-hour period.	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Breakdown check (DDT Method 8270 only)	Correct problem then repeat breakdown check.	Degradation ≤ 20% for DDT. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2.	At the beginning of each 12-hour period, prior to analysis of samples.	Flagging criteria are not appropriate.	No samples shall be run until degradation ≤ 20%.
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	1. Average response factor (RF) for SPCCs ≥ 0.050. 2. RSD for RFs for CCCs ≤ 30% and one option below: Option 1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression r ≥ 0.995; Option 3: non-linear regression–coefficient of determination (COD) r2 ≥ 0.99 (6 points shall be used for second order).	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin.
Second source calibration verification (ICV)	Once after each ICAL.	All project analytes within ± 20% of true value.	Correct problem and verify second source standard. Rerun second source verification. 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.

TABLE 2 (cont)

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention time window position establishment for each analyte and surrogate	Once per ICAL.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	
Evaluation of relative retention times (RRT)	With each sample.	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	Laboratories may update the retention times based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping). With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ±0.06 RRT units since the last update, this indicates a significant change in system performance and the laboratory must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the retention times.
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	1. Average RF for SPCCs ≥ 0.050. 2. %Difference/Drift for all target compounds and surrogates ≤ 20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration).	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	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 last acceptable CCV.	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.
Internal standards verification	Every field sample, standard, and QC sample.	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.

TABLE 2 (cont)

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > ½ RL (> RL for common lab contaminants) and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result. Contact Client if samples cannot be reprepped within hold time.	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.
LCS containing all analytes to be reported, including surrogates	One per preparatory batch.	The laboratory shall use laboratory control limits (CLs) or use DoD-generated LCS-CLs, if available depending on project requirements. In-house CLs may not be greater than ± 3 times the standard deviation of the mean LCS recovery. A number of analytes may fall outside the CL but within marginal exceedance limit depending on the total number of analytes in the LCS.	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. Refer to Table G-1 for number of marginal exceedences allowed. Contact Client if samples cannot be reprepped within hold time.	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.
Matrix Spike (MS)	One per preparatory batch per matrix if sufficient sample is available.	For matrix evaluation, use LCS acceptance criteria.	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.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix if sufficient sample is available.	MSD: For matrix evaluation, use LCS acceptance criteria. MS/MSD: RPD ≤ 30%.	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.

TABLE 2 (cont)

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate spike	All field and QC samples.	The laboratory shall use laboratory surrogate CLs or use DoD-generated surrogate CLs, if available depending on project requirements. .	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Contact Client if samples cannot be reprepped within hold time.	Apply Q-flag to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-204-12	Method 8270, current revision
Apparatus/Materials	none	
Reagents	none	
Sample preservation/ handling	none	
Procedures	none	
QC - Spikes	none	
QC - LCS	none	
QC - Accuracy/Precision	none	
QC - MDL	PQL – Practical Quantitation Level – three to ten times the MDL.	EQL – Estimated Quantitation Level – five to ten times the MDL

TABLE 4

Analyte Quantitation and Internal Standards

Internal Standard: 1,4-dichlorobenzene-d4

Target and Surrogates:

Pyridine
N-Nitrosodimethylamine (not on TCL list)
Aniline (not on TCL list)
Phenol
Bis (2-chloroethyl) ether
2-Chlorophenol
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,2-Dichlorobenzene
Benzyl alcohol (not on PP list)
2-Methylphenol (not on PP list)
2,2'-oxybis(1-chloropropane) (also known as Bis (2-Chloroisopropyl) ether)
4-Methylphenol (not on PP list)
N-Nitroso-di-n-propylamine
Hexachloroethane
Ethyl methanesulfonate (8270 C)
Methyl methanesulfonate (8270 C)
2-Picoline (8270 C)
N-Nitrosomethylethylamine (Appendix IX)
N-Nitrosodiethylamine (Appendix IX)
N-Nitrosopyrrolidine (Appendix IX)
N-Nitrosomorpholine (Appendix IX)
2-Fluorophenol (surrogate)
Phenol-d6 (surrogate)

Internal Standard: Naphthalene-d8

Target and Surrogates:

Nitrobenzene
Isophorone
2-Nitrophenol
2,4-Dimethylphenol
Acetophenone (8270 C)
Benzoic acid (not on PP list)
Bis (2-chloroethoxy) methane
2,4-Dichlorophenol
2,6-Dichlorophenol (8270 C)
1,2,4-Trichlorobenzene
a, a-Dimethyl-phenethylamine (8270 C)
Naphthalene
4-Chloroaniline (not on PP list)
Hexachlorobutadiene
4-Chloro-3-methylphenol
2-Methylnaphthalene
N-Nitrosodi-n-butylamine (8270 C)
N-Nitrosopiperidine (8270 C)
o-toluidine (Appendix IX)
o, o, o-Triethylphosphorothioate (Appendix IX)
Hexachloropropene (Appendix IX)
Isosafrole (Appendix IX)
Nitrobenzene-d5 (surrogate)

Internal Standard: Acenaphthene-d10

Target and Surrogates:

Hexachlorocyclopentadiene
2,4,6-Trichlorophenol
2,4,5-Trichlorophenol (not on PP list)
1-Chloronaphthalene (8270 C)
2-Chloronaphthalene
2-Nitroaniline (not on PP list)
Dimethyl phthalate
Acenaphthylene
3-Nitroaniline (not on PP list)
Acenaphthene
2,4-Dinitrophenol
4-Nitrophenol
Dibenzofuran (not on PP list)
2,4-Dinitrotoluene
2,6-Dinitrotoluene
Diethyl phthalate
4-Chlorophenylphenyl ether
Fluorene
4-Nitroaniline (not on PP list)
1-Naphthylamine (8270 C)
2-Naphthylamine (8270 C)
Pentachlorobenzene (8270 C)

TABLE 4 (cont.)

Analyte Quantitation and Internal Standards

1, 2, 4, 5-Tetrachlorobenzene (8270 C)
2, 3, 4, 6-Tetrachlorophenol (8270 C)
p-Phenylenediamene (Appendix IX)
Safrole (Appendix IX)
1,4-Naphthoquinone (Appendix IX)
Thionazine (Appendix IX)
5-Nitro-o-toluidine (Appendix IX)
1,2-Diphenylhydrazine (not on TCL list)
2-Fluorobiphenyl (surrogate)
2,4,6-Tribromophenol (surrogate)

Internal Standard: Phenanthrene-d10

Target and Surrogates:

4,6-Dinitro-2-methylphenol
N-Nitrosodiphenylamine
Diphenylamine (8270 C)
4-Bromophenylphenyl ether
Phenacetin (8270 C)
Hexachlorobenzene
4-Aminobiphenyl (8270 C)
Pentachlorophenol
Pentachloronitrobenzene (8270 C)
Pronamide (8270 C)
Phenanthrene
Anthracene
Di-n-butylphthalate
Carbazole (8270 B)
Fluoranthene
Sym-Trinitrobenzene (Appendix IX)
Diallate (Appendix IX)
4-Nitroquinoline-1-oxide (Appendix IX)
Methapyrilene (Appendix IX)
Isodrin (Appendix IX)
Dinoseb (Appendix IX)

Internal Standard: Chrysene-d12

Target and Surrogates:

Benzidine (not on TCL list)
Pyrene
Butylbenzyl phthalate
3,3'-Dichlorobenzidine
p-Dimethylaminoazobenzene (8270 C)
Benzo (a) Anthracene
Bis (2-ethylhexyl) phthalate
Chrysene
3-Methylcholanthrene (8270 C)
Aramite (Appendix IX)
Chlorobenzilate (Appendix IX)
3,3'-Dimethylbenzidine (Appendix IX)
2-Acetylaminofluorene (Appendix IX)
Terphenyl-d14 (surrogate)

Internal Standard: Perylene-d12

Target and Surrogates:

Di-n-octyl phthalate
Benzo (b) fluoranthene
Benzo (k) fluoranthene
Benzo (a) pyrene
Indeno (1,2,3-cd) pyrene
Dibenz (a, h) anthracene
Dibenz (a, j) acridine (8270 C)
Benzo (ghi) perylene
7,12-Dimethylbenz (a) anthracene (8270 C)
Hexachlorophene (Appendix IX)

TABLE 5

PROCEDURE CONDENSATION

Clock

12 hours from injection of 50ng DFTPP.

Calibration Curve Criteria

<30% RSD for CCCS
minimum RF criteria for SPCCs
<15% RSD average for all analytes in calibration standard

Continuing Calibration Check Criteria

<20% D for CCCS
minimum RF criteria for SPCCs

Additional QC

LCS every extraction batch
MS/MSD every 20 samples

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270.

TABLE 6

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

COMPOUND	PRIMARY ION	SECONDARY ION(S)
2-Picoline	93	66,92
Aniline	93	66,65
N-Nitrosodimethylamine	42	74,43
Phenol	94	65,66
Bis(2-Chloroethyl)ether	93	63,95
2-Chlorophenol	128	64,130
1,3-Dichlorobenzene	146	148,111
1,4-Dichlorobenzene	146	148,111
1,2-Dichlorobenzene	146	148,111
N-Nitrosomethylethylamine	88	42,43,56
Benzyl alcohol	108	77,79
2-Methylphenol	107	108,77,79,90
Bis(2-Chloroisopropyl)ether	45	77,121
4-Methylphenol	107	108,77,79,90
N-Nitroso-di-n-propylamine	70	42,101,130
Hexachloroethane	117	201,199
Nitrobenzene	77	123,65
Isophorone	82	95,138
2-Nitrophenol	139	65,109
2,4-Dimethylphenol	122	121,107
Benzoic acid	122	105,77
Bis(2-chloroethoxy)methane	93	95,123
2,4-Dichlorophenol	162	164,98
1,2,4-Trichlorobenzene	180	182,145
Naphthalene	128	129,127
4-Chloroaniline	127	129,65,92
Hexachlorobutadiene	225	223,227
4-Chloro-3-methylphenol	107	144,142
2-Methylnaphthalene	142	141
Hexachlorocyclopentadiene	237	235,272
2,4,6-Trichlorophenol	196	198,200
2,4,5-Trichlorophenol	196	198,97,132,99
2-Chloronaphthalene	162	164,127
2-Nitroaniline	65	92,138
Dimethyl phthalate	163	194,164
Acenaphthylene	152	151,153
3-Nitroaniline	138	108,92
Acenaphthene	153	152,154
2,4-Dinitrophenol	184	63,154
4-Nitrophenol	109	139,65
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63,89
2,6-Dinitrotoluene	165	89,63
Diethyl phthalate	149	177,150
4-Chlorophenylphenylether	204	206,141
Fluorene	166	165,167
4-Nitroaniline	138	92,108,65,80,39
4,6-Dinitro-2-methylphenol	198	105,51
N-Nitrosodiphenylamine	169	168,167
4-Bromophenylphenylether	248	250,141

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270.

TABLE 6 (cont.)

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

COMPOUND	PRIMARY ION	SECONDARY ION(S)
Hexachlorobenzene	284	142,249
1,2-Diphenylhydrazine	184	77,92
Pentachlorophenol	266	264,268
Phenanthrene	178	179,176
Di-n-butyl phthalate	149	150,104
Carbazole	167	166,139
Fluoranthene	202	101,203
Benzidine	184	92,185
Pyrene	202	200,203
Butylbenzylphthalate	149	91,206
3,3-Dichlorobenzidine	252	254,126
Benzo(a)anthracene	228	229,226
Bis(2-ethylhexyl)phthalate	149	167,279
Chrysene	228	229,226
Di-n-octyl phthalate	149	167,43
Benzo(b)fluoranthene	252	253,125
Benzo(k)fluoranthene	252	253,125
Benzo(a)pyrene	252	253,125
Indeno(1,2,3-cd)pyrene	276	138,277
Dibenz(ah)anthracene	278	139,279
Benzo(ghi)perylene	276	138,277
N-Nitrosodiethylamine	102	42,57,44,56
N-Nitrosopyrrolidine	100	41,42,68,69
N-Nitrosomorpholine	56	116,86
Acetophenone	105	71,51,120
2,6-Dichlorophenol	162	63,98
α,α-Dimethylphenethylamine	58	91,65,134,42
N-Nitrosodi-n-butylamine	84	57,41,116,158
N-Nitrosopiperidine	114	42,55,56,41
O-toluidine	106	107,77,51,79
O,O,O-Triethylphosphorothioate	198	121,97,65
Hexachloropropene	213	211,215,117,106,141
Isosafrole	162	131,104,77,51
1-Chloronaphthalene	162	127,164
1-Naphthylamine	143	115,89,63
2-Naphthylamine	143	115,116
Pentachlorobenzene	250	252,108,248,215,254
1,2,4,5-Tetrachlorobenzene	216	214,179,108,143,218
2,3,4,6-Tetrachlorophenol	232	131,230,166,234,168
p-Phenylenediamene	108	80,53,54,52
Safrole	162	104,77,103,135
1,4-Naphthquinone	158	104,102,76,50,130
Thionazine	107	96,97,143,79,68
5-Nitro-o-toluidine	152	77,79,106,94
4-Aminobiphenyl	169	168,170,115
Diphenylamine	169	168,167
Pentachloronitrobenzene	237	142,214,249,295,265
Phenacetin	108	180,179,109,137,80
Pronamide	173	175,145,109,147
sym-Trinitrobenzene	75	213,120
Dinoseb	211	163, 240

TABLE 6 (cont.)

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

COMPOUND	PRIMARY ION	SECONDARY ION(S)
Diallate	86	234,43,70
4-Nitroquinoline-1-oxide	174	101,128,75,116
Methapyrilene	97	50,191,71
Isodrin	193	66,195,263,265,147
p-Dimethylaminoazobenzene	225	120,77,105,148,42
7,12-Dimethylbenz(a)anthracene	256	241,239,120
3-Methylcholanthrene	268	252,253,126,134,113
Aramite	185	191,319,334,197,321
Chlorobenzilate	251	139,253,111,141
3,3'-Dimethylbenzidine	212	106,196,180
2-Acetylaminofluorene	181	180,223,152
Dibenz(a,j)acridine	279	280,277,250
Hexachlorophene	196	198,209,21,406,408
Phenol-d6 (surrogate)	99	42,71
2-Fluorophenol (surrogate)	112	64
2,4,6-Tribromophenol (surrogate)	330	332,141
Nitrobenzene-d5 (surrogate)	82	128,54
2-Fluorobiphenyl (surrogate)	172	171
Terphenyl-d14 (surrogate)	244	122,212
1,4-Dichlorobenzene-d4 (istd.)	152	115,150
Naphthalene-d8 (istd.)	136	68
Acenaphthene-d10 (istd.)	164	162,160
Phenanthrene-d10 (istd.)	188	94,80
Chrysene-d12 (istd.)	240	120,236
Perylene-d12 (istd.)	264	260,265

Primary ions must not be changed except in unusual instances where interference occurs with a co-eluting non-target analyte. In this case, a secondary ion may be used for quantitation with the following rules:

(1) The corresponding standard(s) (initial calibration curve and continuing calibration standard) must be re-quantitated with the secondary ion.

(2) Approval must be obtained from the Department Manager or the laboratory Operations Manager.

The quantitation ion must then be changed back to the one specified in Table 4 after quantitation of the samples(s).

Secondary ions are recommended only and may be changed depending upon instrument conditions (sensitivity, etc.). However, it is Katahdin policy that a minimum of 2 ions (primary and one secondary) be used for all GC/MS analyses.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270.

FIGURE 1

EXAMPLE OF RUNLOG LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES
GC/MS SVOA INJ LOG INSTRUMENT: 5973-U

DATE OF DFTPP INJECTION: 010708

JOB	SAMPLE	DATAFILE	DF	ALS #	METHOD	UL INJ	CHEMIST	COMMENTS
	50 ng DFTPP	U0626	1	1	DFTPP390	2.0	JW	ok
B	SSTD050U0107	U0563		2	U8270C02	1.0		✓
	010	64		3				✓
	025	65		4				✓ Runs ok
	100	66		5				✓
	150	67		6				✓
	200	68		7				✓
A	SSTD050U0107	69 (a)		8				✓ (a) = UTCLP02
	010	70 (a)		9				✓ files
	025	71 (a)		10				✓
	100	72 (a)		11				✓
	150	73 (a)		12				✓
	200	74 (a)		13				✓
	8270 IND CHK	75 (a)		14				
				JW 010808				

STANDARD	CODE
DFTPP	51176
CAL. STD.	51181 51182
IS MIX	STC 3505

REVIEWED AND APPROVED BY: _____
DATE: _____

QAMS364

0000015

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270.

FIGURE 2

EXAMPLE OF GC/MS STANDARDS RECEIPT LOGBOOK ENTRY

KATAHDIN ANALYTICAL SERVICES

STOCK STANDARDS RECEIVED

GCMS LABORATORY
REVIEWED BY/DATE:

AMP0946

AccuStandard 125 Market St. • New Haven, CT 06513 • USA Tel. 203-786-5290 • www.accustandard.com

APP-9-176-D-20X
Pentachlorophenol
2.0 mg/mL in CH2Cl2
Lot: B3010100
Exp. Jan 10, 2013.

1 mL

FOR LABORATORY USE ONLY

WARNING: This product contains a chemical(s) known to the State of California to cause cancer.

STORAGE Ambient

POISON [redacted]

8/16/06
JZ

AMP0947

AccuStandard 125 Market St. • New Haven, CT 06513 • USA Tel. 203-786-5290 • www.accustandard.com

APP-9-090-50X
4,6-Dinitro-o-cresol
5.0 mg/mL in MeOH
Lot: B1100296
Exp. Aug 16, 2012

1 mL

FOR LABORATORY USE ONLY

STORAGE Ambient

FLAMMABLE [redacted]

AMP0948

AccuStandard 125 Market St. • New Haven, CT 06513 • USA Tel. 203-786-5290 • www.accustandard.com

APP-9-145-50X
p-Nitrophenol
5.0 mg/mL in MeOH
Lot: B5050205
Exp. May 18, 2015

1 mL

FOR LABORATORY USE ONLY

STORAGE Ambient

POISON [redacted]

QAMS294

0000045

TITLE: **ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270.**

FIGURE 3

EXAMPLE OF SVOA STANDARDS PREPARATION LOGBOOK ENTRY

GC/MS SVOA STANDARD PREP LOGBOOK

50863	8270 Stock (w/o MeOH)	3-15-06	7-7-06	JLW	ANP0844	8270 Megaplug	300	2-22-07	4.2ml	150 ng/ml
					ANP0837	↓	350	3-14-07		
					ANP0911	APP IX #2	600	3-2-07		
					ANP0910	↓ 1	100	3-9-07		
					ANP0570	↓ 1	200	7-7-06		
					ANP0659	Organophosphorus pest	300	8-19-06		
					ANP0917	Benzoic Acid		3-9-07		
					ANP0907	Hexachlorophene		2-22-07		
					ANP0906	Benzidine		3-9-07		
					ANP0936	3,3'-Dichlorobenzidine		3-14-07		
					ANP0932	8270 Surr	150	3-9-07		
					50861	DEA	300	3-13-07		
					B43590	MeCl2	550	—		
50864	8270 level 1	3-15-06	7-7-06	JLW	50863	8270 Stock	70	7-7-06	1.05ml	10 ng/ml
					B43590	MeCl2	980			
50865	8270 level 2	3-15-06	7-7-06	JLW	50863	8270 Stock	150	7-7-06	0.90ml	25 ng/ml
					B43590	MeCl2	750			
50866	8270 level 3	3-15-06	7-7-06	JLW	50863	8270 Stock	600	7-7-06	1.8ml	50 ng/ml
					B43590	MeCl2	1200			
50867	8270 level 4	3-15-06	7-7-06	JLW	50864	8270 Stock	700	7-7-06	1.05ml	100 ng/ml
					B43590	MeCl2	350			

Reviewed by/Date:

CAMS2041

0000157

TITLE: **ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**

Prepared By: GC/MS Department Date: 6/98

Approved By:

Group Supervisor: J Halay Date: 020101

Operations Manager: John C. Benton Date: 1/31/01

QA Officer: Deborah J. Nadeau Date: 1.31.01

General Manager: Dennis P. Lufan Date: 2/01/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 8270C Mod.	Format changes, added pollution prevention, added instrument and other calibration options. Other minor changes to sections 7,8 & QA Table.	DN	1.31.01	1.31.01
02 8270C	Many changes in formatting. Some additions to sections & Table 1 to comply with Navy.	DN	09.30.04	09.30.04
03 8270C	Sect. 7.2: Removed "K" Instrument & added "R" instrument. Added Pentafluorophenol surr. to Tables 3, 5 and Sect. 8.2. Removed all References to TIC's.	LAD	04/06	04/06
04 8270C	Sect. 8.2 - changed 5 to 4 and removed pentachlorophenol. Table 3 and 5 - removed pentachlorophenol. Changed linear regression correlation coefficient criteria. Added M1 SOP reference. Added LCS exceedance criteria. Added ICV requirement and criteria. Added RT window procedure.	LAD	06/07	06/07
05 8270C	Added "G" instrument, Removed "X" instrument Edited section 7.5.1 - initial cal. table	LAD	02/08	02/08

TITLE: **ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Section 5.3.2.3- Added calibration Mix B. Section 7.5.1- Edited to address diffent SIM compounds may need to be calibrated at different levels depending on the compound and project requirements.	LAD	04/09	04/09
07	Changes made for compliance with DoD QSM version 4.1	LAD	08/09	08/09
08	Updated Standard prep. Added compounds to Table 3 and 5. Updated references. Added DoD QSM QC requirements Table.	LAD	04/10	04/10

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document SOP CA-213-08, titled "**ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**".

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP CA-213-08, titled "**ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**".

Recipient: _____ Date: _____

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services, Inc. laboratory personnel to prepare and analyze water and soil sample extracts for semivolatile organics by EPA SW-846 Method 8270, current revision, modified for selected ion monitoring.

In order to maintain consistency in data quality, this SOP consolidates all aspects of the analyses in one working document, to be revised as necessary.

1.1 Definitions

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution that is different from the stock used to prepare standards.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

STOCK STANDARD SOLUTION: A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

QUICKFORMS: A laboratory reporting software for Target and Target DB. The QuickForms report module for Target is preconfigured with generalized forms and US EPA CLP report forms and disk deliverables, which can be customized.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of semivolatile organic compounds by EPA Method 8270. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, "Personnel Training & Documentation of Capability," current revision.

It is the responsibility of all Katahdin technical personnel involved in analysis of semivolatiles by Method 8270 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with the Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves, and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

After analysis, autosampler vials containing sample extracts in methylene chloride are returned to the SVOA hood, and the contents transferred to a labeled waste container. The contents of this container are disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD

The process involves the extraction of semivolatiles from a sample using an appropriate solvent followed by clean up steps (where applicable) and concentration of the extract (refer to Katahdin SOP CA-502, "Preparation of Aqueous Samples for Extractable Semivolatile Analyses", SOP CA-512, "Preparation of Sediment/Soil Samples by Sonication Using Method 3550 for Subsequent Extractable Semi-Volatiles Analysis" and SOP CA-526, "Preparation of Sediment/Soil Samples by Soxhlet Extraction Using Method 3540 for Subsequent Extractable Semivolatile Analysis"). An aliquot of the final extract is injected into the gas chromatograph

for compound separation by capillary column, followed by the electron impact mass spectrometer for identification and quantitation.

3.0 INTERFERENCES

Interfering contamination may occur when a sample containing low concentrations of SVOCs is analyzed immediately after a sample containing high concentrations of SVOCs. Any samples that have suspected carryover must be reanalyzed.

4.0 APPARATUS AND MATERIALS

4.1 GC: Hewlett Packard 5890 and/or 6890

4.2 Mass Spectrometers (MS): HP5973, HP5972 and/or HP5970

4.3 Helium: Carrier gas for routine applications. All carrier gas lines must be onstructed from stainless steel or copper tubing; non-polytetrafluoroethylene (non-PTFE) thread sealant or flow controllers with rubber component are not to be used.

4.4 Autosamplers: HP 7673As

4.5 Hamilton syringes: 2.00 uL to 10 mL

4.6 Volumetric glassware: Grade A or equivalent

4.7 Columns: DB-5MS 30m, 0.25mm I.D., 25um film thickness, columns (J&W Scientific) or equivalent.

4.8 Acquisition System: The acquisition system must be interfaced to the MS and allow continuous acquisition of data throughout the duration of the chromatographic program. It must permit, at a minimum, the output of time vs. intensity (peak height or peak area). Hewlett Packard Chemstation or equivalent.

4.9 Data System: The Target software is used for processing data and generating forms.

5.0 REAGENTS

5.1 J.T. Baker Ultra Resi-Analyzed methylene chloride (or equivalent)

5.2 Purge and trap grade methanol

5.3 Standards: Stock standards and working standards are received and recorded in accordance with SOP CA-106 "Standard Preparation and Documentation".

5.3.1 The expiration date for all standards is one year from date of opening the ampule. If the manufacturer's expiration date is before this one year date, the manufacturer's expiration must be followed. New standards must be opened if degradation is observed.

5.3.2 Secondary dilution standards

5.3.2.1 The standards are prepared on an as needed basis (or every 6 months) and stored in screw-cap amber bottles with Teflon liners in the BNA standards freezer between uses. Standards prepared from various stock solutions must always use the first expiration date of any of the solutions used for preparation.

5.3.2.2 Calibration Mix A – Prepare standards in methylene chloride containing the compounds listed in Table 3. The final concentration of each compound is 20 ug/mL.

5.3.2.3 Calibration Mix B - Some compounds must be calibrated at higher concentrations. For these compounds a secondary standard is prepared which will "boost" the concentration of these compounds in the initial calibration. The concentration of this standard is determined on a project to project basis.

5.3.2.4 Internal Standard Solution – Prepare standard in methylene chloride containing 1,4-dichlorobenzene-d4, naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12, and perylene-d12 at a final concentration of 80 ug/mL.

5.3.2.5 DFTPP Solution – Prepare standard in methylene chloride containing DFTPP at a final concentration of 25 ug/mL.

5.3.2.6 Independent Calibration Verification (ICV) Standard – From a source independent of the calibration standards, prepare a standard in methylene chloride containing the compounds listed in Table 3. The final concentration of each compound is 2 ug/mL.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

All semivolatile sample extracts must be analyzed within forty days following the date of extraction.

7.0 PROCEDURES

7.1 NAMING AND CODING CONVENTIONS FOR ANALYTICAL STANDARDS – Used in accordance with SOP CA-106 "Standard Preparation and Documentation".

7.2 COMPUTER (DATA SYSTEM) CONVENTIONS -

Conventions for all instruments are as follows:

Sub-Directory for data acquisition and storage: C:\HPCHEM\1\DATA
Tune file: DFTPP.U

Method files: LSPSIMXX.M (all samples and standards)
Where:
XX = the calibration number in chronological order
L = instrument ID (R, U, or G)
DFTPP390.M (DFTPP tuning acquisition)

NOTE: All acquisition parameters must be identical for LSPSIMXX.M and DFTPP2. M.

Data Files: L_ _ _ _.D, where _ _ _ _ is a number in chronological order from 0001 to 9999 and L is the instrument ID (R, U, or G). This file also contains the Quantitation output file.

Data Files for DFTPP: LD_ _ _.D, where _ _ _ is a number in chronological order from 001 to 999 and L is the instrument ID (R, U, or G).

7.3 INSTRUMENT SPECIFIC PROCEDURES

It is the policy of the GC/MS group that all data be acquired in the batch mode. The following items must be checked prior to data acquisition in the batch mode:

- Ensure that the proper sequence and tune files are being used.
- Check the autosampler syringe (Is it clean? Does the plunger move freely? etc.), its alignment and make sure the solvent rinse vial is full. Ensure that the knurled nut holding the top of the syringe plunger is tight.
- Look at the batch to be analyzed and check the following:

  -Make sure that the data files are in numerical order with no duplication and that the method file is the same as that used for ICAL or Continuing Calibration analysis.

-Bottle numbers match with the numbers on the autosampler tray.

After the batch has been deemed free of errors, start the batch by using the "Position and run" command under the SEQUENCE menu in MSTop.

7.4 INSTRUMENT TUNING - Prior to the analysis of any calibration standards, blanks or samples, the GC/MS system must be shown to meet the mass spectral key ion and ion abundance criteria for decafluorotriphenylphosphine (DFTPP) tabulated below. Pentachlorophenol, benzidine and DDT are also present in this standard.

DFTPP Key Ions and Ion Abundance Criteria	
Mass	**Criteria**
51	30.0-80.0 percent of mass 198
68	less than 2.0 percent of mass 69
69	present
70	less than 2.0 percent of mass 69
127	40.0 – 60.0 percent of mass 198
197	less than 1.0 percent of mass 198
198	base peak, 100 percent of mass 198
199	5.0-9.0 percent of mass 198
275	10.0-30.0 percent of mass 198
365	greater than 1.00 percent of mass 198
441	present, but less than mass 443
442	greater than 40.0 percent of mass 198
443	17.0-23.0 percent of mass 442

All ion abundances must be normalized to m/z 198, the nominal base peak.

The following are the GC/MS operating conditions for injection of DFTPP.

GC/MS OPERATING CONDITIONS - DFTPP	
Initial column temperature hold	140°C for 3 minutes
Column temperature program	140-275°C at 15 degrees/minute
Final column temperature hold	275°C
Injection port temperature	280°C
Transfer line/source temperature	285°C
Injector - splitless, valve time	0.18 minutes
EPC	inlet B
Constant flow	ON
Constant flow pressure	10psi

Constant flow temperature	30°C
Vacuum comp.	ON
Run time	10-12 minutes
Scan start time	5.0 minutes
Sample volume	2.0 uL of 25 ng/uL DFTPP solution
Carrier gas	helium at @ 1.0 mL/minute
Mass range	35 to 500 amu
Number of A/D samples	4
GC Peak threshold	500 counts
Threshold	10 counts

Set up the run on the Enviroquant system using "Edit Sample Log Table". For a more detailed explanation of the Enviroquant software, consult the appropriate manual, Organic Department Manager, or senior chemist within the GC/MS group.

The DFTPP solution must be analyzed once at the beginning of each twelve hour period during which standards and/or samples are analyzed. The 12 hour time period for GC/MS system begins at the moment of injection of the DFTPP analysis. The time period ends after twelve hours has elapsed according to the system clock. The last injection must be accomplished prior to the expiration of 12 hours; conceivably, the run-time of an injection could end after the twelve hours.

When the DFTPP has concluded, the run must be evaluated to determine if sample analysis can proceed. The chromatography and the ion ratios must be examined. The DFTPP run is processed using the current algorithms in the Target software.

If the results indicate the system does not meet acceptance criteria, the GC/MS must be manually tuned. Once the manual tune procedure is completed, DFTPP must be re-injected and reevaluated. If the instrument still does not meet criteria, notify your Department Manager. Under no circumstances should calibration proceed if the instrument DFTPP is not in criteria.

7.5 INSTRUMENT CALIBRATION

7.5.1 Initial Calibration for Method 8270-SIM

Prior to the analysis of samples and required method blanks, and after the instrument DFTPP tuning criteria have been met, the GC/MS system must be calibrated at six different concentrations, typically, 0.20, 0.50, 1.0, 2.0, 5.0 and 8.0 ng/uL. This is done to determine instrument sensitivity and the linearity of GC/MS response for the semivolatile target and surrogate compounds.

Some SIM compounds may need to be calibrated at higher concentrations. A second standard is prepared containing these compounds. The two

standards are combined as in the example below. A 100 uL aliquot of each of the standards above is spiked with 1 uL of internal standards and analyzed.

Example –
For a calibration at the following levels:
Calibration mix A would be prepared containing ALL analytes at 20 ng/ul
Calibration Mix B would be prepared containing phenols and phthalates at 20 ng/ul.

Final PAH conc. (ng/uL)	Final Conc. Phenols and phthalates (ng/ul)	Cal-Mix A Added (uL)	Cal-Mix B Added (uL)	$MeCl_2$ Added (uL)	Final Volume (uL)
0.20	1.0	10	40	950	1000
0.50	2.0	25	75	900	1000
1.0	3.0	50	100	850	1000
2.0	4.0	100	100	800	1000
5.0	5.0	250	0	750	1000
8.0	8.0	400	0	600	1000

Note: Calibration Mix B only is used to boost the phenols and phthalates concentrations in Cal. levels 1 through 4.

The GC/MS operating conditions for the calibration standards injections are the same as for the DFTPP with the following exceptions:

GC/MS OPERATING CONDITIONS – CALIBRATION and SAMPLES	
Column temperature program	40°C for 3 min. to 300°C at 10°/min.
Final column temperature hold	300°C
Run time	35 minutes (time may vary dependent upon column length)
Scan start time	2.0-6.0 minutes (time may vary dependent upon column length)
Sample volume	1 uL

The conditions are set up in the method file LSPSIMXX.M

After analysis of the five calibration points, they must be quantitated and evaluated for adherence to QC criteria. Minimum requirements of ID files are the use of specific quantitation ions and quantitating a specific set of targets and surrogates with a set internal standard. Of particular importance when performing SIM analysis are the ion ratios. These requirements are found in Tables 3 and 5.

7.5.2 Initial Calibration Criteria

Relative response factors (RRFs) must be calculated and evaluated for each target compound and surrogate. The RRF is defined as follows:

$$RRF = \frac{Ax}{A_{IS}} \times \frac{C_{IS}}{Cx}$$

where:
- Ax = area of the primary ion for the target compound
- A_{IS} = area of the primary ion for the corresponding istd
- C_{IS} = concentration of the istd (ng/uL)
- C_x = concentration of the target compound

After the calibration points have been quantitated, update the calibration curve points using the Target data processing software to generate the RRF's and %RSD's for all analytes. If information is needed concerning the use of these programs, consult the Organic Department Manager or a senior chemist within the group.

Response factor criteria have been established for the calibration of the semivolatile target and surrogate compounds. These criteria must be met in order for the calibration curve to be considered valid. The percent RSD for each calibration check compound (CCC) must be less than or equal to 30 percent. There are three CCC's: Acenaphthene, Fluoranthene, and Benzo(a)pyrene. There are no criteria for the SPCC compounds. This is also applicable to clients that request DOD criteria.

7.5.2.1 Linearity of Target Analytes (This is also applicable to clients that request DOD criteria.)

If the RSD of any target analyte is 15% or less, then the response factor is presumed to be constant over the calibration range, and the average response factor may be used for quantitation.

If the RSD of any target analyte exceeds 15%, then a calibration option outlined in section 7.0 of method 8000 will need to be employed.
Option 1 (Section 7.5.2 of method 8000 - Rev. 2, 12/96), is a linear regression of instrument response versus the standard concentration.

The correlation coefficient (r) for each target analyte and surrogate must be greater than or equal to 0.995. Target software calculates the

correlation coefficient squared (r^2). This must be equal to or greater than 0.990.

Option 2 (Section 7.5.3 of method 8000 - Rev. 2, 12/96), is a non-linear calibration model not to exceed a third order polynomial. The lab would use a quadratic model or second order polynomial. The use of a quadratic model requires six calibration points. In order for the quadratic model to be acceptable, the coefficient of determination must be greater than or equal to 0.990.
If time remains in the clock after meeting the initial calibration acceptance criteria, samples may be analyzed. The calibration must be verified each twelve hour time period (time period starts from the moment of the DFTPP injection) for Method 8270-SIM. The SSTD1.0 in the curve may be used as the continuing calibration standard as long as it meets the continuing calibration acceptance criteria. All sample results must be quantitated using the initial calibration response factors.

7.5.2.2 Immediately following calibration an Independent Calibration Verification Standard must be analyzed. For clients requiring DOD criteria, all project analytes must be within +/- 20% of true value.

7.5.3 Continuing Calibration

A check of the calibration curve must be performed once every twelve hours immediately following analysis of the tuning compound DFTPP. This check contains all target compounds and surrogates at a concentration of 1.0 ng/uL.

After quantitation of the 1.0 ng/uL continuing calibration check, response factors must be calculated and compared to the average response factors in the initial calibration. The Target program calculates the calibration check response factors and compares them to the average RFs in the calibration curve by calculating percent differences. The method 8270 CCC's must have a % difference of +/- 20%D in order to be considered in criteria. These conditions must be met before method blank and/or sample analysis can begin. For clients requiring DOD criteria, all project analytes and surrogates must be within +/- 20%.

If the continuing calibration check does not meet criteria, corrective action must be taken. Depending on the situation, corrective action may be as follows:

- Re-analyze the 1.0 ng/uL continuing calibration check.

- Change the septum; clean the injection port; install a clean, silanized quartz liner; cut off a small portion (1" to 3") of the front end of the capillary column. This is usually performed when chromatography is poor. Record any of these actions in the appropriate instrument maintenance logbook.

- Analyze a new initial calibration curve.

The last option, the generation of a new initial calibration curve, is usually chosen when percent difference are >30%. In these instances, there is little or no chance of a continuing calibration reanalysis meeting criteria. If there is any doubt concerning which corrective action to undertake, consult the Organic Department Manager or a senior chemist within the group.

If the continuing calibration does meet the criteria specified above then analysis may proceed using initial calibration response factors.

7.5.4. Retention Time Windows

Retention time windows are set at the midpoint standard of the calibration curve, following every ICAL. When a CV is analyzed (and not an ICAL), the retention time windows of the daily CV must be within 30 seconds of the midpoint calibration standard of the most recent ICAL. The samples analyzed following the daily CV must have retention times within 30 seconds of those for the daily CV. Each successive daily CV must be compared to the most recent ICAL midpoint standard.

7.6 SAMPLE ANALYSIS

Sample extracts may be analyzed only after the GC/MS system has met tuning criteria, initial calibration and continuing calibration requirements. Ensure that the same instrument conditions are being used for tuning, calibration and sample analysis by reviewing the GC parameters using the "Edit entire method" option under the Method menu in MSTOP. Note that you can not edit a method if the instrument is running.

Extracts are stored in the refrigerator in the organics extraction laboratory at 4°C ±2°C. Remove them from the refrigerator and place them in the GC/MS laboratory semivolatile hood when ready for analysis.

Prepare a 1.8 mL clear glass vial (crimp top) with a disposable insert (350 uL). Add 100 uL of sample extract and 1.0 uL of the 80 ng/uL IS stock to the vial and then cap. This gives a 0.8 ng/uL final concentration for the internal standard compounds. The samples are topped with Teflon lined crimp top caps.

7.7 FINAL DATA PACKAGE

7.7.1 Initial Data Review (IDR)

The initial data review is accomplished by the analyst who ran the samples and is a review of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed sample. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed:

- Surrogate Recoveries
- Internal Standard Area Stability
- Method Blank Acceptance
- Chromatography
- Target Compound Detection/Quantitation/Review for false positives
- Laboratory Control Sample Recoveries
- Matrix Spike/Matrix Spike Duplicate Recoveries

The analyst must evaluate all data using the QA Acceptance Criteria table found within this SOP (Table 1). This table gives acceptance criteria and corrective actions for criteria that are not met. In addition to evaluating QC elements, the chromatography and quantitation of target analytes must be reviewed. During this review, the analyst checks the integration of each individual peak. The hardcopy has false positives crossed out so they can be reviewed for appropriateness by the Organic Department Manager.

7.7.2 Chromatography

The chromatography should be examined for the presence or absence of any ghost peaks and can also be used as an indication of whether or not matrix interferences might be affecting surrogate recoveries and/or istd area recoveries. Whether or not the chromatography is acceptable is a judgment call on the part of the analyst and should be used in conjunction with other monitored QC (e.g. surrogate recoveries) to determine the necessity of reanalyzing.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of

concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), an "m" qualifier will automatically be printed on the quantitation report summary.

This manual integration package must then be submitted to the Department Manager or his/her designee, who will review each manual integration.

For specific manual integration procedures, refer to Katahdin SOP QA-812, "Manual Integration", current revision.

7.7.3 Target Compound Detection/Quantitation

The semivolatile ID files have been set up to err on the side of false positives; that is to identify and quantitate peaks as target compounds that may not necessarily be valid hits. It is the responsibility of the GC/MS analyst to use his/her technical judgment to determine if the identification of a target compound is correct or not.

If any target concentration exceeds the upper limit, a dilution must be made and analyzed. The dilution chosen should keep the concentration of the largest target compound hit in the upper half of the initial calibration range. LCS and MS/MSD samples need not be diluted to get spiked analytes within the calibration range.

The requirements for qualitative verification by comparison of mass spectra are as follows:

- All ions present in the standard mass spectra at a relative intensity > 10% must be present in the sample spectrum.
- The relative intensities of primary and secondary ions must agree within ±20% between the standard and sample spectra.
- Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst.

If a compound cannot be verified by all three criteria above, but, in the technical judgment of the mass spectral interpretation specialist, the

identification is correct, then the laboratory shall report that compound on the Form 1 as a valid hit.

The GC/MS laboratory initial data review must be completed within twelve hours of batch completion; in the majority of instances, the initial review should be accomplished at the beginning of a work shift for the previous set of analyses.

7.7.4 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into QuickForms. Depending on the QC label requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A complete review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

7.8 INJECTION PORT LINER CLEANING AND SILANIZING PROCEDURE

- Remove the rubber o-ring from the liner and place the liner in a large Erlenmeyer flask.
- In the hood, pour nitric acid into the flask until the liner is covered. Place the flask on a hotplate and boil for 2-3 hours.
- Let cool; drain nitric acid and thoroughly flush the liner with water.
- Bake briefly in the muffle oven until liner is dry and cool to room temperature.
- Place the liner in a beaker, fill with Sylon and let it soak for at least two hours.
- Take out the liner and rinse it thoroughly with toluene.
- Rinse the liner thoroughly with purge and trap grade methanol.
- Bake the liner in the muffle oven for a minimum of three hours.

7.9 Instrument Maintenance

Instrument preventative maintenance is performed on a semi-annual basis by GC/MS chemists. This maintenance includes a thorough inspection and cleaning of all parts, including changing rough and turbopump oils. GC/MS analysts perform other maintenance on an as-needed basis. Typically, routine maintenance involves clipping off the front end of the DB-5MS column, replacing the injection port septum, and installing a freshly silanized quartz liner after sample analysis.

All maintenance must be documented in the instrument-specific maintenance log, whether it is routine or not. The Department Manager must authorize any maintenance over and above a routine source cleaning.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 Method Blank Criteria

A method blank is defined as a volume of a clean reference material (deionized distilled water for water samples, baked organic-free sand for soil/sediment matrices)

that is carried through the entire analytical procedure. One method blank must be extracted with each group of samples of a similar matrix and must be analyzed on the GC/MS system that was used to analyze the samples.
An acceptable method blank must contain less than or equal to the PQL of any target compound. For clients requiring DOD criteria, no analytes detected at > ½ PQL and > 1/10 the amount measrued in any sample or 1/10 the regulatory limit.

If the method blank exceeds these contamination levels, the analytical system is considered out of control and corrective action must be taken before sample analysis.

Reanalysis of the blank is the first step of the corrective action; if that does not solve the problem, a Katahdin Corrective Action Report (CAR) will be initiated. Corrective action will be specified after consultation including the Department Manager, Operations Manager, and QA Officer.

8.2 Surrogate Recoveries

The four surrogates (2-Methylnaphthalene-d10, 2,4-Dibromophenol, Fluorene-d10 and Pyrene-d10) must meet the current statistically derived acceptance limits. If statistical limits have not been established then the surrogate recovery must meet the nominal limits of 30-150%. For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

If specifications are not met, the sample (or blank) should be reanalyzed. If specifications are met in the reanalysis, this reanalysis should only be submitted. If surrogate specifications are not met in the sample or method blank reanalysis, a Corrective Action Report (CAR) should be initiated. Corrective action will be specified after consultation including the Department Manager and Operations Manager.

For further information regarding the acceptance of surrogate recoveries, consult the Organic Department Manager.

8.3 Internal Standard Responses

Internal standard responses and retention times (RT) in all samples and blanks must be evaluated as part of the technical data review. The method files have been set up to only detect compounds that fall within a set RT window. For Method 8270-SIM analysis, if the extracted ion current profile (EICP) area for any internal standard changes by more than a factor of two (-50% to +100%) as compared to the daily continuing calibration standard, reanalysis must occur. If the reanalysis meets criteria, only the in-criteria run should be reported. If the reanalysis is still out of criteria, both analyses should be included in the sample package set.
MS/MSD samples that do not meet the EICP area criteria above do not have to be reanalyzed.

8.4 Laboratory Control Sample (LCS)

An LCS must be performed for each group of samples of a similar matrix, for the following, whichever is more frequent:

- Every 20 samples of a similar matrix or similar concentration, or
- Every batch of samples extracted.

Statistical limits are compiled annually for LCS recoveries (archived in QA office). Statistical limits are only calculated when at least 20 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the section supervisor, Laboratory Operations Manager and Quality Assurance Officer. Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts", current revision.

The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Organic Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 30-150% may be used for some projects or states (i.e. South Carolina). For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

The LCS recoveries for all analytes are evaluated. All of the compounds of interest must fall within the established statistical limits with the following sporadic exceedance allowances.

Number of Analytes	Number of Allowable Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
<11	0

If less than the number of allowable exceedances fail the statistical limits, no corrective action is needed. If greater than the number of allowable exceedances fail the statistical limits, corrective action may be taken. Corrective actions may vary with each situation. However, in the case where the failures are high and the samples are non-detect for those compounds, then no corrective action is required. Otherwise, corrective action may involve reanalysis or recalibration. The specific corrective actions taken will rely on analyst experience to make sound scientific judgments while

considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time

Please note that for compounds with only nominal limits (i.e. insufficient data points were available to generate statistical limits), no corrective action is required for out-of-criteria recoveries until enough data points are established to generate statistical limits.

8.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Criteria

Matrix Spike and Matrix Spike Duplicates must be extracted and analyzed for each group of up to 20 samples of a similar matrix or similar concentration. In the event insufficient sample volume is available an LCS/LCS Duplicate is extracted and analyzed in place of the MS/MSD.

Statistical limits are compiled annually for MS/MSD recoveries for a short list of the spiked compounds (Acenaphthene, Pentachlorophenol and Pyrene). Nominal limits of 30-130% are used for all other compounds. Generally, corrective action is only taken for the short list of the spiked compounds. The specific corrective actions will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time. For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

A Corrective Action Report (CAR) must be filled out and filed if any criteria for percent recovery or relative percent difference are not met to document any decisions with reporting data.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of Method 8270 for other method performance parameters and requirements.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods", SW-846, third Edition, Final Updates I, II, IIA, III, IIIA and IIIB, November 2004, Method 8270C.

"USEPA Contract Laboratory Program Statement of Work for Organics Analysis," Rev. 02/88.

Code of Federal Regulations (40 CFR), Part 136, Appendix A, Rev. June, 1998.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM), Version 4.1, 04/22/09

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

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TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Check of mass spectral ion intensities using DFTPP	Prior to initial calibration and calibration verification	Refer to the criteria listed in Section 7.4	Retune instrument, and verify
Six-point initial calibration for all analytes	Initial calibration prior to sample analysis	RSD ≤30 for RFs of the CCCs; Average %RSD < 15% for all compounds. Refer to section 7.5.2.1 for more details.	Repeat calibration if criterion is not met
Independent calibration verification	Once after Initial calibration	± 20 % D	1) Reanalyze standard 2) Reprep standard 3) Reprep standard from fresh stock.
Continuing calibration verification	Once per each 12 hours, prior to sample analysis	CCCs ≤ 20%D	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification
ISs	Immediately after or during data acquisition of calibration check standard	Retention time ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning
Demonstration of ability to generate acceptable accuracy and precision	Once per analyst initially and annually thereafter	All recoveries within method QC acceptance limits.	Recalculate results; locate and fix problem; reextract/reanalyze P&A study for those analytes that did not meet criteria
Method blank	One per prep batch	No analytes detected > PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e.If the blank results are above the PQL, report samples that are <PQL or > 10X the blank result. Reprep a blank and the remaining samples.
LCS for all analytes	One LCS per prep batch	Statistically derived from lab data or nominal limits depending on the project. . See also section 8.4 of this SOP for more information on allowable exceedances.	(1) Evaluate the samples and associated QC: i.e.If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS rec. is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Surrogate spike	Every sample, control, standard, and method blank	Statistically derived limits.	(1) Check chromatogram for interference; if found, flag data (2) If not found, check instrument performance; if problem is found, correct and reanalyze (3) If still out reextract and analyze sample (4) If reanalysis is out, flag data
MS/MSD	One MS/MSD per every 20 samples	Statistically derived from lab data or nominal limits depending on the project. Nominal limits are used as default limits.	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable reprep the samples and QC.
MDL and/or LOD/LOQ Verification study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TABLE 2

DoD QSM QC REQUIREMENTS

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-specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria.	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	Refer to current revision of SOP QA-806				
LOQ establishment and verification	Refer to current revision of SOP QA-806				
Tuning	Prior to ICAL and at the beginning of each 12-hour period.	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Breakdown check (DDT Method 8270 only)	Correct problem then repeat breakdown check.	Degradation ≤ 20% for DDT. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2.	At the beginning of each 12-hour period, prior to analysis of samples.	Flagging criteria are not appropriate.	No samples shall be run until degradation ≤ 20%.
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	1. Average response factor (RF) for SPCCs ≥ 0.050. 2. RSD for RFs for CCCs ≤ 30% and one option below: Option 1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression r ≥ 0.995; Option 3: non-linear regression– coefficient of determination (COD) r2 ≥ 0.99 (6 points shall be used for second order).	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin.
Second source calibration verification (ICV)	Once after each ICAL.	All project analytes within ± 20% of true value.	Correct problem and verify second source standard. Rerun second source verification. 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.

TABLE 2 (cont)

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention time window position establishment for each analyte and surrogate	Once per ICAL.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	
Evaluation of relative retention times (RRT)	With each sample.	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	Laboratories may update the retention times based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping). With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ±0.06 RRT units since the last update, this indicates a significant change in system performance and the laboratory must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the retention times.
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	1. Average RF for SPCCs ≥ 0.050. 2. %Difference/Drift for all target compounds and surrogates ≤ 20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration).	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	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 last acceptable CCV.	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.
Internal standards verification	Every field sample, standard, and QC sample.	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.

TABLE 2 (cont)

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > ½ RL (> RL for common lab contaminants) and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result. Contact Client if samples cannot be reprepped within hold time.	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.
LCS containing all analytes to be reported, including surrogates	One per preparatory batch.	The laboratory shall use laboratory control limits (CLs) or use DoD-generated LCS-CLs, if available depending on project requirements. In-house CLs may not be greater than ± 3 times the standard deviation of the mean LCS recovery. A number of analytes may fall outside the CL but within marginal exceedance limit depending on the total number of analytes in the LCS.	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. Refer to Table G-1 for number of marginal exceedences allowed. Contact Client if samples cannot be reprepped within hold time.	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.
Matrix Spike (MS)	One per preparatory batch per matrix if sufficient sample is available.	For matrix evaluation, use LCS acceptance criteria.	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.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix if sufficient sample is available.	MSD: For matrix evaluation, use LCS acceptance criteria. MS/MSD: RPD ≤ 30%.	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.

TABLE 2 (cont)

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate spike	All field and QC samples.	The laboratory shall use laboratory surrogate CLs or use DoD-generated surrogate CLs, if available depending on project requirements. .	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Contact Client if samples cannot be reprepped within hold time.	Apply Q-flag to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-213-08	METHOD 8270, current revision
Apparatus/Materials	none	
Reagents	none	
Sample preservation/ handling	none	
Procedures	none	
QC - Spikes	none	
QC - LCS	none	
QC - Accuracy/Precision	none	
QC - MDL	none	

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 4

ANALYTE QUANITIATION AND INTERNAL STANDARDS

Internal Standard: 1,4-dichlorobenzene-d4	2,4-Dinitrotoluene
Target and Surrogates:	2,4-Dinitrophenol
1,4-Dioxane	2,3,4,6-Tetrachlorophenol
Benzaldehyde	Diethylphthalate
Phenol	4-Chlorophenyl-phenyl ether
bis(2-Chloroethyl)ether	4,6-Dinitro-2-methylphenol
2-Chlorophenol	N-nitrosodiphenylamine
2-Methylphenol	2-Nitroaniline
3&4-Methylphenol	3-Nitroaniline
2,2'-Oxybis(1-chloropropane)	4-Nitroaniline
Nitrobenzene	Dibenzofuran
Hexachloroethane	4-Nitrophenol
Acetophenone	**Internal Standard: Phenanthrene-d10**
N-nitroso-di-n-propylamine	Target and Surrogates:
Internal Standard: Naphthalene-d8	Pentachlorophenol
Target and Surrogates:	1-Methylphenanthrene (dredge)
Naphthalene	Phenanthrene
1-Methylnaphthalene (dredge)	Hexachlorobenzene (special)
2-Methylnaphthalene	Anthracene
2-Methylnaphthalene-D10 (surrogate)	Fluoranthene
Isophorone	Carbazole
2-Nitrophenol	Di-n-butylphthalate
2,4-Dimethylphenol	4-Bromophenyl-phenyl ether
bis(2-Chloroethoxy)methane	Atrazine
2,4-Dichlorophenol	**Internal Standard: Chrysene-d12**
4-Chloroaniline	Target and Surrogates:
Hexachlorobutadiene	Butylbenzylphthalate
Caprolactam	3,3'-Dichlorobenzidine
4-Chloro-3-methylphenol	Pyrene
Internal Standard: Acenaphthene-d10	Benzo(a)Anthracene
Target and Surrogates:	Chrysene
1,1'-Biphenyl (dredge)	Bis-(2-ethylhexyl)phthalate
2,6 Dimethylnapthalene (dredge)	Pyrene-d10 (surrogate)
Acenaphthylene	**Internal Standard: Perylene-d12**
Acenaphthene	Target and Surrogates:
Fluorene	Perylene (dredge)
2-Fluorene-d10 (surrogate)	Benzo(b)fluoranthene
2,4-Dibromophenol (surrogate)	Benzo(k)fluoranthene
2-Chloronaphthalene	Benzo(e)pyrene (dredge)
Hexachlorocyclopentadiene	Di-n-octylphthalate
2,4,6-Trichlorophenol	Benzo(a)pyrene
2,4,5-Trichlorophenol	Indeno(1,2,3-cd)pyrene
Dimethylphthalate	Dibenz(a,h)anthracene
2,6-Dinitrotoluene	Benzo(ghi)perylene

TABLE 5

PROCEDURE CONDENSATION

Clock

12 hours from injection of 50ng DFTPP.

Calibration Curve Criteria

<30% RSD for CCCS
<15% RSD average for all analytes in calibration standard

Continuing Calibration Check Criteria

<20% D for CCC compounds

Additional QC

LCS every extraction batch
MS/MSD every 20 samples

TABLE 6

SVOA COMPOUNDS AND CHARACERISTIC IONS

COMPOUND	PRIMARY ION	SECONDARY IONS
1,4-Dioxane	88	58
Benzaldehyde	77	105,106
Phenol	94	65,66
bis(2-Chloroethyl)ether	93	63,95
2-Chlorophenol	128	64,130
1,4-Dichlorobenzene-d4 (IS)	152	150,115
2,2'-Oxybis(1-choropropane)	45	77,121
2-Methylphenol	108	107,77
Acetophenone	105	77,51
N-nitroso-di-n-propylamine	70	52,101
Hexachloroethane	117	201,199
3&4-Methylphenol	108	107,77
Nitrobenzene	77	123,51
Isophorone	82	54,138
2-Nitrophenol	139	109,81
2,4-Dimethylphenol	107	122,121
bis(2-Chloroethoxy)methane	93	63,123
2,4-Dichlorophenol	162	164,98
Naphthalene-d8 (IS)	136	137,134
Naphthalene	128	129,127
4-Chloroaniline	127	129
Hexachlorobutadiene	225	223,227
Caprolactam	113	55,56
4-Chloro-3-methylphenol	107	77,142
2,4-Dibromophenol (surr)	252	63,143
2-Methylnaphthalene-d10 (surr)	152	150
2-Methylnaphthalene	142	141,115
1-Methylnaphthalene	142	141,115
Hexachlorocyclopentadiene	237	235,239
2,4,6-Trichlorophenol	196	198,200
2,4,5-Trichlorophenol	196	198,200
2-Chloronaphthalene	162	127,164
1,1'-Biphenyl	154	153,76
2-Nitroaniline	65	92,138
Dimethylphthalate	163	194,164
2,6-Dinitrotoluene	165	63,89
Acenaphthylene	152	151,153
Acenaphthene	152	154,152
Acenaphthene-d10 (IS)	164	162
3-Nitroaniline	138	65,92
2,4-Dinitrophenol	184	107
Dibenzofuran	168	139

TABLE 6 (cont.)

SVOA COMPOUNDS AND CHARACERISTIC IONS

COMPOUND	PRIMARY ION	SECONDARY IONS
2,4-Dinitrotoluene	165	63
4-Nitrophenol	109	139,65
2,3,4,6-Ttrachlorophenol	232	230
Diethylphthalate	149	177,176
Fluorene-d10 (surr)	176	174,178
Fluorene	166	165
4-Chlorophenyl-phenyl ether	204	206,141
4-Nitroaniline	138	108,65
4,6-Dinitro-2-methylphenol	198	121
N-nitrosodiphenylamine	169	168,167
4-Bromophenyl-phenyl ether	248	250,141
Hexachlorobenzene	284	142,249
Atrazine	200	173,215
Pentachlorophenol	266	264,268
Phenanthrene-d10 (IS)	188	189
Phenanthrene	178	179,176
Anthracene	178	179,176
Carbazole	167	166,139
Di-n-butylphthalate	149	150,104
Fluoranthene	202	200,203
Pyrene	202	200,201
Pyrene-d10 (surr)	212	210,106
Butylbenzylphthalate	149	91,206
Benzo(a)anthracene	228	229,226
Chrysene-d12 (IS)	240	236,120
3,3-Dichlorobenzidine	252	254,126
Chrysene	228	226,229
bis(2-Ethylhexyl)phthalate	149	167
Di-n-octylphthalate	149	150
Benzo(b)fluoranthene	252	253,125
Benzo(k)fluoranthene	252	253,125
Benzo(a)pyrene	252	253,250
Perylene-d12 (IS)	264	260
Indeno(1,2,3-cd)pyrene	276	277
Dibenzo(a,h)anthracene	278	279
Benzo(g,h,i)perylene	276	277

Primary ions must not be changed except in unusual instances where interference occurs with a co-eluting non-target analyte. In this case, a secondary ion may be used for quantitation with the following rules:
(1) The corresponding standard(s) (initial calibration curve and continuing calibration standard) must be re-quantitated with the secondary ion.

TABLE 6 (cont.)

SVOA COMPOUNDS AND CHARACERISTIC IONS

(2) Approval must be obtained from the Organic Department Manager or the laboratory Operations Manager.

The quantitation ion must then be changed back to the one specified in the table above after quantitation of the samples(s).

Secondary ions are recommended only and may be changed depending upon instrument conditions (sensitivity, etc.). However, it is Katahdin policy that a minimum of 2 ions (primary and one secondary) be used for all GC/MS analyses.

TITLE: **ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**

FIGURE 1

EXAMPLE OF RUNLOG LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES
GC/MS SVOA INJ LOG INSTRUMENT: 5970-X

DATE OF DFTPP INJECTION: 03/009

JOB	SAMPLE	DATAFILE	DF	ALS #	METHOD	UL INJ	CHEMIST	COMMENTS
	50ng DFTPP	X0600	1	1	DFTPP390	2.0	JK	OK
	SSTD060X0316	X9437		2	X625A022	1.0		✓
	150	38		3				✓
	100	39		4				✓
	30	40		5				✓
	05	41		6				✓
	3560 MIX 1	42		7				OK
	2	43		8				
	3	44		9				
	4	45		10				
	5	46		11				
	6	47		12				
	7	48		13				
	8	49		14				
	9	50		15				
	BLANK 1	51		16				
	2	52		17				

STANDARD	CODE
DFTPP	50856
CAL. STD.	5089-30 5089-49
IS MIX	AMP0934

REVIEWED AND APPROVED BY: _____
DATE: _____

QAMS302

00000034

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

FIGURE 2

EXAMPLE OF GC/MS STANDARDS RECEIPT LOGBOOK ENTRY

KATAHDIN ANALYTICAL SERVICES

STOCK STANDARDS RECEIVED

GCMS LABORATORY
REVIEWED BY/DATE:

AMP0946

AccuStandard® 125 Market St. • New Haven, CT 06513 • USA Tel. 203-786-5290 • www.accustandard.com

APP-9-176-D-20X
Pentachlorophenol
2.0 mg/mL in CH2Cl2
Lot: B3010100
Exp. Jan 10, 2013.

1 mL

FOR LABORATORY USE ONLY

WARNING: This product contains a chemical(s) known to the State of California to cause cancer.

STORAGE Ambient

POISON

[redacted]

8/16/06
JLG

AMP0947

AccuStandard® 125 Market St. • New Haven, CT 06513 • USA Tel. 203-786-5290 • www.accustandard.com

APP-9-090-50X
4,6-Dinitro-o-cresol
5.0 mg/mL in MeOH
Lot: B1100296
Exp. Aug 16, 2012

1 mL

FOR LABORATORY USE ONLY

STORAGE Ambient

FLAMMABLE

[redacted]

AMP0948

AccuStandard® 125 Market St. • New Haven, CT 06513 • USA Tel. 203-786-5290 • www.accustandard.com

APP-9-145-50X
p-Nitrophenol
5.0 mg/mL in MeOH
Lot: B5050205
Exp. May 18, 2015

1 mL

FOR LABORATORY USE ONLY

STORAGE Ambient

POISON

[redacted]

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

FIGURE 3

EXAMPLE OF SVOA STANDARDS PREPARATION LOGBOOK ENTRY

GC/MS SVOA STANDARD PREP LOGBOOK

50863	8270 Stock (w/o MeOH)	3-15-06	7-7-06	JLM	AMP0844	8270 Mixes/Comp	300	2-22-07	4.2ml	150 ng/ml
					AMP0857	↓	300	3-15-07		
					AMP0911	APP IX #2	600	3-2-07		
					AMP0910	↓ 1	100	3-9-07		
					AMP0570	↓ 1	200	7-7-06		
					AMP0659	Organophosphorus	300	8-19-06		
					AMP0878	Benzoic Acid	↓	3-9-07		
					AMP0867	Hexachlorophene		2-22-07		
					AMP0806	Benzidine		3-9-07		
					AMP0836	3,3'-Dichlorobenzidine	↓	3-14-07		
					AMP0532	8270 Surr	150	3-9-07		
					50861	DEA	300	3-13-07		
					B43590	MeCl2	550	—		
50864	8270 level 1	3-15-06	7-7-06	JLM	50863	8270 Stock	70	7-7-06	1.05ml	10 ng/ml
					B43590	MeCl2	980			
50865	8270 level 2	3-15-06	7-7-06	JLM	50863	8270 Stock	150	7-7-06	0.90ml	25 ng/ml
					B43590	MeCl2	750			
50866	8270 level 3	3-15-06	7-7-06	JLM	50863	8270 Stock	600	7-7-06	1.8ml	50 ng/ml
					B43590	MeCl2	1200			
50867	8270 level 4	3-15-06	7-7-06	JLM	50864	8270 Stock	700	7-7-06	1.05ml	100 ng/ml
					B43590	MeCl2	350			

Reviewed by/Date:

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

Prepared By: Peter Lemay Date: 4/98

Approved By:

Group Supervisor: [signature] Date: 1/15/01

Operations Manager: John C. Burton Date: 1/15/01

QA Officer: Deborah J. Nadeau Date: 1.22.01

General Manager: [signature] Date: 1/16/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 8082	Format changes, added pollution prevention, minor changes to sections 7, 8 and Table 1	DN	1.22.01	1/22/01
02 8082	Revised sections 7.3.1, 7.4.5 and 7.6.1 to be compliant with South Carolina requirements.	DN	5.23.01	5.23.01
03 8082	Changed to practice of reporting higher value. Other minor changes to sections 7.5.2, 7.7.3 + to Table 2.	DN	5.21.02	5.21.02
04 8082	Revised SOP to indicate Turbochrom is being used as instrument control + data collection software. Included Target-related definitions. Changes to sections 7.7.3, 7.7.4 and 7.8.	MRC	08.20.04	08.20.04
05 8082	Changed 7.5.2 to reflect alternating CV Changed Table 2 Sect. 7.3.1 New Checklist added wording to sect. 8	LAD	020305	020305

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06 8082	Changed PCB 1260 to Aroclor 1260. Removed references to 3541. Updated table 2. Added instructions to shake extract before vialing	LAD	04/06	04/06
07	Added waste streams to sect. 1.0. Added ICV to definitions, sect. 5, sect. 7 and Table 1. Added wording regarding 2nd column confirmation criteria and flagging rules to sect. 7.7.4. Added CCV criteria to sect. 7.5.3 and Table 1. Added wording regarding MI to sect. 7.7.3	LAD	08/07	08/07
08	Added tissue, wipe and oil matrices. Added extraction method 3535. Added DDT analog interference, Std. information and analysis frequency criteria. Added HTs are a recommendation. Added note that 2 detectors must be used for dual column. Updated method references. Removed calibration and surrogate method mod. from Table 2. Added more info @ linear calib. Added extraction references.	LAD	02/09	02/09
09	Added Chemstation to definitions. Clarified that Surrogates are added to only the aroclor 1660 standards, not ALL standards.	LAD	05/09	05/09
10	Revised sections 7, 8, and 10 to reflect compliance with the DoD QSM version 4.1	LAD	08/09	08/09
11	Added Table 2 with DoD QSM Ver. 4.1 QC criteria. Minor changes to Table 1.	LAD	04/10	04/10

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

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TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

1.0 SCOPE AND APPLICATION

This SOP describes all aspects of the analysis of extracts of aqueous, solid, tissue, wipe and oil samples for PCBs by EPA Method 8082A as performed by Katahdin Analytical Services, Inc. including sample analysis, data review, standard preparation and instrument calibration.

It is applicable to the following compounds: Aroclor-1242, Aroclor-1254, Aroclor-1221, Aroclor-1232, Aroclor-1248, Aroclor-1260 and Aroclor-1016. Extracts are analyzed by Gas Chromatography-Electron Capture Detector (GC-ECD)

1.1 Definitions

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, laboratory reagent grade water is used as a blank matrix; for soil samples, muffled sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution that is different from the stock used to prepare standards.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

INDEPENDENT CALIBRATION VERIFICATION STANDARD (ICV): A solution prepared from a stock standard solution independent of the calibration mix that is used to verify the calibration.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample

extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

STOCK STANDARD SOLUTION: A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, Aroclor 1660 standard, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS): A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. KIMS utilizes these features through a database.

PE NELSON TURBOCHROM OR HP CHEMSTATION: data acquisition systems that are used to collect chromatographic data. The systems can also be used to archive raw data files.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

QUICKFORMS: A laboratory reporting software for Target and Target DB. The QuickForms report module for Target is preconfigured with generalized forms and US EPA CLP report forms and disk deliverables, which can be customized.

1.2 Responsibilities

1.2.1 This method is restricted to use by, or under the supervision of analysts experienced in the analysis of PCBs by method 8082. Each analyst must demonstrate and document their ability to generate acceptable results with this

method. Refer to Katahdin SOP QA-805, "Personnel Training & Documentation of Capability," current revision.

1.2.2 It is the responsibility of all Katahdin technical personnel involved in analysis by method 8082 to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

1.2.3 It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Health and Safety

1.3.1 Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

1.3.2 Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

1.4.1 Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Management Program for further details on pollution prevention techniques.

1.4.2 Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

1.4.3 Wastes generated during standards preparation are disposed of in the Mixed Flammable Waste (O). After the extracts have been analyzed, the autosampler vials and any expired standard vials or ampules are disposed of in the PCB Vial Waste (H).

2.0 SUMMARY OF METHOD

2.1 Method 8082 provides gas chromatographic conditions for the detection of PPB concentrations of certain PCBs. Prior to the use of this method, appropriate sample extraction techniques must be used. Both neat and diluted organic liquids (Method 3580, waste dilution) may be analyzed by direct injection. A 2 to 5 ul aliquot of sample is injected into a gas chromatograph (GC) using the direct injection technique, and compounds in the GC effluent are detected by an electron capture detector (ECD).

2.2 The sensitivity of Method 8082 usually depends on the concentration of interferences rather than on instrumental limitations. If interferences prevent detection of the analytes, Method 8082 may also be performed on samples that have undergone the following cleanups: Method 3660 - Sulfur Cleanup and Method 3665 - Sulfuric Acid Cleanup.

3.0 INTERFERENCES

Interferences by phthalate esters can pose a problem in PCB determinations when using the electron capture detector. Common flexible plastics contain various amounts of phthalates. Care has to be taken to avoid using any plastic materials during the extraction process. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.

Compounds from the sample matrix to which the detector will respond, such as single-component chlorinated pesticides including the DDT series.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph

4.1.1 GC Hewlett Packard 5890 series I or II connected to the Turbochrom or HP Chemstation data system, or equivalent.

4.1.2 Columns - Instruments are configured with a pre-column originating from the injection port, which is connected to a deactivated glass Y splitter that connects two different columns to two detectors. The most commonly used columns are: RTX-35 30M x 0.53 mm ID, RTX-5 30M x 0.53 MM ID, or RTX-1701 30M x 0.53 mm ID. Equivalent columns can be used.

4.1.3 Detectors: Electron capture detectors (ECD). Note: Two detectors must be employed when using dual columns.

4.2 Volumetric flasks, class A: sizes as appropriate with the ground-glass stoppers.

4.3 Syringes: various sizes for preparing standards and injecting samples on the instrument.

4.4 Vials: various sizes and types including crimp tops.

4.5 Balances: Analytical, 0.0001 g

4.6 Refrigerator for storage of extracts and standards.

5.0 REAGENTS

5.1 Solvents

5.1.1 Hexane: pesticide quality or equivalent for diluting samples and standards.

5.2 Standards

5.2.1 Stock standard solutions: Solutions purchased from suppliers like Restek or other acceptable retailers. Expiration dates are one year from date of opening vial or sooner if manufacturers date is less. Upon receipt, all standards are logged into the appropriate logbook with the date of receipt, expiration date, source, lot number, solvent and concentration of compounds. Standard solutions are stored at 4°C in polytetrafluoroethylene (PTFE)-sealed containers in the dark.

5.2.2 Calibration standards: Prepared through the dilution of the stock standards with hexane. Expiration date is 6 months or sooner. Information is documented in standards prep logbook. The concentrations of the working PCB calibration

standards are 0.05 ug/ml, 0.10 ug/ml, 0.25 ug/ml, 1.0 ug/ml, 2.5 ug/ml, and 10.0 ug/ml. The Aroclor 1660 standard also contain the surrogates Tetrachloro-m-xylene (TCX) and Decachlorobiphenyl (DCB) at the respective concentrations: 0.001 ug/ml, 0.002 ug/ml, 0.005 ug/ml, 0.020 ug/ml, 0.050 ug/ml, and 0.20 ug/ml.

5.2.3 Independent Calibration Verification standard (ICV): Prepared through the dilution of the stock standards with hexane. Expiration date is 6 months or sooner. Information is documented in standards prep logbook. The concentration of the ICV PCB standard is 1.0 ug/ml.

5.2.4 DDT Analog Standard: Standard containing the DDT series in hexane at 0.05 ug/ml.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Extracts must be stored under refrigeration and analyzed within 40 days of extraction.

Note: The holding time above is a recommendation. PCBs are very stable in a variety of matrices, and holding times under the conditions listed above may be as long as a year.

7.0 PROCEDURES

7.1 Extraction

Refer to the appropriate SOPs for the correct extraction procedure. In general, water samples are extracted using methods 3510, 3520 or 3535 while solid samples use methods 3540, 3545, or 3550. Tissue samples are extracted using method 3545. Wipes and oils are generally extracted using method 3580.

7.2 Instrument conditions

Refer to the instrument logbook for the current column and conditions.

Typical conditions are:

Makeup flow: 60 ml/min Ar/Methane or Nitrogen
Column flow: 6 ml/min
Injector Temp: 200
Detector Temp: 300
Oven Ramp: 160(0) - 5/min - 260(10)

Run time: 30 min
Injection size: 2 ul

7.3 Calibration

7.3.1 The GC system is calibrated using the external standard calibration procedure. Six-point calibration standards of Aroclor 1660 (Aroclor 1016 and Aroclor 1260), Aroclor 1242, Aroclor 1248 and Aroclor 1254 are prepared along with mid-point calibration standards of Aroclor 1221 and Aroclor 1232. If Aroclor 1221 or Aroclor 1232 are suspected, then a six-point curve of the respective Aroclor will be analyzed prior to the analysis and quantitation of the sample.

Each calibration standard is injected using the technique that is used to introduce the actual samples into the GC. Three to five characteristic peaks from each Aroclor are used to calibrate a curve. The Target system will calculate a peak height for all three to five peaks in each Aroclor. A separate calibration curve for each of the three to five peaks can be prepared in Target using the peak height against the concentration of the standard. A non-linear calibration applying a second order polynomial (quadratic fit) equation is used to prepare the curve. In order to be used for quantitative purposes, the Coefficient of Determination (r^2) must be greater than or equal to 0.990. The quadratic equation is:

$$y = ax^2 + bx + c$$

where: y = Instrument response
b = Slope of the line
x = Concentration of the calibration standard
c = the intercept

7.3.2 A non-linear calibration model may not be allowable for certain states, federal programs, or clients. South Carolina does not allow non-linear calibration work originating in their state. In these cases, a linear calibration model must be used. Each calibration standard is injected using the technique that is used to introduce the actual samples into the GC. Three to five characteristic peaks from each Aroclor are used to calibrate a curve. The Target system will calculate a peak height for all three to five peaks in each Aroclor. A separate calibration curve for each of the three to five peaks can be prepared in Target using the peak height against the concentration of the standard.

7.3.2.1 Linear calibration using the average calibration factor

The calibration factor (CF) is calculated using the following formula:

$$CF = A_s / C_s$$

Where: A_s = Peak area (or height) of the analyte or surrogate.
C_s = Concentration of the analyte or surrogate, in µg/L.

To evaluate the linearity of the initial calibration, calculate the mean CF, the standard deviation (SD), and the RSD.

If the RSD of the calibration factor is less than or equal to 20% over the calibration range, then linearity through the origin may be assumed, and the average calibration or response factor may be used to determine sample concentrations.

7.3.2.2 Linear calibration using a least squares regression

$$y = bx + c$$

where: y = Instrument response
b = Slope of the line
x = Concentration of the calibration standard
c = the intercept

The analyst should not force the line through the origin, but have the intercept calculated from the five data points. In addition, do not include the origin (0,0) as a sixth calibration point. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.995. The ICAL must be successful before any samples or other QC check samples can be analyzed.

7.3.3 The AR1660 calibration curve must be checked initially by analyzing a standard containing the same analytes as the curve but prepared from another source. If the response of the analytes from the independent source varies by more than ± 20%, a new independent source standard must be analyzed or a new calibration curve must be prepared and/or analyzed.

7.3.4 The working calibration curve must be verified prior to sample analysis and every 10 samples thereafter by injecting the mid-point calibration standard. If the response for any analyte varies from the expected response by more than ± 15%, a new calibration curve must be prepared for that analyte. The average result for 5 peak heights of the Aroclors is used for quantitation.

For clients or projects requiring DoD QSM 4.1, the response for any analyte must not vary from the expected response by more than ± 20%, or a new calibration curve must be prepared for that analyte. If the CCV fails the above criteria, reanalyze all samples since the last successful calibration verification. If reanalysis cannot be performed, data must be qualified and explained in the narrative. Additionally, apply a Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.

7.4 Retention time windows

7.4.1 Three injections are made of all the PCBs throughout the course of a 72 hour period.

7.4.2 A major peak from the envelope is chosen and a standard deviation is calculated using the three retention times for that peak.

7.4.3 Plus or minus three times the standard deviation of the retention times for each standard is used to define the retention time window; however, the experience of the analyst should weight heavily in the interpretation of chromatograms. The analyst should use the retention time window, but should primarily rely on pattern recognition.

7.4.4 Retention time windows are calculated for each standard on each GC column at method setup and after major maintenance, including whenever a new GC column is installed. The data is kept on file in the laboratory.

7.4.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply nominal windows. This is done in order to avoid any false negative hits because of the window being to narrow. The windows are: ± 0.07 for all target analytes. By utilizing these windows, a false positive hit may be initially indicated, but an experienced analyst could determine a false positive from scrutinizing the chromatograms. Please note that the use of nominal retention time windows may not be allowable for certain states, federal programs, or clients. South Carolina does not allow the use of nominal limits for compliance work originating in their state. In these cases, a window of ± 0.03 minutes must be used if the established retention time window is less than 0.03 minutes.

7.5 DDT Analog standard; This standard should be analyzed to determine if the commonly found DDT analogs (DDT, DDE, DDD) elute at the same retention times of any of the target PCBs. This standard should be analyzed in conjunction with the retention time window determination.

7.6 Gas chromatographic analysis

7.6.1 Shake samples and let them sit for one minute before vialing for analysis.

7.6.2 All instrument injections are performed using the direct injection technique with an autosampler set for 2-5 ul injection volumes.

7.6.3 Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration as listed in section 7.3 followed by sample extracts interspersed with mid-concentration calibration standards. Before any samples are analyzed the instrument must be calibrated by analyzing a six-point calibration or a 1.0ppm concentration standard (CV-calibration verification standard) for Aroclor 1660, Aroclor 1242, Aroclor 1248 and Aroclor 1254. If a CV is run, the calculated concentration must not exceed a difference of ± 15%. For clients or projects requiring DoD QSM 4.1, the response for any analyte must not vary from the expected response by more than ± 20%, or a new calibration curve must be prepared for that analyte. If the CCV fails the above criteria, reanalyze all samples since the last successful calibration verification. If reanalysis cannot be performed, data must be qualified and explained in the narrative. Additionally, apply a Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification. Each sample analysis must be bracketed with an acceptable initial calibration or an opening CV and an ending CV for each 12-hour shift. The closing CV for Aroclor 1660 is a 0.25ppm concentration standard. All other Aroclors at the closing of the run remain at 1.0ppm concentration. If a second window of samples is run immediately after the closing CVs, the concentration of Aroclor 1660 at the completion of this window would be 1.0ppm. The calibration standard must also be injected at intervals of not less than once every ten samples and at the end of the analysis sequence. If the CV fails, the instrument is checked for any obvious problems and maintenance is performed if deemed necessary. Another CV is analyzed or the instrument is recalibrated and then samples are injected. All samples that were injected after the standard exceeding the criterion must be reinjected to avoid errors in quantitation, if the initial analysis indicated the presence of the specific target analyte that exceeded the criterion.

7.6.3.1 However, if the standard analyzed <u>after</u> a group of samples exhibits a response for an analyte that is <u>above</u> the acceptance limit, i.e. >15%, and the analyte was not detected in the specific samples analyzed during the analytical shift, then the extracts for those samples do not need to be reanalyzed, as the CV standard has demonstrated that the analyte would have been detected were it present. In contrast, if an analyte above the QC limits <u>was</u> detected in a sample extract, then re-

injection is necessary to ensure accurate quantitation. If an analyte was not detected in the sample and the standard response is more than 15% below the initial calibration response, then re-injection is necessary to ensure that the detector response has not deteriorated to the point that the analyte would not have been detected even though it was present.

7.6.4 The center of the retention time window for each analyte and surrogate is established by using the absolute retention time for each analyte and surrogate from the daily opening calibration verification or initial calibration.

7.6.5 The identification of PCBs is based on agreement between the retention times of peaks in the sample chromatogram with the retention time windows established through the analysis of standards of the target analytes. An analyte is tentatively identified when a peak from a sample falls within the daily retention time window. Each tentative identification must be confirmed using a second GC column of dissimilar stationary phase or using another technique such as GC/MS. If the retention times of the peaks on both columns fall within the retention time windows on the respective columns, then the target analyte identification has been confirmed.

7.6.5.1 An additional criterion is applied for the identification and quantitation of PCBs. Identification is based on the characteristic fingerprint retention time and shape of the major peaks. Major peaks are defined as those peaks in the Aroclor standard that are at least 25% of the height of the largest Aroclor peak. The sample chromatogram is compared to the individual Aroclor standard chromatograms. Once the Aroclor pattern has been identified, a concentration is then calculated in Target.

7.6.5.2 Three to five Aroclor concentrations are calculated using the peak heights of the three to five characteristic peaks of the Aroclor. These three to five concentrations are then averaged to determine the concentration of that Aroclor.

7.6.6 When samples are analyzed from a source known to contain specific Aroclors, the results from a single-column analysis may be confirmed on the basis of a clearly recognizable Aroclor pattern.

7.6.7 If the response for an analyte exceeds the calibration range of the system, the sample must be diluted and reanalyzed.

7.6.8 If peak detection and identification are prevented due to interferences, the hexane extract may need to undergo a cleanup. The extract may be subjected to a sulfur cleanup (method 3660) and/or a sulfuric acid cleanup (method 3665).

Note: Samples routinely receive a sulfuric acid clean up. However, for samples from a known site with a clean matrix, a sulfuric acid clean up may not be performed. Whenever a sample receives a cleanup, the associated QC must also be subjected to the same cleanup(s) and reanalyzed.

7.6.9 When a GC system is determined to be out of control because either a CV cannot pass or a six point calibration does not meet the correlation coefficient criteria, instrument maintenance is likely necessary. Routine instrument maintenance may involve changing the septum, replacing the liner, clipping the pre-column, or replacing the column. This information is recorded in the instrument run log (Figure 1). When an instrument requires more severe maintenance like replacing the ECD or an electronic board, this information is written in the instrument maintenance logbook.

7.7 Calculations

7.7.1 The concentration of an analyte is calculated by using the calibrated curve that is prepared in Target. When an analyte is identified, Target displays a concentration after the file is processed through the appropriate calibration method. Aroclor quantitation is accomplished by the method described in section 7.5.4.1.1. However, if a sample contains more than one Aroclor, a peak common to both analytes must not be used to quantitate either compound.

7.7.2 The concentrations from the reports are then incorporated with the extraction data to arrive at a final concentration.

Water: Concentration (ug/L) = (C) (Vt)/ (Vs)

Soil/Sediment: Concentration (mg/kg) = (C) (Vt)/ (Ws) (D)

where,
C = concentration calculated by Target in ug/ml
Vt = Volume of total extract including any instrument dilutions
Vs = Volume of sample extracted
Ws = Weight of sample extracted
D = Decimal total solids

7.8 Data Review

7.8.1 Initial Data Review

The initial data review is accomplished by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed samples. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed and/or extracted. These criteria include:

- QC criteria for method blank, LCS, MS/MSD, and calibration – refer to section 8.0.
- Surrogate recovery
- Chromatography: cleanups, manual integration.
- Target compound detection: quantitation, confirmation, false positives.

The requirement of the GC laboratory is that this initial data review be completed no later than the end of the next workday. After the analyst has completed his or her initial data review, the information is then ready to be processed for reporting. Refer to section 7.8.

7.8.2 Surrogate recovery

All recoveries must meet the most recently laboratory established acceptance limits, which are listed on the GC Laboratory Surrogate Acceptance Limit sheet.

The sample is evaluated for recoveries of the two surrogates. If the recovery of one surrogate is within the acceptance limit, and the second is out, the data is narrated. If the surrogate recoveries are high for both and the sample contains less than the PQL for all target analytes, the data is narrated. If the surrogate recoveries are low and may be attributable to matrix interference or a matrix effect, the data is narrated. If the surrogate recoveries are low and the sample concentration is less than the PQL for all target analytes and there is no apparent matrix effect, reextract the sample.

For method blanks, if the recoveries of both surrogates are low or high, and the blank does not contain any target analytes above the PQL, and the recoveries of both surrogates in the sample(s) are acceptable, the data is narrated. If the recoveries in the blank are low and it does not contain any target analytes above the PQL, and the recoveries in the samples are acceptable but the

sample contains one or more target analytes above the PQL, the sample may be reextracted.

For laboratory control samples (LCS), if the only discrepancy in the extraction batch is with the LCS, and the analyte spike recoveries are acceptable, the data is narrated. If the recoveries of both the surrogates and the analyte spikes are low, the samples may need to be reextracted.
For DoD QSM 4.1, use QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. When the surrogate recoveries fall outside of the acceptance criteria, apply Q-flag to all associated analytes.

7.8.3 Chromatography

The chromatography should be examined for the presence of any non-target peaks, which can be used as an indication of whether or not matrix interference might be influencing surrogate recoveries. If the chromatogram indicates interferences, then a cleanup may be needed. See section 7.5.7.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.
Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), an "m" qualifier will automatically be printed on the quantitation report summary. The analyst will date and initial the "m" on the quanitation report summary and assign a code that indicates the reason for the manual integration. Refer to Katahdin SOP QA-812 "Manual Integration on GC/MS, GC, HPLC and IC Datasystems" for more information.

7.8.4 Target Compound Detection

GC analysis relies heavily on the experience of the analyst. Sample chromatograms must be evaluated focusing on scientific judgment, knowledge of the column behavior and matrix effects. The chromatogram from channel A is evaluated with that from channel B. If a target analyte is present on both channels and the concentration is within the calibration range, and the quantitation from both chromatograms agrees within ±40%, the analyte is considered present in the sample. In cases where the RPD is greater than 40% and the analyte is reported, the analyte must be J-flagged and narrated. The higher of the two concentrations is reported unless matrix

interference is causing erroneously high results. In this case report the lower result and narrate. In some cases a non-confirming analyte may be reported. In these cases the analyte must be Q-flagged and narrated...

In order to avoid reporting false positives, identified peaks on a chromatogram may need to be undetected electronically in Target. The possible scenarios are: If an analyte is present on one column but its concentration is below the PQL, if an analyte is present on one column but does not confirm on the other channel, if an analyte is present on both columns but the concentrations differ by more than 40%, or if an analyte is present but its retention time is ±0.04 minutes or more than the retention time of the analyte in the preceding CV. The GC Analyst must rely on technical experience in reviewing chromatograms in determining if a hit is an actual analyte or a false positive.

If reporting data that has an RPD that is >40%, the data must be flagged with a "J" indicating that the result is an estimated value. Sometimes interference on one column (i.e. sulfur) will prevent a target analyte from detection and it is present on the conformational column. In this scenario, the result would be reported from one column and need to be "Q" flagged to indicate that it was not confirmed on a second column.

7.8.5 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into QuickForms. Depending on the QC level requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the Department Manager for final review. A completed review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed

in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 For each analytical batch (up to 20 samples), a method blank, laboratory control sample (LCS), matrix spike and matrix spike duplicate are analyzed. They are carried through all stages of the sample preparation and analysis steps.

8.2 Spike concentrations: The LCS and the MS/MSD are spiked at the same concentration with Aroclor 1660. The spike concentrations are:

Compound	WATER ug/L	SOILS mg/kg
Aroclor 1660	5.0	0.17

The surrogate spike concentrations in the final extract are:

Compound	WATER ug/ml	SOILS ug/ml
Tetrachloro-m-xylene(TCX)	0.10	0.10
DCB	0.10	0.10

8.3 LCS and MS/MSD acceptance criteria and Corrective Action: All QC samples are calculated for percent recovery of the spiked analyte. The recoveries are compared to laboratory established acceptance limits. The LCS acceptance limits for PCBs are established for both water and soil matrices. The MS/MSD acceptance limits for PCBs use the respective matrix LCS acceptance limits. Separate limits for MS/MSD pairs are not calculated because of the varying matrices involved. In addition many of the MS/MSD data points cannot be used (i.e. recoveries not calculable due to a matrix effect).

If any spike compound in the laboratory control sample falls outside of the established recovery acceptance limit window, the QC sample is considered to be out of control and any sample that is associated should be evaluated with other QC elements to determine the corrective action. If the recovery is high and the associated samples do not contain the specific compound(s), the data can possibly be accepted with narration. In other cases, the associated samples must be extracted.
If a spike compound is outside of the acceptance limits in the matrix spike sample but is acceptable in the LCS, the data is considered acceptable. The cause of the failure is possibly attributable to matrix interference. However, if the compound fails in both the LCS and the MS/MSD, the result for that analyte is suspect and may not be reported for regulatory compliance purposes.

For DoD QSM 4.1, use QC acceptance criteria specified by DoD, if available. Otherwise use in-house control limits. In-house control limits must not be greater than $\pm$ 3 times the standard deviation of the mean LCS recovery. If the LCS fails the acceptance criteria, 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. If reanalysis cannot be performed, data must be qualified and explained in the narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.

For MS, when applying DoD QSM 4.1, apply J-flag to specific analyte(s) also in parent sample, if acceptance criteria not met. RPD must be $\leq$ 30% between MS and MSD.

8.4 Surrogate acceptance criteria and Corrective Action: Surrogate recoveries are calculated on all samples, blanks and spikes. The recoveries are compared to laboratory established acceptance limits.

When a sample has a surrogate that falls outside of the laboratory established acceptance limit window, the problem should be investigated. If the recovery looks like it is affected by the sample matrix, the sample may be reinjected to confirm matrix interference. When a sample has no detectable surrogate recovery, the sample should be reextracted.

For DoD QSM 4.1, use QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. When the surrogate recoveries fall outside of the acceptance criteria, apply Q-flag to all associated analytes.

8.5 CAR: Whenever data is not acceptable because of a failing LCS or surrogate recovery, a corrective action report (CAR) must be initiated as soon as possible to document resolution.

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined prior to sample analysis per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of Method 8082 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition. Final Update IV, dated February, 2007, Method 8082A.

Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

Katahdin Analytical Services, Inc., SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin Analytical Services, Inc., SOP CA-106, Standard Preparation, Documentation and Traceability, current revision.

Katahdin Analytical Services, Inc., SOP CA-500, Preparation of Soil/Sediment Samples by Sonication Using Method 3550 for Subsequent Pesticides/PCBs Analysis, current revision.

Katahdin Analytical Services, Inc., SOP CA-515, Preparation of Aqueous Samples for Pesticides/PCBs Analysis-Methods 3510 and 3520, current revision.

Katahdin Analytical Services, Inc., SOP CA-524, Preparation of Soil/Sediment Samples by Soxhlet Extraction Using Method 3540 for Subsequent Pesticides/PCBs Analysis, current revision.

Katahdin Analytical Services, Inc., SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

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Table 2 DoD QSM Version 4.1 QC Requirements
Table 2 Summary of Method Modifications
Figure 1 Instrument Run Log
Figure 2 Review Checklist
Figure 3 PQLs

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
6pt calibration of Aroclor 1660, 1242, 1248, 1254 and mid-point cal of other Aroclors	Initial cal prior to sample analysis	6 pt calibration – Coefficient of Determination (r^2) ≥ 0.990	(1) Repeat Initial calibration (2) If single pt cal Aroclor is identified in analysis of sample, 6-pt calibration run of identified compound with reanalysis of sample.
5pt calibration of Aroclor 1660, 1242, 1248, 1254 and mid-point cal of other Aroclors	Initial cal prior to sample analysis	5 pt calibration – (r) ≥ 0.990	(1) Repeat Initial calibration (2) If single pt cal Aroclor is identified in analysis of sample, 6-pt calibration run of identified compound with reanalysis of sample.
Independent Calibration Verification	Immediately following calibration	± 20 % D	(1) Reanalyze standard (2) Reprep standard (3) Reprep standard from fresh stock.
CCV	After every 10 samples; If calibration curve previously analyzed, analyze daily before samples.	± 20 % D	(1) Evaluate the samples: If the %D >+15% and sample results are <PQL, narrate. (2) If %D >±15% only on one channel, narrate. If %D >±15% for closing CV, and is likely a result of matrix interference, narrate. (3) Otherwise, reanalyze all samples back to last acceptable CV.
Method blank	One per prep batch	No analyte detected >PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are <PQL or > 10X the blank concentration. (3) Otherwise, reprep a blank and the remaining samples.
LCS	One per prep batch of twenty or fewer samples	± 15% D	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. (2) If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. (3) If the surrogate recoveries in the LCS are also low but are acceptable in the blank and samples, narrate. (4) If the LCS recovery is high but the sample results are <PQL, narrate. (5) Otherwise, reprep a blank, QC and the remaining samples.
Matrix Spike\ Matrix Spike Duplicate	One for every set of 20 samples	Same as for LCS	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable reprep samples and QC.

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 1 (cont.)

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Sample Duplicate	One sample duplicate per ten samples if requested	RPD $\leq$20	(1) If lab QC in criteria and matrix interference suspected, flag data or narrate (2) Otherwise, reanalyze
Demonstration of analyst proficiency – 4 replicates	Once per analyst initially and annually thereafter	P&A meet method criteria	(1) Repeat P&A study
MDL study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 2

DOD QSM VERSION 4.1 QC REQUIREMENTS

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.	Not Applicable (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	Refer to current revision of SOP QA-806				
LOQ establishment and verification	Refer to current revision of SOP QA-806				
Retention time (RT) window width calculated for each analyte and surrogate	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from a 72-hour study.	NA.	NA.	
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	6 point calibration of Aroclors 1660, 1242, 1248, and 1254 - One of the options below: Option 1: RSD for each analyte ≤ 20%; Option 2: linear least squares regression: r ≥ 0.995; Option 3: non-linear regression: coefficient of determination (COD) r2 ≥ 0.99 (6 points shall be used for second order). Mid point calibration of Aroclors 1221 and 1232; if targets are detected, 6-point calibration is performed.	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin. Quantitation for multicomponent analytes such as chlordane, toxaphene, and Aroclors must be performed using a 5-point calibration. Results may not be quantitated using a single point.
Retention time window position establishment for each analyte and surrogate	Once per ICAL and at the beginning of the analytical shift.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 2 (cont)

DOD QSM VERSION 4.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Second source calibration verification (ICV)	Immediately following ICAL.	All project analytes within established retention time windows. GC methods: All project analytes within ± 20% of expected value from the ICAL.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
calibration verification (CCV)	Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All project analytes within established retention time windows. GC methods: All project analytes within ± 20% of expected value from the ICAL.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	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. Retention time windows are updated per the method.
Method blank	One per preparatory batch.	No analytes detected > ½ RL (> RL for common lab contaminants) and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result. Contact Client if samples cannot be reprepped within hold time.	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.
Laboratory control sample (LCS) containing all analytes to be reported, including surrogates	One per preparatory batch.	The laboratory shall use laboratory control limits (CLs) or use DoD-generated LCS-CLs, if available depending on project requirements. In-house CLs may not be greater than ± 3 times the standard deviation of the mean LCS recovery. A number of analytes may fall outside the CL but within marginal exceedance limit depending on the total number of analytes in the LCS.	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. Refer to Table G-1 for number of marginal exceedences allowed. Contact Client if samples cannot be reprepped within hold time.	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.

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 2 (cont.)

DOD QSM VERSION 4.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike (MS)	One per preparatory batch per matrix if sufficient sample is available.	For matrix evaluation, use LCS acceptance criteria.	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.
Matrix Spike duplicate (MSD)	One per preparatory batch per matrix if sufficient sample is available.	MSD: For matrix evaluation, use LCS acceptance criteria. MS/MSD: RPD ≤ 30%.	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.
Surrogate spike	All field and QC samples.	The laboratory shall use laboratory control limits (CLs) or use DoD-generated LCS-CLs, if available depending on project requirements.	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Contact Client if samples cannot be reprepped within hold time.	Apply Q-flag to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Confirmation of positive results (second column or second detector)	All positive results must be confirmed (with the exception of Method 8015).	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column RPD ≤ 40%.	NA.	Apply J-flag if RPD > 40%. Discuss in the case narrative.	Use project-specific reporting requirements if available; otherwise, use method reporting requirements; otherwise, report the result from the primary column (see Box D-16).
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-329-11	METHOD 8082, current revision
Procedures	7.4.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply nominal windows. This is done in order to avoid any false negative hits because of the window being to narrow. The windows are: ± 0.07 for all target analytes. By utilizing these windows, a false positive hit may be initially indicated, but an experienced analyst could determine a false positive from scrutinizing the chromatograms. Please note that the use of nominal retention time windows may not be allowable for certain states, federal programs, or clients. South Carolina does not allow the use of nominal limits for compliance work originating in their state. In these cases, a window of ± 0.03 minutes must be used if the established retention time window is less than 0.03 minutes.	9.3 refers to method 8000B section 7.6.3: If the standard deviation of the retention times for a target compound is 0.000 (i.e., no difference between the absolute retention times), then the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes. (Recording retention times to three decimal places rather than only two should minimize the instances in which the standard deviation is calculated as 0.000).
Apparatus/Materials		
Reagents		
Sample Preservation and handling		
QC – Spikes		
QC – LCS		
QC – Accuracy/ Precision		
QC - MDL	PQL Practical Quantitation Level – three to ten times the MDL.	EQL Estimated Quantitation Level – five to ten times the MDL

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

FIGURE 1

EXAMPLE OF INSTRUMENT RUN LOG

Katahdin Analytical Services, Inc.
GC Laboratory Instrument Runlog
Instrument: GC06
Amount Injected 2 µl

Method: SW846 8082 or EPA 608
Reviewed by/ Date:

Date	Init.	Result File	Sample ID	Y/N	Method	Column	Comments
7.31.07	SJC	6AG3141152	WG41666-3 3510	Y	PCBA1B074A	298/299	
		153	Hexane	N			
		154	AR1660 0.25	Y			↑DCB ↑1260 both P4274
		155	AR1242 1.0	Y			P4274
		156	AR1248				P4275
		157	AR1254				P4276
		158	SA3963-1 3540				
		159	SA3987-1				
		160	-2				
		161	-3				
		162	-4				
		163	SA3978-1				
		164	-2				
		165	-3				
8.1.07		166	-4				
		167	-5	N			needs SC.
		168	Hexane				
		169	AR1660 1.0	Y			P4273
		170	AR1242				P4274
		171	AR1248				P4275
		172	AR1254				P4276
		173	SA3978-6 3540	N			needs SC
		174	SA3958-4 3510	Y			
		175	-6				
		176	WG41667-1				
		177	-2				
		178	-3				
		179	-4				
		180	~~SA3958-5~~				
		181	SA3958-1				
		182	-2				
		183	Hexane	N			

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

FIGURE 2

DATA REVIEW CHECKLIST

PRIMARY REVIEW CHECKLIST

Verbal Due Date _____ (☐ **Verbals turned in**). **DueDate**_____

Client:		Primary	Secondary
Method:		Date:	Date:
SDG No:	Level:	Initials:	Initials:
KAS No:			Approved : ☐ Yes

DODQSM 3.0 ☐ **DODQSM 4.1** ☐ **WITH LAB. LIMITS** ☐
QUAPP ☐ **LAB** ☐ (*REPORT* ***ND's*** *to* ***MDL*** ☐)

List all curves that are scanned. _____ _____

_____ _____ _____ _____

Narrate which QC limits were used for (Surr., LCS,s MS/MSDs.) _____

All needed forms are present . _____

Correct Work Order Number or SDG name (all forms). _____

Correct project name and spelling (all forms). (Truncated ☐). _____

Correct file numbers (all forms). _____

Analysis Date Correct. _____

Extraction Method & Analysis Method Correct. _____

Product list compared to ROAs (compounds & PQLs). _____

Chromatogram reviewed for unlabeled peaks (check product list). _____

Flagging of all ROAs correct (Florida Flagging ☐). _____

All tunes included (level IV) . _____

All log book pages included (Soil weights,TCLP & SPLP). _____

Verify DOD QSM criteria. _____

Narrate any method deviations. (Blanks, LCS,s etc.) _____

Sign & Date Manual integration (**Narrate as needed**). _____

Sample I.D's Truncated (**NARRATED**). YES ☐ Please list KAS # below :

_____

QA-044 – Revision 1 – 04/05/2010

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

FIGURE 3

PQLs FOR METHOD 8082

ANALYTE	Practical Quantitation Level (PQL) (ug/L)	Practical Quantitation Level (PQL) (ug/kg)
PCB-1016	0.50	17
PCB-1221	0.50	17
PCB-1232	0.50	17
PCB-1242	0.50	17
PCB-1248	0.50	17
PCB-1254	0.50	17
PCB-1260	0.50	17

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-500
Revision History
Cover Page
Page 1

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS

Prepared By: Mike Thomas Date: 7/96

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: [illegible] Date: 10/25/00

QA Officer: Deborah J. Nadeau Date: 10·23·00

General Manager: Duncan F. Hughes Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	10·23·00	10/23/00
02	removed references to medium level extraction. New logbook figures minor changes through out	LAD	020305 ~~DT~~ LAD 020305	020305
03	updated compound list changes in wording to clarify updated logbook	LAD	04/06	04/06
04	Added definitions, added waste information, added LCSD, updated solvent exchange, updated Table 1, replaced fig. 2, added PCB cleanup to Sect. 2	LAD	09/07	09/07
05	Updated LB example. Added temp. of nitrogen water bath, lot numbers of filter paper, lot #'s of acids need to be recorded in LB. Change "N-Lo" waste to "K" waste.	LAD	07/08	07/08

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added requirement to add spike before Na_2SO_4. Changed N_2 waterbath temperature from < 37°C to < 30°C. Removed respirator references. Added KAEHS Manual, Added CA-108 reference for subsampling	LAD	02/09	02/09
07	Removed targeting sample weights. Minor changes to section 7. Updated logbook example.	LAD	08/10	08/10

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-500-07**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS.**

Recipient: _____Date:_____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-500-07**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS.**

Recipient: _____Date:_____

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures and requirements for the preparation of solid samples for pesticides/PCBs analysis in accordance with SW-846 Method 3550, current revision..

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in the extraction of sediment/soil samples for pesticides/PCBs analysis. Each analyst must demonstrate the ability to generate acceptable results with this method.

It is the responsibility of all Katahdin personnel involved in the preparation of solid samples for pesticides/PCBs analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also

be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for the data.

It is the responsibility of the Department Manager to oversee that the members of his/her group follow this SOP, that their work is properly documented, and to indicate periodic review of the associated logbooks.

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials, and appropriate segregation of hazardous wastes. Everyone involved with the procedure must be familiar with the material safety data sheets for all the materials used in this procedure. Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the “D” waste stream satellite accumulation area nearest

the point of generation. Acetone and hexane are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples, used glass wool, and sodium sulfate waste should be disposed of in the soil with organics "I" waste stream satellite accumulation area nearest the point of generation. Acid waste generated during the cleanup of PCB samples should be disposed of in the "K" satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

Pesticides/PCBs are extracted from solid samples by sonication with a methylene chloride/acetone solution (1:1 by volume) following EPA Method 3550, current revision. The resulting extract is dried, concentrated, and solvent exchanged to hexane for analysis by GC. Sulfuric acid cleanup is performed on extracts for 8082 PCB analysis.

This SOP applies to low level extraction of pesticide/PCB pollutants from solid sample matrices.

3.0 INTERFERENCES

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Whenever possible, plastic items in this lab, must be replaced with metal, teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

4.0 APPARATUS AND MATERIALS

Prior to use, all glassware must be rinsed three times with methylene chloride.

4.1 Beakers - 400 mL

4.2 Kuderna-Danish (KD) apparatus - Concentrator tube - 10 mL
Evaporative flask - 500 mL
Snyder column - 3-ball macro

4.3 Powder funnels, 100 mm diameter, 35 mm stem

4.4 Vacuum filtration flask - 500 mL Erlenmeyer

4.5 Buchner funnel, porcelain, Coors® with 85 mm plate diameter (or equivalent)

4.6 Sonic disruptor – Misonix XL2015 (or equivalent), equipped with dual titanium horn extenders for extracting two samples at a time.

4.7 Spatula - stainless steel

4.8 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

4.9 Boiling chips - 12 mesh, silicon carbide (or equivalent)

4.10 Water bath - eight position concentric ring bath or equivalent, equipped with a calibrated thermometer

4.11 Filter paper - 7.0 cm, Whatman, #4, or equivalent

4.12 Syringe - gas tight, 1.0 mL, solvent rinsed between each use

4.13 Balance – top-loading, capable of weighing to 0.01 g

4.14 Nitrogen evaporation apparatus

5.0 REAGENTS

5.1 Sodium sulfate - (ACS reagent grade) powdered, anhydrous, certified by the manufacturer/vendor as purified by heating to 400 °C prior to receipt by the laboratory. Solvent rinse immediately before use by rinsing three times with pesticide grade methylene chloride.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

5.2 Sodium sulfate - (ACS reagent grade) granular, anhydrous, purified as described in section 5.1.

5.3 Methylene chloride - (pesticide grade or equivalent) purchased by lot, evaluated prior to use by concentration of 300mLs to 1.0 mL followed by GC/MS analysis.

5.4 Acetone and hexane - pesticide grade or equivalent

5.5 Organic-free sand, purified by baking at 400 °C for four hours. Method blanks serve as checks on the baked sand.

5.6 Surrogate spiking solution - Prepare a solution of decachlorbiphenyl (DCB) and tetrachloro-meta-xylene (TCMX) at a concentration of 1 ug/mL each in acetone. Store the solution at –10 to -20 °C (±2 °C) in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

5.7 Pesticide Matrix spike/Lab control sample spiking solution - Prepare a spiking solution in pesticide grade methanol that contains all target analytes listed below:

Analyte	ug/mL
4,4'-DDD	0.5
4,4'-DDE	0.5
4,4'-DDT	0.5
Aldrin	0.5
alpha-BHC	0.5
beta-BHC	0.5
delta-BHC	0.5
Dieldrin	0.5
Endosulfan I	0.5
Endosulfan II	0.5
Endosulfan Sulfate	0.5
Endrin	0.5
Endrin Aldehyde	0.5
Endrin Ketone	0.5
gamma-BHC (Lindane)	0.5
Heptachlor	0.5
Heptachlor Epoxide	0.5
Methoxychlor	0.5
alpha-Chlordane	0.5
gamma-Chlordane	0.5

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Store the solution at –10 to -20 °C (±2 °C) in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

5.8 PCB Matrix Spike/Lab Control Sample Spiking Solution – Prepare spiking solution in pesticide grade acetone that contains PCB's Arochlor 1016 and Arochlor 1260 (1660), both at 5.0 ug/mL.

Store the solution at –10 to -20 °C (±2 °C) in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Soil samples are collected in glass soil jars and stored at 4°C (±2°C) until time of extraction.

Holding time for extraction of sediment/soil samples for Method 3550 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

The following information must be recorded in the extraction logbook.

- Extraction method
- Surrogate and spike IDs
- Lot numbers of all solvents, acids and bases, sodium sulfate, filter paper
- Nitrogen evaporation water bath temperature
- Sample pH (if applicable)
- Extraction and Concentration dates
- Extraction and Concentration analyst
- Sample ID or QC sample ID
- Initial and final volumes or weight
- Surrogate and spike amounts
- Any sample cleanup preformed
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)

- Prep batch start time and end time
- CLLE or Soxhlett start time and end time (if applicable)
- Lot numbers of the vials the concentrated extracts are stored in.

LOW LEVEL EXTRACTION OF SOIL/SEDIMENT FOR PESTICIDES/PCBs

The low level extraction procedure is designed for the preparation of soil/sediment samples that may contain analytes at levels lower than 20,000 ug/kg. The procedure involves extraction of pesticides and PCBs from an initial sample weight of approximately 30.0 g using an ultrasonic cell disruptor.

Many solid samples may need to be cleaned up to reduce matrix interferences. The cleanup procedure employed will be dependent upon the nature of the interferences and the target compounds to be analyzed, and options may include acid wash, sulfur cleanup, florisil cleanup, or gel permeation chromatography (GPC). The Department Manager should be consulted to determine if a particular sample should be subjected to further cleanup procedures; the decision should consider sample history, sample appearance, and project/client needs. All extracts or extract splits for subsequent 8082 PCB analysis will, at a minimum, undergo acid cleanup. (Refer to SOP CA-525, current revision)

7.1 Do not decant any water on the sediment sample. Mix with a stainless steel spatula to ensure homogeneity of the sample. If the sample container is full to the extent that stirring the sample is impractical, try to remove the “best representative” aliquot from the jar based on color, particle size, moisture, etc. Remove any foreign objects such as sticks, leaves, or rocks, and note actions taken in the appropriate extraction logbook. **Please refer to the current revision of Katahdin Analytical Services SOP CA-108, “Basic Laboratory Technique“, for more detailed guidance on subsampling to ensure reproducibility.**

7.2 Weigh out approximately 30.0 g plus (> 30 g) portion of sample into a labeled 400 mL beaker. Record sample weight to nearest 0.01 g in appropriate extraction logbook. Refer to sections 7.6 and 7.7 for spike and surrogate addition instructions. Add between 30 g and 60 g of powdered sodium sulfate, as required, to produce a “free-flowing” mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula. Keep the spatula in the sample beaker and <u>cover</u> the beaker with aluminum foil.

7.3 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare method blank, weigh out approximately 30.0 g portion of purified sand in a labeled 400 mL beaker. Refer to sections 7.6 and 7.7 for spike and surrogate addition instructions. Add 60 g sodium sulfate and mix well. (Although a “free-flowing” mixture can be achieved with less than 60 g sodium

sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.)

7.4 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out approximately 30.0 g of purified sand into a labeled 400 mL beaker. Refer to sections 7.6 and 7.7 for spike and surrogate addition instructions. Add 30 g sodium sulfate and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB LCS's must be prepared (refer to sections 5.7 and 5.8). If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.5 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out two approximately 30.0 g plus (>30 g) portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.01 g in appropriate extraction logbook. Refer to sections 7.6 and 7.7 for spike and surrogate addition instructions. Add 30 - 60 g sodium sulfate to each to produce a free-flowing mixture, and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB MS/MSD pairs must be prepared (refer to sections 5.7 and 5.8).

7.6 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL pesticide/PCBs surrogate spiking solution using a 1.0 mL gas tight syringe. The surrogate spike should be added **prior** to the addition of the sodium sulfate. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to each use.

7.7 To LCS/LCSD and MS/MSD pairs add 1.0 mL of pesticide or PCB matrix spike/LCS spiking solution using a 1.0 mL gas tight syringe. The LCS/MS spike should be added **prior** to the addition of the sodium sulfate. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to spiking a different solution and when spiking is completed.

7.8 To assure optimum operation and maximum energy output, the sonicators <u>must</u> be tuned daily prior to extracting samples. The following tuning procedure must be performed with the sonicator probes vibrating in air.

7.8.1 Turn OUTPUT CONTROL knob counter-clockwise to zero, and turn Pulsar Duty Cycle to off (or continuous mode).

7.8.2 Press POWER SWITCH to ON (up) position. Engage the Timer Switch (W-375)

7.8.3 Press and hold down the TUNE switch.

7.8.4 Turn the Output Control Knob towards setting 10. Note the position of the needle on the % output power meter. **DO NOT exceed 70%. If you reach 70% - STOP!!** Rotate the Tuning Knob clockwise or counter-clockwise until a minimum (not maximum) reading (usually less than 20%) is obtained.

7.8.5 Turn the Output Control Knob towards setting 10. Again, note the position of the needle and do not exceed 70%. Rotate the Tuning Knob until you obtain a minimum reading of 20% or below.

7.8.6 Release the TUNE switch. **CAUTION: Do NOT touch probe. The probe is still active.**

7.8.7 Turn OUTPUT CONTROL KNOB counter-clockwise to zero (or disengage timer).

7.8.8 Confirm that the sonicators were tuned by recording the date and/or percent in the extractions logbook.

Note: If the unit will not be used immediately, please turn the power switch to off.

7.9 Prior to extracting any samples, ensure that the sonicator probes are decontaminated by rinsing with a methylene chloride wash bottle. Collect the waste in a waste beaker. It may sometimes be necessary to wipe the upper part of each probe with a methylene chloride dampened KimWipe. Repeat this decontamination step between each sample on each probe.

7.10 To the mixed and spiked blank and LCS, add 100 mL of the 1:1 methylene chloride/acetone solution and proceed with steps 7.11 through 7.14.

7.11 It may be necessary at this time to stir the sample/sodium sulfate mixture with the spatula to loosen up the mixture prior to extracting. Position beaker in the ultrasonic cell disruptor so that the bottom surface of the tip of the 3/4 inch disruptor horn is about halfway below the surface of the solvent and above the sediment layer.

7.12 Sonicate for 3 minutes with the output control knob set at 10, and mode switch on “pulsed” and % duty cycle knob set at 50%. While the mixture is sonicating, one should be able to see all, or most of the material, moving in the beaker under the influence of the energized probes. If not, stir the mixture again.

7.13 Prepare a filter flask fitted with a Buchner funnel. The Buchner funnel should contain a 7.0 cm Whatman #4 filter; prerinse flask, funnel and filter with methylene chloride and discard rinsings into solvent waste container. Decant extract into the filter flask through Buchner funnel. A vacuum pump may be used to facilitate filtration or the extract may be gravity filtered.

Note: The lot number of the filter paper must be recorded in the extraction logbook.

7.14 Repeat the extraction two additional times using 100 mL portions of 1:1 methylene chloride:acetone. Before each extraction, make certain that the sodium sulfate is free-flowing and not a consolidated mass. As required, break up large lumps with the clean spatula. Decant the extraction solvent into the Buchner funnel after each sonication. On the final sonication, pour the entire sample contents into the Buchner funnel and rinse thoroughly with methylene chloride to complete the quantitative transfer of the extract.

CONCENTRATION OF LOW LEVEL EXTRACTS

7.15 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add two boiling chips to the K-D. Insert 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride. Place the assembled K-D's under the funnels.

Note: The lot number of the filter paper must be recorded in the extraction logbook.

7.16 If samples are to be GPC'd, refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures.

7.17 For a solvent exchange, add approximately 50 mL hexane to funnel and let drain through. Since methylene chloride has a lower boiling point than hexane, this will result in a final extract in hexane only.

Note: For Pesticide / PCB samples originating from South Carolina (see worknotes) do not add the hexane at this step. Solvent exchange will be during the nitrogen blow down procedure.

7.18 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through

the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mLs of methylene chloride and allow to drain.

7.19 If samples are not to be GPC'd follow Steps 7.19 through 7.23 to concentrate extracts to final volume of 10.0 mLs. Otherwise proceed to GPC cleanup procedure as described in the current revision of Katahdin SOP CA-513.

7.20 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.21 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.22 Reduce the extract in the concentrator tube to approximately 10 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than 35° C. Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of hexane (methylene chloride for samples not yet solvent exchanged). The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Note any problems or extract losses, if they occur, in the extractions logbook.

Note: The temperature of the water in the nitrogen evaporation water bath must be recorded in the logbook.

7.23 For samples that still need to be solvent exchanged, reduce the methylene chloride extract to ~ 1 mL. Add 10 mL of hexane to the concentrator tube and reduce to ~ 1 mL again on the N-evap. Adjust final extract to 10 mL by rinsing sides of tube with hexane and transferring rinsings to vial.

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7.24 Complete quantitative transfer of the extract to a vial by using hexane. Adjust the volume of the hexane extract to 10 mL in either a 12 or 16 mL vial using the appropriate "reference vial" for volume comparison.

7.25 Transfer the sample label from the concentrator tubes to the vials. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extractions logbook the box number and "tray location" of the individual extract vials.

7.26 All sample extracts for 8082 PCB analysis must undergo a sulfuric acid wash (cleanup) prior to analysis. All sample extracts for 8081 pesticide analysis do not undergo further cleanup unless requested by the client. All sample extracts for combined 8081/8082 analyses must be split. One portion must be acid cleaned for 8082 analysis. The associated method blank must be split and acid-cleaned in the same fashion. Prior to splitting, contents of vial must be shaken well. PCB LCSs and matrix spikes are acid cleaned also. Pesticide LCSs and matrix spikes are not subjected to further cleanup. Please refer to Katahdin SOP CA-525 (current revision), Extract Cleanup Using Sulfuric Acid, for further instructions.

Note: The lot number of the acid used in PCB cleanup must be recorded in the extraction logbook.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

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Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 A method blank must be extracted for each and every item listed below:

- Each extraction method
- Every extraction batch of twenty or fewer samples
- Each analysis (pesticides and/or PCBs)

8.2 A laboratory control sample (LCS) is required for each and every item listed below:

- Each extraction method
- Every extraction batch of twenty or fewer samples
- Each analysis (pesticides and/or PCBs)

Refer to the current revision of the applicable Katahdin SOP for analysis of Pesticides and PCBs for quality control acceptance criteria.

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOP.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Method 3550C, USEPA SW-846, Third Edition, Final Update IV, February 2007.

Table 1 Summary of Method Modifications
Figure 1 Example of Pest./PCB Soil Sample Prep Logbook Page

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

TABLE 1

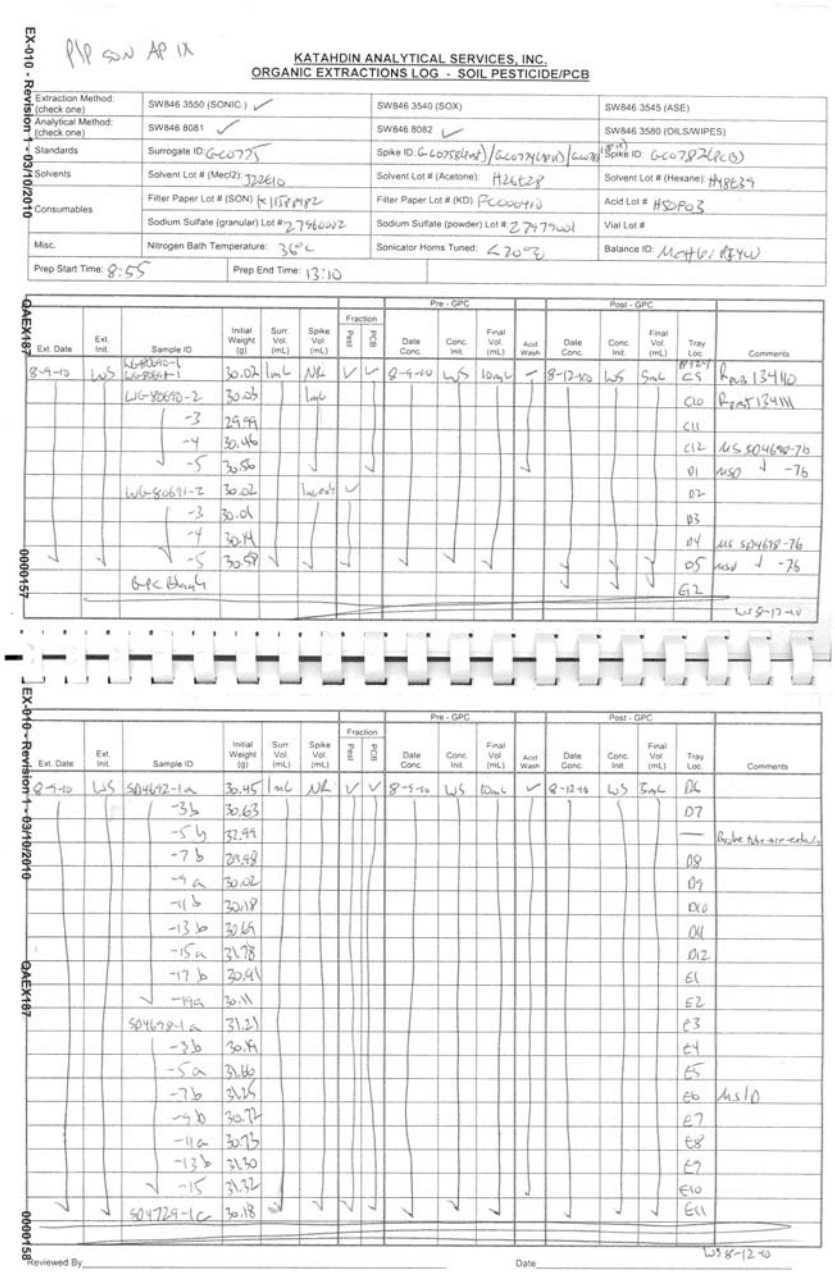
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-500-07	METHOD 3550, current revision
Apparatus/Materials	1. short stem funnels	1. drying columns
Reagents		
Sample preservation/ handling		
Procedures	1. extract dried using Na_2SO_4 in short stem funnels 2. place sonicator horns ½ way between the surface of the solvent and the sediment layer 3. no apparatus height specification for concentration on water bath 4. water bath at 75-85 deg C 5. sample removed from water bath when volume reaches ~6 mL 6. Solvent exchange to hexane is performed using K-D apparatus with addition of approximately 50 mL hexane at the start of concentration process	1. extract dried using Na_2SO_4 in drying columns 2. place sonicator horns ½ inch below the solvent surface but above sediment layer 3. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4. water bath at 80-90 deg C 5. sample removed from water bath when volume reaches 1-2 mL 6. Solvent exchange to hexane is performed using K-D apparatus with addition of approximately 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes	Refer to analytical SOP	
QC - LCS		
QC - Accuracy/Precision		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE



EX-010 - Revision 1 - 03/10/2010

PIP SON AP IX

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - SOIL PESTICIDE/PCB

Extraction Method: (check one)	SW846 3550 (SONIC.) ✓	SW846 3540 (SOX)	SW846 3545 (ASE)
Analytical Method: (check one)	SW846 8081 ✓	SW846 8082 ✓	SW846 3580 (OILS/WIPES)
Standards	Surrogate ID: GC0775	Spike ID: GC0758(pest)/GC0761(pcb)/GC0787	(8081) Spike ID: GC0782(PCB)
Solvents	Solvent Lot # (MeCl2): J22610	Solvent Lot # (Acetone): H26E28	Solvent Lot # (Hexane): H48E39
Consumables	Filter Paper Lot # (SON) K1558982	Filter Paper Lot # (KD) PC000410	Acid Lot # H5P03
	Sodium Sulfate (granular) Lot # 27960002	Sodium Sulfate (powder) Lot # 27979001	Vial Lot #
Misc.	Nitrogen Bath Temperature: 36°C	Sonicator Horns Tuned: 6 20°C	Balance ID: MEHU/RFYU
Prep Start Time: 8:55	Prep End Time: 13:10		

QAEX187 — 00001157

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Fraction Pest	Fraction PCB	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Acid Wash	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	Tray Loc.	Comments
8-9-10	WS	WG-80690-1 WG-80691-1	30.02	1mL	NA	✓	✓	8-9-10	WS	10mL	✓	8-12-10	WS	5mL	C9	Rest 13410
		WG-80690-2	30.03		1mL										C10	Rest 13411
		-3	29.99												C11	
		-4	30.46												C12	MS 504698-7b
		-5	30.50				✓				✓				D1	MSD ↓ -7b
		WG-80691-2	30.02		1mL ✓	✓									D2	
		-3	30.01												D3	
		-4	30.14												D4	MS 504698-7b
↓	↓	-5	30.59	✓	✓	✓		✓	✓	✓		↓	↓	↓	D5	MSD ↓ -7b
		GPC Blank													E12	

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QAEX187 — 00001158

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Fraction Pest	Fraction PCB	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Acid Wash	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	Tray Loc.	Comments
8-9-10	WS	504692-1a	30.45	1mL	NA	✓	✓	8-9-10	WS	10mL	✓	8-12-10	WS	5mL	D6	
		-3b	30.63												D7	
		-5b	32.99												—	Probe tube are exhausted
		-7b	29.98												D8	
		-9a	30.02												D9	
		-11b	30.18												D10	
		-13b	30.69												D11	
		-15a	31.78												D12	
		-17b	30.94												E1	
		-19a	30.11												E2	
		504698-1a	31.21												E3	
		-3b	30.19												E4	
		-5a	31.66												E5	
		-7b	31.25												E6	MS 1D
		-9b	30.72												E7	
		-11a	30.73												E8	
		-13b	31.30												E9	
		-15	31.32												E10	
↓	↓	504729-1c	30.18	✓	✓	✓	✓	↓	↓	↓		↓	↓	↓	E11	

WS 8-12-10

Reviewed By_____ Date_____

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

Prepared By: Michael Thomas Date: 07-24-00

Approved By:

Department Manager: [signature] Date: 6-23-06

Operations Manager: Deborah J. Radeau Date: 6-23-06

QA Officer: Susie Dimond Date: 6-23-06

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
03	Changes to sect. 5.5 & Figures 3 & 4 to reflect current spike solutions and concentrations. Replaced cover page. Original cover page filed with SOP CA502-02	LAD	04/06	04/06
04	Added definitions, added waste information added LCSD, added SIM LCS/D, MS/D, updated Table 1, added use of narrow range pH paper. Minor changes throughout to reflect current practice.	LAD	09/07	09/07
05	Removed MS/MSD 14 day requirement. Changed CLLE extraction time to 18 → 24 hours. Added information on determining initial sample volume. Added extracted sample disposal. Removed all references to method 625.	LAD	09/08	09/08
06	Added to check pH after B/N CLLE extraction to ensure pH ≥11. If not add more NaOH and continue extracting. Added information for initial volume determination. Added reference to CA-108. Updated logbook example. Added if extract goes dry - re extract.	LAD	10/09	10/09

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date

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TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-502-06**, titled **PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**.

Recipient: _____Date:_____

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STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-502-06**, titled **PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**.

Recipient: _____Date:_____

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe procedures utilized by Katahdin Analytical personnel in the preparation of all non-CLP aqueous samples for analysis of extractable semivolatile organic compounds.

The goal of this procedure is to ensure uniformity involving the preparation of samples for subsequent SVOA analysis by GC/MS. This SOP is applicable to EPA Methods 3510 (modified separatory funnel extraction) and 3520 (continuous liquid-liquid extraction), current revisions.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for semivolatile analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to

Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for semivolatile analysis to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their department follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDS's for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety

Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware, disassembly of CLLEs after extraction, etc. should be disposed of in the "D" waste stream satellite accumulation area nearest the point of generation. Acetone and methanol are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Post-extraction aqueous samples are considered either N-Hi or N-Low waste and should be disposed of in the corresponding satellite waste accumulation area nearest the point of generation. Sodium sulfate used for sample drying should be disposed of in the soil with organics "I" waste stream satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

For aqueous samples extracted by CLLE, a one liter aliquot of sample is adjusted to pH $\leq$ 2 and extracted with methylene chloride using a continuous liquid-liquid extractor. The pH is then adjusted to pH $\geq$ 11 and the sample is extracted again with methylene chloride. A modified separatory funnel extraction may also be used. If this procedure is used, the sample aliquot is first adjusted to pH $\geq$ 11 and then to pH $\leq$ 2. The methylene chloride extract is dried and concentrated to a volume of 1.0 mL.

3.0 INTERFERENCES

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC/MS analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in the total ion current profiles (TICPs). Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in semivolatile analysis. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or teflon or other non-phthalate plastic substitute.

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

Special care should be taken to ensure that clean glassware and apparatus are used and pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

Brand names and catalog numbers are included for illustration purposes only.

4.1 Continuous liquid-liquid extractors - including body, 500 mL round bottom flask and Alhin condensers and equipped with Teflon or glass connecting joints requiring no lubrication (Hershberg-Wolf Extractor, Ace Glass Company, Vineland, NJ, P/N 6841-10 or equivalent).

4.2 Glass powder funnels.

4.3 Fluted filter paper, 18.5cm diameter.

4.4 Concentrator tube - Kuderna-Danish, 10 mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test.

4.5 Evaporation flask - Kuderna-Danish, 500 mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with neck clips.

4.6 Snyder column - Kuderna-Danish, three- or four-ball macro (Kontes K-503000-0121 or equivalent).

4.7 Syringe - gas tight, 1.0 mL, solvent rinsed between each use.

4.8 Vials - Glass, 1.8 mL capacity, with polytetrafluoroethylene (PTFE)-lined screw top and 12 mL with Teflon-lined caps.

4.9 2 L separatory funnel, equipped with Teflon stopper and stopcock; Nalgene Teflon FEP separatory funnels may also be used.

4.10 Organic Free Boiling Chips - approximately 10/40 mesh, Teflon or silicon carbide (or equivalent). Cleaned by Soxhlet for 18 hours.

4.11 Water bath - heated, with concentric ring cover, capable of temperature control (± 20°C). The bath should be used in a hood.

4.12 Nitrogen evaporation apparatus.

4.13 Wide range pH test strips, pH 0-14, Whatman CF Type.

4.14 Glass rods for stirring samples.

4.15 Amber bottles or other appropriate containers for collection of extracts from separatory funnel extraction.

4.16 5 ¾" Pasteur pipets.

4.17 Narrow range pH test strips, pH 0 to 2.5 pH, EMD ColorpHast or equivalent.

4.18 Narrow range pH test strips, pH 11 to 13 pH, EMD ColorpHast or equivalent.

5.0 REAGENTS

All reagent and solvent lots must be checked for possible contamination. Refer to the current version of Katahdin SOP CA-105, Reagent and Solvent Handling, for further details. The extraction staff is responsible for submitting samples to the GC or GC/MS sections for appropriate analysis. All information concerning preparation of the reagent/solvent lot sample will be recorded in the Organic Extraction Log (Figure 1) and acceptance or rejection of these lots must be recorded in the solvent/reagent lot check logbook (Figure 2). All reagents and solvents must be free (<PQL) of any target compounds.

5.1 Laboratory Reagent Grade Water - defined as water in which an interferent is not observed at or above the PQL of each parameter of interest. Deionized water filtered through activated charcoal.

5.2 Sodium sulfate - granular. Bake at 400°C for 4 hours (may be done by vendor). Purify by rinsing three times with pesticide grade methylene chloride. Allow residual methylene chloride to evaporate before each use. Cool in a desiccator and store in a glass bottle with a Teflon-lined cap.

5.3 Sulfuric acid solution (1:1 H_2SO_4 : H_2O) - slowly add 500 mL of H_2SO_4 (sp gr 1.84) to 500 mL reagent water.

5.4 Acetone, methanol, methylene chloride - pesticide residue analysis grade or equivalent.

5.5 Standard Preparation - For all standard preparations, see current revision of the following Katahdin Analytical SOPs:

- "Standards Preparation, Documentation and Traceability", (CA-106, current revision)
- "Balance Calibration," (CA-102, current revision)

5.5.1. Base/Neutral and Acid (SVOA) Surrogate Spiking Solution - Surrogate standards are added to all samples and calibration solutions. Prepare a surrogate standard spiking solution that contains the following compounds at the indicated concentrations in acetone.

Compound	Conc.
phenol-$_{d6}$	100 ug/mL
2,4,6-tribromophenol	100 ug/mL
2-fluorophenol	100 ug/mL
nitrobenzene-$_{d5}$	50 ug/mL
p-terphenyl-$_{d14}$	50 ug/mL
2-fluorobiphenyl	50 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5.2 SIM Surrogate Spiking Solution- Surrogate Standards are added to all samples and calibration solutions. Prepare a surrogate solution that contains the following compounds at a concentration of 2 ug/mL in acetone.

Compound	Conc. ug/mL
Fluorene-d10	2.0 ug/mL
2-Methylnaphthalene-d10	2.0 ug/mL
Pyrene-d10.	2.0 ug/mL
2,4-Dibromophenol	2.0 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5.3 SVOA Matrix Spike/Lab Control Samples Spiking Solution - the matrix spike/LCS solution consists of the compounds listed in Figure 3.

Prepare a spiking solution that contains each of the base/neutral compounds listed in Figure 3 at 50 ug/mL in methanol and the acid compounds at 100 ug/mL in methanol. Matrix spike/LCS standards are stored in the freezer (-10°C to -20°C) located in the storage area.

5.5.4 Base/Neutral (SIM) Matrix Spike/ Lab Control Sample Spike Solution for SIM-SVOA. Prepare a spiking solution in methanol that contains the compounds listed in Figure 3 at a concentration of 2 ug/mL for base/neutral. Take out 1.0 mL of Base/Neutral and Acid Matrix Spike/Lab Control Spiking Solution for SVOA and dilute it to 25.0 mL of methanol. Store the solution Spiking at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.5.5 Base/Neutral and Acid (SVOA) Appendix IX Lab Control Sample / Matrix Spike Spiking Solution – Prepare a spiking solution in methanol that contains the compounds listed in Figure 4 at concentrations of 100 ug/ml. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Continuous liquid-liquid (Method 3520) and/or separatory funnel (Method 3510) extractions for semivolatiles must be started within seven days of date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific. If sampling date is unknown, the hold time is counted from one day prior to date received.

7.0 PROCEDURES

The following information must be recorded in the extraction logbook.

- Extraction method
- Surrogate and spike IDs
- Lot numbers of all solvents, acids and bases, sodium sulfate, filter paper
- Nitrogen evaporation water bath temperature
- Sample pH if appicable
- Extraction and Concentration dates
- Extraction and Concentration analyst
- Sample ID or QC sample ID

- Initial and final volumes or weight
- Surrogate and spike amounts
- Any sample cleanup preformed
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)
- Prep batch start time and end time
- CLLE start time and end time
- Lot number of the vials the concentrated extracts are stored in.

The internal chain-of-custody must be signed when removing and replacing samples in storage locations.

7.1 CONTINUOUS LIQUID-LIQUID EXTRACTION (Method 3520)

7.1.1 Set up the CLLE apparatus. All glassware should be pre-rinsed three times with methylene chloride in order to eliminate any contamination factors.

7.1.2 Add approximately 500 - 600 mL of methylene chloride to the CLLE body. Label each flask with the following: sample number (or QC identification number), analyte (SVOA), extraction method (CLLE), and extraction date.

7.1.3 A method blank and a laboratory control sample (LCS) must be prepared for each daily extraction batch of twenty samples or fewer (if a work order consists of more than twenty samples, a new batch must be started on a separate page with its own method blank and LCS). To prepare method blank and LCS, add 1 L reagent water to a CLLE body. Be sure that no water leaks into the round bottom flask. If combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis. This blank and LCS are carried through the entire extraction and analytical procedure.

7.1.4 Mark the sample level (meniscus) on the sample bottle with a wax crayon so that the volume can be measured (this may be done prior to removal from the walk-in cooler). Transfer the sample to a CLLE body, being sure that no water leaks into the round bottom flask.

7.1.5 If the batch requires a MS/MSD, transfer two 1 L portions of the sample selected/designated for MS/MSD to CLLE bodies for preparation of a matrix spike/matrix spike duplicate if required. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis. If extra MS/MSD aliquots of sample are unavailable a laboratory control sample duplicate (LCSD) may be substituted.

7.1.6 Check the pH of each sample with wide range pH paper by removing a couple of sample drops with a clean disposable pipet or on the tip of a stirring rod. Adjust the pH of the samples (including method blank, LCS/LCSD, and MS/MSD) to $\leq$ pH 2 with 1:1 H_2SO_4 after addition of surrogates and spikes and prior to attaching Allihn condensers (Step 7.1.11). Stir with a glass stirring rod and check pH by tapping the glassrod onto wide range pH paper. The pH must be $\leq$ 2. If the pH test strip does not clearly indicate the pH is less than 2, narrow range pH paper must be used.

7.1.7 For each sample, rinse the original sample container with approximately 30 mL of methylene chloride. Add this rinse to the CLLE body.

7.1.8 Determine the initial volume of the samples by comparing the grease marking where the sample meniscus was to the reference bottle located in the lab. Record the volume and any notable characteristics (e.g. color, presence of sediment, or odor) in the extraction logbook.

7.1.9 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL base/neutral and acid (SVOA) surrogate spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with acetone before and after each use.

7.1.9.1 If the request is for SVOA, use the SVOA Surrogate Solution (sect. 5.5.1).

7.1.9.2 If the request is for SIM, use the SIM surrogate solution (sect. 5.5.2).

7.1.9.3 If the request is for SIM-SVOA, use the SIM surrogate solution as well as SVOA surrogate solution. NOTE: Separate LCS/LCSD and/or MS/MSD are needed for each analysis and should be spiked with the appropriate surrogate.

7.1.10 To LCS/LCSD and MS/MSD add 1.0 mL base/neutral and acid (SVOA) matrix spike/LCS spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with methanol before and after each use.

7.1.10.1 If the request is for SVOA -
add 1.0 mL of SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.3).

7.1.10.2 If the request is for SIM -
add 1.0 mL of SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.3) and
add 1.0 mL of Base/Neutral (SIM) Matrix Spike/ Lab Control Sample Spike Solution (sect 5.5.4).

7.1.10.3 If the request is for SVOA Appendix IX, use the SVOA Appendix IX Spiking solution as well as the SVOA spiking solution -
add 1.0 mL of SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.3) and
add 1.0 mL of Base/Neutral and Acid (SVOA) Appendix IX Lab Control Sample / Matrix Spike Spiking Solution (sect 5.5.5).

7.1.11 Attach cooling water Allihn condensers, after first rinsing each 45/50 joint with methylene chloride. Turn on the heating mantles and allow the samples to extract for 18 to 24 hours. Turn off the mantles and let samples cool.

7.1.12 Detach condensers and verify that the pH is still $\leq$ 2 in the same manner mentioned in 7.1.6. If the pH has changed, more acid should be added to make the pH $\leq$ 2 and the sample extracted for several more hours.

7.1.13 Upon completion of acid extraction, allow the sample to cool. Detach condensers and add enough 10N NaOH to adjust the pH to $\geq$ 11 with stirring. Use glass stirring rods to stir and check the pH of each sample in the same manner mentioned in 7.1.6.

7.1.14 Re-attach Allihn condensers, turn on heating mantles, and allow samples to extract for 18 to 24 hours. Turn off mantles and allow samples to cool.

7.1.15 Detach condensers and verify that the pH is still $\geq$ 11 in the same manner mentioned in 7.1.6. If the pH has changed, more NaOH should be added to make the pH $\geq$ 11 and the sample extracted for several more hours.

7.1.16 Once samples are cool to the touch, the CLLE apparatus can be disassembled. The round bottom flask is removed, covered foil and placed in the interim extract refrigerator. The remaining sample in the CLLE body is poured in the "N-Hi" satellite.

Proceed to Step 7.3 for sample extract concentration procedures.

7.2 SEPARATORY FUNNEL EXTRACTION (Modified Method 3510)

If an emulsion prevents acceptable recovery or client history indicates samples may demonstrate matrix interference, then samples should be extracted by continuous liquid-liquid extraction (CLLE).

7.2.1 Rinse all glassware, including teflon separatory funnels, three times with methylene chloride prior to use.

7.2.2 Label 2 L separatory funnels and amber collection bottles clearly. Each label should include: sample number (or QC indicator number), analyte (SVOA), matrix (Aq), extraction date.

7.2.3 A method blank and a laboratory control sample (LCS) must be prepared for every 20 samples or with each extraction batch, whichever is more frequent. To prepare method blank and LCS, add 1 L reagent water to a separatory funnel. If combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis. This blank and LCS are carried through the entire extraction and analytical procedure.

7.2.4 Measure the initial volume by comparing the meniscus of the sample with the reference bottle of the same bottle type. Please refer to SOP CA-108, "Basic Laboratory Technique", for the reference bottle verification procedure. Record the volume and any notable characteristics (e.g. color, presence of sediment, or odor) in the extraction logbook.

7.2.5 If the batch requires a MS/MSD, transfer two 1 L portions of the sample selected/designated for MS/MSD to separatory funnels for preparation of a matrix spike/matrix spike duplicate if required. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis. If extra MS/MSD aliquots of sample are unavailable, a laboratory control sample duplicate (LCSD) may be substituted.

7.2.6 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL base/neutral and acid (SVOA) surrogate spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with acetone before and after each use.

7.2.6.1 If the request is for SVOA, use the SVOA Surrogate Solution.

7.2.6.2 If the request is for SIM, use the SIM Surrogate Solution.

7.2.6.3 If the request is for SIM-SVOA, use the SIM surrogate solution as well as SVOA surrogate solution. NOTE: Separate LCS/LCSD and/or MS/MSD are needed for each analysis and should be spiked with the appropriate surrogate.

7.2.7 To LCS/LCSD and MS/MSD add 1.0 mL base/neutral and acid (SVOA) matrix spike/LCS spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in the extraction logbook. Thoroughly rinse syringe with methanol before and after each use.

7.2.7.1 If the request is for SVOA, use the SVOA Spiking Solution.

7.2.7.2 If the request is for SIM, use the SIM Spiking solution.

7.2.7.3 If the request is for SVOA Appendix IX, use the SVOA Appendix IX Spiking solution as well as the SVOA spiking solution

7.2.8 For each sample, rinse the original sample container with 60 mL of methylene chloride. Add this rinse to the separatory funnel.

7.2.9 Adjust the pH of the samples (including method blank, LCS/LCSD, and MS/MSD) to pH $\geq$ 11 with 10N NaOH after addition of surrogates and spikes. Stir with a glass stirring rod and check pH by tapping the glass stirring rod onto wide range pH paper. The pH must be $\geq$ 11. If the pH test strip does not clearly indicate the pH is greater than 11, narrow range pH paper must be used.

7.2.10 Add 60 mL of methylene chloride directly to the method blank and LCS/LCSD separatory funnels.

7.2.11 Extract the samples by shaking the funnel for two minutes, venting often, but gently, in a hood to release pressure. A mechanical shaker may be used, where samples are shaken for 3 minutes. Following each shake, allow phases to separate for at least 10 minutes. Drain the methylene chloride layer into an amber collection bottle.

7.2.12 If an emulsion forms, mechanical techniques must be employed to achieve maximum separation. Such means include swirling, centrifugation, and draining through a small separatory funnel. In certain instances, transferring the entire sample into a continuous liquid-liquid extractor (CLLE) may be the only alternative. If any such techniques are used, they must be noted in the

extractions logbook, and the batch transferred to a CLLE batch with its own batch ID.

7.2.13 Add a second 60 mL aliquot of methylene chloride to the separatory funnel and extract for the second time (see 7.2.12 – 7.2.13). Collect the methylene chloride layer in the same amber collection bottle.

7.2.14 Repeat the extraction for a third time as described in 7.2.14.

7.2.15 Following the third shake, using a glass stirring rod, check the pH to ensure that it has remained at $\geq$ 11. If the pH has changed back to neutral range, it must be readjusted to $\geq$ 11 and the sample must be extracted at least one more time, adding the methylene chloride to the same amber bottle, that was previously used. If the pH has remained at a value $\geq$ 11, the pH is then adjusted to $\leq$ 2 with 1:1 H_2SO_4. Add enough 1:1 H_2SO_4 to adjust the pH to $\leq$ 2 with stirring. Use glass stirring rods to stir.

7.2.16 Add 60 mL methylene chloride and extract the samples three times in the same manner described in 7.2.11 – 7.2.13. Collect the methylene chloride layer in the same amber collection bottle used to collect the acid fraction.

7.2.17 Sample waste should be poured into the "n-lo" sattalite.

7.2.18 Proceed to Section 7.3 for extract concentration procedures.

7.3 CONCENTRATING THE EXTRACTS

For Methods 3510 and 3520, the combined fractions are concentrated to a final volume of 1.0 mL.

7.3.1 Rinse the K-D glassware (flask, concentration tube, and snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride. Add two boiling chips to the K-D prior to final rinse. Also rinse the assembled funnels, filter paper, and granular sodium sulfate used for drying the extracts.

7.3.2 Transfer the methylene chloride extract to a K-D concentrator setup through a short stem funnel filled with 1-2 inches of sodium sulfate in fluted filter paper. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mls of methylene chloride. Add the rinsings through the sodium

sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mls of methylene chloride and allow to drain

7.3.3 Transfer the label from the collection bottle or round bottom flask (for CLLE) to a K-D. Remove the funnel and attach a 3- or 4-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.3.4 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.3.5 Reduce the methylene chloride extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39°C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Note any problems or extract losses, if they occur, in the extractions logbook.

7.3.6 Reduce each extract to slightly less than 1 mL and then, using a 5 ¾" pasteur pipet, transfer the final extract and label to a 1.8 mL vial with PTFE-lined cap.

7.3.7 If at any time during the concentration process the concentrator tube goes dry, reextraction must occur immediately.

7.3.8 Using methylene chloride for a quantitative transfer, adjust the final volume of each extract to 1 mL. Use the 1 mL oil-filled reference vial for volume comparison.

7.3.9 Store in refrigerator until GC/MS analysis.

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)
- Each extraction method or level
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

Refer to the current revision of the applicable Katahdin SOP for analysis of semivolatiles for quality control acceptance criteria.

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOP.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, Methods 3510 and 3520 (current revisions), SW-846 Third Edition, Updates I, II, IIA, and IIB, Revised January 1995, US EPA.

LIST OF TABLES AND FIGURES

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Figure 4 Appendix Ix LCS/Matrix Spike Component List

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

TABLE 1

SUMMARY OF METHOD MODIFICATIONS (METHOD 3510, current revision)

TOPIC	KATAHDIN SOP CA-502-06	METHOD 3510, current revision
Apparatus/Materials	1) 250 mL amber bottle or flask 2) 1.0 mL syringe 3) short stem funnels	1) 250 mL Erlenmeyer flask 2) 5.0 mL syringe 3) drying columns
Reagents		
Sample preservation/ handling		
Procedures	1) extract collection in amber bottle or Erlenmeyer flask 2) Add surrogate/spike to sample in CLLE 3) Extract for 3 minutes on mechanical shaker 4) extract three times at pH $\geq$ 11, then extract three times at pH $\leq$ 2. 5) extract dried using Na_2SO_4 in short stem funnels 6) Rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride then rinse the sodium sulfate with ~ 15 mLs of methylene chloride to complete a quantitative transfer 7) water bath temp 75-85 deg C 8) no apparatus height specification for concentration on water bath 9) sample removed from water bath when volume reaches ~6 mL 10) N bath temp no higher than 39 deg C	1) extract collection in Erlenmeyer flask 2) Add surrogate/spike directly to sample bottle 3) Extract by shaking vigorously for 1 - 2 minutes with periodic venting 4) extract three times at pH $\leq$ 2, then extract three times at pH $\geq$ 11. 5) extract dried using Na_2SO_4 in drying columns 6) Rinse the Erlenmeyer flask, which contained the solvent extract, with 20 - 30 mL of methylene chloride to complete the quantitative transfer 7) water bath temp 15-20 deg C above solvent boiling temp 8) partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-20 min 9) sample removed from water bath when volume reaches 1 mL 10) N bath temp 35 deg C
QC - Spikes	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

TABLE 1, continued

SUMMARY OF METHOD MODIFICATIONS (METHOD 3520, current revision)

TOPIC	KATAHDIN SOP CA-502-06	METHOD 3520, current revision
Apparatus/Materials	1) short stem funnels	1) drying columns
Reagents		
Sample preservation/ handling		
Procedures	1) Add surrogate/spike to sample in CLLE 2) Add approximately 500 - 600 mL of methylene chloride to the CLLE body 3) CLLE for 22 ± 2 hours 4) Extract dried using Na_2SO_4 in short stem funnels 5) Rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride then rinse the sodium sulfate with ~ 15 mLs of methylene chloride to complete a quantitative transfer 6) water bath temp 75-85 deg C 7) no apparatus height specification for concentration on water bath 8) sample removed from water bath when volume reaches ~6 mL 9) N bath temp no higher than 39 deg C	1) Add surrogate/spike directly to sample bottle 2) Add 300 - 500 mL of methylene chloride to the distilling flask of the extractor 3) CLLE for 18 - 24 hours 4) Extract dried using Na_2SO_4 in drying columns 5) Rinse the Erlenmeyer flask, which contained the solvent extract, with 20 - 30 mL of methylene chloride to complete the quantitative transfer 6) water bath temp 15-20 deg C above solvent boiling temp 7) partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-20 min 8) sample removed from water bath when volume reaches 1 mL 9) N bath temp 35 deg C
QC - Spikes	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

FIGURE 1

EXAMPLE OF SEMIVOLATILES LOGBOOK PAGE

SVBLM
Sep

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - AQUEOUS SEMI-VOLATILES

Extraction Method: (check one)	SW846 3520 (CLLE)		SW846 3510 (SEP) ✓		SW846 3535 (SPE)	
Analytical Method: (check one)	SW846 8270 ✓	EPA 625	CLP OLM04.2	CLP OLC02.1	CLP SOM01.2	
Standards	Surrogate ID (1): SV2350		Spike ID (1): SV852		Spike ID (3):	
	Surrogate ID (2): SV2349		Spike ID (2): SV2351			
Solvents/Acid/Base	Solvent Lot # (Mec12): H90631		H_2SO_4 # 635026		NaOH Lot # H22101	
Consumables	Filter Paper Lot # K11472365		$NaSO_4$ Lot # 27969003		Vial Lot # 00094428	
Nitrogen Water Bath Temperature 35°C			pH (1st Extraction) ≥11		pH (2nd Extraction) ≤2	
Prep Start Time: 0930	Prep End Time: 1230	CLLE Acid Start:	CLLE Acid End:		CLLE B/N Start:	CLLE B/N End:

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Date Extracted	Ext. Init.	Sample ID	Initial Vol. mL	Surr. Vol.	Spike Vol.	Fraction SV	Fraction SW	Final Vol. mL	Date Conc.	Tray Location	Initials	Comments
10/24/09	CB	W670483-1 W670483-1	1000	1mL each	NR	✓	✓	1mL	10/26/09	SV492 B8	CB	R_{spike} R112490
↓	↓	W670483-2	↓	1mL	1mL		↓	↓		B9	↓	R_{surr} R112487
		-3	980	↓	↓		↓			B10		W6SC6526-11k
		-4	1000				✓			B1		MSD -11L
		W670484-2	1000			✓				C2		
↓	↓	-3	↓	↓	↓	↓		↓	↓	C3	↓	
					CB	10/26/09						

0000089

Date Extracted	Ext. Init.	Sample ID	Initial Vol. mL	Surr. Vol.	Spike Vol.	Fraction SV	Fraction SW	Final Vol. mL	Date Conc.	Tray Location	Initials	Comments
10/24/09	CB	SC6526-1a	1060	1mL	NR		✓	1mL	10/26/09	SV492 C4	CB	
↓	↓	-3c	1010	↓	↓		↓	↓	↓	C5	↓	
		-5d	1030							C6		
		-7c	1060							C7		less double sand.
		-9c	1030							C8		
		-11h	1030							C9		MSD
		-13c	1020							C10		
		-15d	1010							D1		
		-17c	1060							D2		
		-19d	↓							D3		
		-21c	↓							D4		
		-23c	1020							D5		
↓		-25c	1040				✓			D6		
		SC6525-2e	1060			✓				D7		
		-3c	↓			↓				D8		
		-4e	↓							D9		
		-5d	1040							D10		
↓	↓	-6d	1040	↓	↓	↓		↓	↓	E1	↓	

QAEX173

Reviewed By_____ Date_____

0000090

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

FIGURE 2

SOLVENT/REAGENT LOT CHECK LOGBOOK

SOLVENT:

LOT#:

DATE RECEIVED:

DATE CONCENTRATED:

CONCENTRATED BY:

PREP METHOD:

TRAY LOCATION:

ANALYZED BY:

PASS/FAIL:

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

FIGURE 3

LCS/MATRIX SPIKE COMPONENT LIST

BASE/NEUTRALS	
1-Methylnaphthalene	Bis (2-chloroethoxy) methane
1,1-Biphenyl	Bis (2-chloroethyl) ether
1,2,4-Trichlorobenzene	Bis (2-Chloroisopropyl) ether)
1,2-Dichlorobenzene	Bis(2-Ethylhexyl)adipate
1,3-Dichlorobenzene	Bis (2-ethylhexyl) phthalate
1,4-Dichlorobenzene	Butylbenzyl phthalate
1,4-Dioxane	Caprolactam
2,4-Dinitrotoluene	Carbazole
2,6-Dinitrotoluene	Chrysene
2-Chloronaphthalene	Dibenz (a, h) anthracene
2-Methylnaphthalene	Dibenzofuran
2-Nitroaniline	Diethyl phthalate
3,3'-Dichlorobenzidine	Diethyl adipate
3-Nitroaniline	Dimethyl phthalate
4-Bromophenylphenyl ether	Di-n-butylphthalate
4-Chloroaniline	Di-n-octyl phthalate
4-Chlorophenylphenyl ether	Fluoranthene
4-Nitroaniline	Fluorene
Acenaphthene	Hexachlorobenzene
Acenaphthylene	Hexachlorobutadiene
Acetophenone	Hexachlorocyclopentadiene
Aniline	Hexachloroethane
Anthracene	Indeno (1,2,3-cd) pyrene
Atrazine	Isophorone
Azobenzene	Naphthalene
Benzaldehyde	Nitrobenzene
Benzidine	N-Nitrosodimethylamine
Benzo (a) Anthracene	N-Nitroso-di-n-propylamine
Benzo (a) pyrene	N-Nitrosodiphenylamine
Benzo (b) fluoranthene	Phenanthrene
Benzo (ghi) perylene	p-toluidine
Benzo (k) fluoranthene	Pyrene
Benzyl alcohol	Pyridine

ACIDS		
2, 3, 4, 6-Tetrachlorophenol	2-Chlorophenol	Benzoic acid
2,4,5-Trichlorophenol	2-Methylphenol	Ethyl methanesulfonate
2,4,6-Trichlorophenol	2-Nitrophenol	Methyl methanesulfonate
2,4-Dichlorophenol	4,6-Dinitro-2-methylphenol	Pentachlorophenol
2,4-Dimethylphenol	4-Chloro-3-methylphenol	Phenol
2,4-Dinitrophenol	4-Methylphenol	
2,6-Dichlorophenol	4-Nitrophenol	

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

FIGURE 4

APPENDIX IX LCS/MATRIX SPIKE COMPONENT LIST

1,2,4,5-Tetrachlorobenzene	Hexachloropropene
1,3,5-Trinitrobenzene	Isodrin
1,4-Naphthoquinone	Isosafrole
1-Chloronaphthalene	Kepone
1-Naphthylamine	m-Dinitrobenzene
2,4-D	Methapyrilene
2-Acetyl aminofluorene	Methyl parathion
2-Naphthylamine	n-Nitrosodiethylamine
2-Picoline	n-Nitrosodi-n-butylamine
3,3-Dimethylbenzidine	n-Nitrosomethylethylamine
3-Methylcholanthrene	n-Nitrosomorpholine
4-Aminobiphenyl	n-Nitrosopyrrolidine
4-Nitroquinoline-1-oxide	n-Nitrotrosopiperidine
5-Nitro-o-toluidine	O,O,O-Triethyl phosphorothioate
7,12-Dimethylbenz(a)anthracene	o-Toluidine
a,a-Dimethylphenethylamine	Parathion
Acetophenone	p-Dimethylaminoazobenzene
Aramite	Pentachlorobenzene
Chlorobenzilate	Pentachloronitriobenzene
Diallate	Phenacetin
Dibenz(a,j)acridine	Phorate
Dimethoate	p-Phenylenediamine
Dinoseb	Pronamide
Diphenylamine	Safrole
Disulfoton	Silvex (2,4,5-TP)
Famphur	Sulfotep
Hexachlorophene	Thionazin

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS

Prepared By: Mike Thomas Date: 09/96

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: J C Bruton Date: 10/25/00

QA Officer: Deborah J. Nadeau Date: 10·24·00

General Manager: Dennis C. Hufnagel Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	10·24·00	10/24/00
02	Addition of compounds to Figure 2.	DN	3·28·02	3·28·02
03	Definitions added to section 1.1. Wording was added or changed to clarify sections 4, 5, 6, 7, 8 + 9. Minor changes throughout. New figures.	HRC	11.08.04	11.08.04
04	Updated Sect. 5.0 with current spike solutions prep. Removed section on medium level soil extraction. Replaced figure 3 and 4 with current LCS/MS spike components. Minor corrections to sect. 1.3, 4.24, 6.0 and 7.12. Updated logbook	LAD	04/06	04/06
05	Many changes made throughout, including but not limited to, waste information, updated spikes and surrogates, added SIM LCS/D and MS/D information, updated Table 1. Please refer to the QAM/SOP change form filed w/ SOP in QA for a detailed list of changes.	LAD	09/07	09/07

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Updated logbook example. Added adipate compounds to fig. 2. Added necessity of recording lot numbers of consumables in logbook. Added to record the temperature of the nitrogen evaporation water bath.	LAD	07/08	07/08
07	Added requirement to add spike before Na_2SO_4. Changed N_2 water bath temperature from <39°C to <30°C. Removed respirator reference. Added KAEHS manual. Added KAS SOP CA-108 reference for additional subsampling information.	LAD	02/09	02/09
08	Removed targeting sample weights. Added KAT. SOP SD-902 reference. Updated logbook page example. Added GPC cleanup is required for all samples. Removed decanting samples	LAD	08/10	08/10

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-512-08**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS.**

Recipient: _____ Date: _____

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STANDARD OPERATING PROCEDURE

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Recipient: _____ Date: _____

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures and requirements for the preparation of solid samples for analysis of extractable semivolatile organic compounds. This SOP is specifically applicable to EPA Method 3550B in accordance with SW-846 Method 8270, current revision.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for semivolatile analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training”.

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for semivolatile analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab

notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to indicate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the “D” waste stream satellite accumulation area nearest the point of generation. Acetone and methanol are considered flammable waste, and should be disposed of in the “O” waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples and sodium sulfate waste should be disposed of in the soil with organics “I” waste stream satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

2.0 SUMMARY OF METHOD

An approximate, greater than 30 gram portion of sediment/soil is mixed with anhydrous powdered sodium sulfate and extracted with 1:1 methylene chloride/acetone (v/v) using an ultrasonic probe. The methylene chloride extract is dried and concentrated to a volume of 1.0 mL.

3.0 INTERFERENCES

Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause method interferences such as discrete artifacts and/or elevated baselines in the total ion current profiles (TICPs). All of these materials routinely must be demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks. Interferences caused by phthalate esters can pose a major problem in semivolatile organics analysis because many phthalates are also target analytes. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory.

At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or Teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis. Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

Prior to use, all glassware must be rinsed three times with methylene chloride. Brand names and catalog numbers are included below for illustration purposes only.

4.1 Syringe - gas tight, 1.0 mL, solvent rinsed between each use.

4.2 Sonicator ultrasonic processor XL – Misonix (or equivalent) equipped with dual titanium 3/4" horn extenders for extracting two samples at a time.

4.3 Powder funnels, 100 mm diameter, 35 mm stem

4.4 Kuderna-Danish (KD) apparatus - Concentrator tube - 10 mL
Evaporative flask - 500 mL
Snyder column - 3-ball macro

4.5 Filter paper, 7.0 cm, Whatman #4

4.6 Vacuum filtration flask - 500 mL Erlenmeyer

4.7 Buchner funnel, porcelain, Coors® with 85 mm plate diameter (or equivalent)

4.8 Beakers - 400 mL

4.9 Boiling chips - approximately 12 mesh, silicon carbide (carborundum or equivalent). Soxhlet extract overnight in methylene chloride.

4.10 Water bath - eight position concentric ring bath, or equivalent, equipped with a calibrated thermometer. The bath should be used in a hood.

4.11 Balance - capable of accurately weighing ± 0.01 g.

4.12 Vials and caps – 1.8 mL with PTFE/silicone septa and 12 mL with Teflon-lined caps for extracts designated for GPC cleanup.

4.13 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

4.14 Pasteur pipets - disposable, 5 ¾ ".

4.15 Nitrogen evaporation apparatus.

4.16 Muffle oven – capable of maintaining 400 °C for baking glass wool and organic-free sand.

4.17 Gel Permeation Chromatograph (GPC) - J2 Scientific AccuPrep MPS™ with internal UV detection

5.0 REAGENTS

5.1 Sodium Sulfate - anhydrous powdered and granular crystals, reagent grade, certified by the manufacturer/vendor as purified heating to 400°C prior to receipt by the laboratory. Solvent rinse immediately prior to use by rinsing three times with pesticide grade methylene chloride. (Jost Chemical anhydrous powder, catalog #2797 or equivalent, and Jost Chemical granular crystals, catalog #2796 or equivalent).

5.2 Methylene chloride, methanol, and acetone - pesticide residue analysis grade or equivalent. Methylene chloride and acetone are evaluated by lot prior to use by concentration of approximately 400 mL to 1.0 mL followed by GC/MS analysis. The lot numbers of all solvents used during an extraction must be recorded in the extraction logbook.

5.3 Organic-free sand, purified by baking at 400 °C. Method blanks serve as checks on the baked sand.

5.4 Base/Neutral and Acid (SVOA) Surrogate Spiking Solution - Surrogate standards are added to all samples and calibration solutions. Prepare a surrogate standard spiking solution that contains the following compounds at the indicated concentrations in acetone.

Compound	Conc.
phenol-$_{d6}$	100 ug/mL
2,4,6-tribromophenol	100 ug/mL
2-fluorophenol	100 ug/mL
nitrobenzene-$_{d5}$	50 ug/mL
p-terphenyl-$_{d14}$	50 ug/mL
2-fluorobiphenyl	50 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5 SIM Surrogate Spiking Solution- Surrogate Standards are added to all samples and calibration solutions. Prepare a surrogate solution that contains the following compounds at a concentration of 2 ug/mL in acetone.

Compound	Conc. ug/mL
Fluorene-d10	2.0 ug/mL
2-Methylnaphthalene-d10	2.0 ug/mL
Pyrene-d10.	2.0 ug/mL
2,4-Dibromophenol	2.0 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.6 Base/Neutral and Acid (SVOA) Matrix Spike/Lab Control Sample Spiking Solution - Prepare a spiking solution in methanol that contains the compounds listed in Figure 2 at a concentration of 50 ug/mL for base/neutrals and 100 ug/mL for acids. Store the

spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.7 Base/Neutral and Acid (SVOA APPENDIX IX) Matrix Spike/Lab Control Sample Spiking Solution. Prepare a spiking solution in methanol that contains the compounds listed in Figure 3 at a concentration of 100 µg/mL for each compound. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem

5.8 Base/Neutral (SIM) Matrix Spike/ Lab Control Sample Spike Solution for SIM-SVOA. Prepare a spiking solution in methanol that contains the compounds listed in Figure 2 at a concentration of 2 ug/mL for base/neutral. Take out 1.0 mL of Base/Neutral and Acid Matrix Spike/Lab Control Spiking Solution for SVOA and dilute it to 25.0 mL in methanol. Store the solution Spiking at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sediment/soil samples must be collected in a soil jar and must be maintained at 4°C (±2°C).

Holding time for extraction of sediment/soil samples for Method 3550 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

The following information must be recorded in the extraction logbook.

- Extraction method
- Surrogate and spike IDs
- Lot numbers of all solvents, acids and bases, sodium sulfate, filter paper
- Nitrogen evaporation water bath temperature
- pH if appicable
- Sonicator horns tuned
- Extraction and Concentration dates

- Extraction and Concentration analyst
- Sample ID or QC sample ID
- Initial and final volumes or weight
- Surrogate and spike amounts
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)

All solid samples should be cleaned using gel permeation chromatography (GPC) to reduce matrix interferences.

The organic department manager should be consulted to determine if a particular sample should be subjected to further cleanup procedures; the decision should consider sample history, sample appearance, and project/client needs.

Samples need to be “swiped” out when removing and “swiped” in when replacing samples in storage locations to maintain the internal chain of custody. Refer to Katahdin SOP, Sample Receipt and Internal Control, current revision, for the proper procedure for removal and return samples. Fill out the sample preparation/extraction log with the necessary information before starting the extraction. Prerinse all glassware three times with methylene chloride.

7.1 Do not decant any water layer on a sediment sample. Mix with a stainless steel spatula to ensure homogeneity of the sample. If the sample container is full to the extent that stirring the sample is impractical, try to remove the “best representative” aliquot from the jar based on color, particle size, moisture, etc. Remove any foreign objects such as sticks, leaves, and rocks, and note actions taken in the appropriate extraction logbook. Please refer to the current revision of Katahdin Analytical Services SOP CA-108, “Basic Laboratory Technique “, for more detailed guidance on subsampling to ensure reproducibility.

7.2 The following steps should be performed <u>rapidly</u> <u>to</u> <u>avoid</u> <u>loss</u> <u>of the</u> <u>more</u> <u>volatile</u> <u>extractable</u>. Weigh out an approximate, greater than 30g portion of sample into a labeled 400-mL beaker. Record sample weight to the nearest 0.01 g in appropriate extraction logbook. Add between 30 g and 60 g of anhydrous powdered sodium sulfate as required for producing a “free-flowing” mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula. Keep the spatula in the sample beaker and <u>cover</u> the beaker with aluminum foil.

7.3 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare a method blank, weigh out one, greater than 30 g portion of purified sand in a labeled 400 mL beaker. Refer to sections 7.7 and 7.7 for spike and surrogate addition instructions. Add 60 g sodium sulfate and mix well. Although a “free-flowing” mixture can be achieved with less than 60 g sodium

sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.

7.4 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one 30 g portion of purified sand in a labeled 400 mL beaker. Refer to sections 7.7 and 7.7 for spike and surrogate addition instructions. Add 30 g sodium sulfate and mix well. If combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.5 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out two approximate, greater than 30 g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.01 g in appropriate extraction logbook. Refer to sections 7.7 and 7.7 for spike and surrogate addition instructions. Add 30 - 60 g sodium sulfate to each to produce a free-flowing mixture, and mix well. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis.

7.6 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL of the appropriate base/neutral and acid surrogate spiking solution listed below using the pre-rinsed 1.0 mL gas tight syringe. The surrogate spike should be added **prior** to the addition of the sodium sulfate. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to using it for another spiking solution.

7.6.1 If the request is for SVOA or SVOA Appendix IX, use the SVOA surrogate solution (sect. 5.4).

7.6.2 If the request is for SIM, use the SIM surrogate solution (sect. 5.5).

7.6.3 If the request is for SIM-SVOA, use both the SIM and SVOA surrogate solutions. NOTE: Separate LCS/LCSD and/or MS/MSD are needed for each analysis and should be spiked with the appropriate surrogate.

7.7 To the LCS/LCSD and the MS/MSD add 1.0 mL of the appropriate base/neutral and acid (SVOA) matrix spike/LCS spiking solution listed below using a 1.0 mL gas tight syringe. The LCS/MS spike should be added **prior** to the addition of the sodium sulfate. Record the matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse the syringe with solvent when spiking is completed.

7.7.1 If the request is for SVOA, add 1 mL of SVOA Spiking Solution (sect 5.6).

7.7.2 If the request is for SIM, add 1 mL SIM Spiking solution (sect 5.8).

7.7.3 If the request is for SVOA and SIM, add 1mL of SVOA Spiking Solution and 1 mL SIM Spiking solution (sect 5.6 and 5.8).

7.7.4 If the request is for SVOA Appendix IX, add 1mL of SVOA Spiking Solution and 1 mL of SVOA Appendix IX Spiking solution (sect 5.6 and 5.7).

7.8 To assure optimum operation and maximum energy output, the sonicators must be tuned daily prior to extracting samples. The following tuning procedure must be performed with the sonicator probes vibrating in air.

7.8.1 Turn OUTPUT CONTROL knob counter-clockwise to zero. This automatically switches the duty cycle to continuous mode.

7.8.2 Press and hold down the power switch to on.

7.8.3 Press and hold down the TUNE switch. Check if the counter is less or equal to 20%; otherwise, rotate the Tuning Knob (tuning button) clockwise until a reading of 20%or less is obtained.

7.8.4 Release the TUNE switch.

7.8.5 Turn OUTPUT CONTROL KNOB counter-clockwise to 50 and the power switch off.

7.8.6 Confirm that the sonicators were tuned by recording the date and/or percent in the extractions logbook.

7.9 Prior to extracting any samples, ensure that the sonicator probes are decontaminated by rinsing three times with a methylene chloride wash bottle. Collect the waste in a waste beaker. It may sometimes be necessary to wipe the upper part of each probe with a methylene chloride dampened KimWipe. Repeat this decontamination step between each sample on each probe.

7.10 To the mixed and spiked blank and LCS, add 100 mL of the 1:1 methylene chloride/acetone (V/V) solution and proceed with steps 7.11 through 7.14. Record the lot numbers of the solvents in the extraction logbook.

7.11 It may be necessary at this time to stir the sample/sodium sulfate mixture with the spatula to loosen up the mixture prior to extracting. Rinse the spatula with methylene chloride and collect the rinsing into a correspondent beaker. Position the beaker in the ultrasonic cell disruptor so that the bottom surface of the tip of the 3/4

inch disruptor horn is about halfway below the surface of the solvent and above the sediment layer.

7.12 Sonicate for 3 minutes with the output control knob set at 10, and mode switch on "pulsed" and % duty cycle knob set at 50%. While the mixture is sonicating, one should be able to see all, or most of the material, moving in the beaker under the influence of the energized probes. If not, stir the mixture again.

7.13 Prepare a filter flask fitted with a Buchner funnel. The Buchner funnel should contain a 7.0 cm Whatman #4 filter. Prerinse the flask, funnel and filter with methylene chloride and discard rinsings into solvent waste container. Decant extract into the filter flask and Buchner funnel. A vacuum pump may be used to facilitate filtration or the extract may be gravity filtered. The lot number of the filter paper must be written ti the extraction logbook.

7.14 Repeat the extraction two more times (sec 7.11 – 7.14) using 100 mL portions of 1:1 methylene chloride: acetone. Before each extraction, make certain that the sodium sulfate is still free-flowing and not a consolidated mass. As required, break up large lumps with the spatula. Decant the extraction solvent into the Buchner funnel after each sonication. On the final sonication, pour the entire sample contents into the Buchner funnel and rinse thoroughly with methylene chloride to complete the quantitative transfer of the extract. Use the vacuum pump to pull all the extract into the flask

CONCENTRATION OF LOW LEVEL EXTRACTS

7.15 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add two boiling chips to the K-D. Insert fluted 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride. Place the assembled K-D's under the funnels. The lot number of the filter paper must be written ti the extraction logbook.

7.16 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mls of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mls of methylene chloride and allow to drain.

7.17 If samples are to be GPC'd, refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures.

7.18 If samples are not to be GPC'd, follow Steps 7.19 through 7.24 to concentrate extracts to final volume of 1 mL. Otherwise proceed to GPC cleanup procedure as described in the current revision of Katahdin SOP CA-513.

7.19 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.20 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. **Do not allow the evaporator to go dry. If the sample extract does go dry, re-extraction must occur immediately.** Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.21 Reduce the extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be < 30°C. Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Record the temperature of the water in the nitrogen evaporation water bath in the logbook also note any problems or extract losses, if they occur, in the extractions logbook.

7.22 When the apparent volume reaches slightly less than 1 mL, remove the concentrator tube and allow it to cool.

7.23 Complete the quantitative transfer of the extract to a 1.8 mL vial by using methylene chloride. Adjust the volume of the methylene chloride extract to 1.0 mL using the 1.8 mL reference vial for volume comparison.

7.24 Label the vial with lab sample number, extraction date, matrix and analysis. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extraction logbook the box number and "tray location" of the individual extract vials.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

A laboratory control sample (LCS) is required for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

Refer to the current revision of the applicable Katahdin SOP for analysis of Semivolatile Organics for quality control acceptance criteria.

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the applicable analytical SOP for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, Method 3550C, USEPA SW-846, Third Edition, Update IV, February 2007.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

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TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

TABLE 1

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-512-08	METHOD 3550, current revision
Apparatus/Materials	1) short stem funnels	1) drying columns
Reagents		
Sample preservation/ handling		
Procedures	1) extract dried using Na_2SO_4 in short stem funnels 2) place sonicator horns ½ way between the surface of the solvent and the sediment layer 3) no apparatus height specification for concentration on water bath 4) water bath at 75-85 deg C 5) sample removed from water bath when volume reaches ~6 mL	1) extract dried using Na_2SO_4 in drying columns 2) place sonicator horns ½ inch below the solvent surface but above sediment layer 3) partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4) water bath at 80-90 deg C 5) sample removed from water bath when volume reaches 1-2 mL
QC - Spikes	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - Accuracy/Precision		
QC - MDL		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE

EX-008 - Revision 1 - 12/20/2009

SV
SOW

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - SOIL SEMIVOLATILE

Extraction Method: (check one)	SW846 3550 (SONIC.) ✓	SW846 3540 (SOX)	SW846 3545 (ASE)
	SW846 3580 (OIL / WIPES)	OTHER	
Analytical Method: (check one)	SW846 8270 ✓	OTHER	
Standards	Surrogate ID (1): SV2401	Spike ID (1): SV2399	Spike ID (3):
	Surrogate ID (2):	Spike ID (2):	
Solvents / Chemicals / Consumables	Solvent Lot # (Mecl2): J22E10	Solvent Lot # (Acetone): H26E28	Sodium Sulfate (granular) Lot # 2796002
	Filter Paper Lot # (SON) R1558982	Filter Paper Lot # (KD) F6000494	Sodium Sulfate (powder) Lot # 2797001
Misc.	Nitrogen Bath Temperature: 37C	Sonicator Horns Tuned: ✓ 20%	Balance ID: Mettler 6140

Start: 9:55 End: 12:00

QAEX183

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spk. Vol. (mL)	Fraction SOX	Fraction SE	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	Tray Loc. (SVOC)	Comments
7/29/10	WS	WW30267-1	29.98	1mL	NR	✓		7/29/10	KF	5mL	7/30/10	WS	1mL	C9	R133081
↓	↓	↓ -2	29.98	↓	1mL	↓		↓	↓	↓	↓	↓	↓	C10	
↓	↓	↓ -3	29.97	↓	↓	↓		↓	↓	↓	↓	↓	↓	D1	
↓		GPC Blank									↓	↓	↓	E2	
															WS 7/30/10

0000147

EX-008 - Revision 1 - 12/20/2009

QAEX183

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spk. Vol. (mL)	Fraction SOX	Fraction SE	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	Tray Loc. (SVOC)	Comments
7/29/10	WS	SO4560-1	31.65	1mL	NR	✓		7/29/10	KF	5mL	7/30/10	WS	1mL	D2	
↓	↓	↓ -2	31.44	↓	↓	↓		↓	↓	↓	↓	↓	↓	D3	
↓	↓	↓ -3	31.81	↓	↓	↓		↓	↓	↓	↓	↓	↓	D4	
↓	↓	↓ -4	32.35	↓	↓	↓		↓	↓	↓	↓	↓	↓	D5	
		SO4481-1	30.00											D6	
		SO4581-1	31.25											D7	
		↓ -2	30.48											D8	
		↓ -3	33.15											D9	
		↓ -4	30.70											D10	
↓	↓	↓ -5	30.79	↓	↓	↓		↓	↓	↓	↓	↓	↓	E1	
															WS 7/30/10

0000148

Reviewed By_____ Date_____

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

FIGURE 2

LCS/MATRIX SPIKE COMPONENT LIST

BASE/NEUTRALS	
1-Methylnaphthalene	Bis (2-chloroethoxy) methane
1,1-Biphenyl	Bis (2-chloroethyl) ether
1,2,4-Trichlorobenzene	Bis (2-Chloroisopropyl) ether)
1,2-Dichlorobenzene	Bis (2-ethylhexyl) adipate
1,3-Dichlorobenzene	Bis (2-ethylhexyl) phthalate
1,4-Dichlorobenzene	Butylbenzyl phthalate
1,4-Dioxane	Caprolactam
2,4-Dinitrotoluene	Carbazole
2,6-Dinitrotoluene	Chrysene
2-Chloronaphthalene	Dibenz (a, h) anthracene
2-Methylnaphthalene	Dibenzofuran
2-Nitroaniline	Diethyl adipate
3,3'-Dichlorobenzidine	Diethyl phthalate
3-Nitroaniline	Dimethyl phthalate
4-Bromophenylphenyl ether	Di-n-butylphthalate
4-Chloroaniline	Di-n-octyl phthalate
4-Chlorophenylphenyl ether	Fluoranthene
4-Nitroaniline	Fluorene
Acenaphthene	Hexachlorobenzene
Acenaphthylene	Hexachlorobutadiene
Acetophenone	Hexachlorocyclopentadiene
Aniline	Hexachloroethane
Anthracene	Indeno (1,2,3-cd) pyrene
Atrazine	Isophorone
Azobenzene	Naphthalene
Benzaldehyde	Nitrobenzene
Benzidine	N-Nitrosodimethylamine
Benzo (a) Anthracene	N-Nitroso-di-n-propylamine
Benzo (a) pyrene	N-Nitrosodiphenylamine
Benzo (b) fluoranthene	Phenanthrene
Benzo (ghi) perylene	p-toluidine
Benzo (k) fluoranthene	Pyrene
Benzyl alcohol	Pyridine

ACIDS		
2, 3, 4, 6-Tetrachlorophenol	2-Chlorophenol	Benzoic acid
2,4,5-Trichlorophenol	2-Methylphenol	Ethyl methanesulfonate
2,4,6-Trichlorophenol	2-Nitrophenol	Methyl methanesulfonate
2,4-Dichlorophenol	4,6-Dinitro-2-methylphenol	Pentachlorophenol
2,4-Dimethylphenol	4-Chloro-3-methylphenol	Phenol
2,4-Dinitrophenol	4-Methylphenol	
2,6-Dichlorophenol	4-Nitrophenol	

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

FIGURE 3

APPENDIX IX LCS/MATRIX SPIKE COMPONENT LIST

1,2,4,5-Tetrachlorobenzene	Hexachloropropene
1,3,5-Trinitrobenzene	Isodrin
1,4-Naphthoquinone	Isosafrole
1-Chloronaphthalene	Kepone
1-Naphthylamine	m-Dinitrobenzene
2,4-D	Methapyrilene
2-Acetyl aminofluorene	Methyl parathion
2-Naphthylamine	n-Nitrosodiethylamine
2-Picoline	n-Nitrosodi-n-butylamine
3,3-Dimethylbenzidine	n-Nitrosomethylethylamine
3-Methylcholanthrene	n-Nitrosomorpholine
4-Aminobiphenyl	n-Nitrosopyrrolidine
4-Nitroquinoline-1-oxide	n-Nitrotrosopiperidine
5-Nitro-o-toluidine	O,O,O-Triethyl phosphorothioate
7,12-Dimethylbenz(a)anthracene	o-Toluidine
a,a-Dimethylphenethylamine	Parathion
Acetophenone	p-Dimethylaminoazobenzene
Aramite	Pentachlorobenzene
Chlorobenzilate	Pentachloronitriobenzene
Diallate	Phenacetin
Dibenz(a,j)acridine	Phorate
Dimethoate	p-Phenylenediamine
Dinoseb	Pronamide
Diphenylamine	Safrole
Disulfoton	Silvex (2,4,5-TP)
Famphur	Sulfotep
Hexachlorophene	Thionazin

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-515
Revision History
Cover Page
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TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

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SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	10·23·00	
02	Addition of SPE Procedure. Minor changes through out Added wording to Sections 6 and 8	LAD	013105	013105
03	Added separate QC for Pest. and PCB. Updated concentration procedure to reflect current practices. Changes in wording for clarification. Update Logbook page	LAD	04/06	04/06
04	Added waste generated and disposal info. Added missing definitions. Updated SPE extraction procedure. Updated Table 1 and 2. Added Table 3.	LAD	09/07	09/07
05	Updated logbook example. Added logbook requirements	LAD	09/08	09/08

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

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06	Added information for determining initial volume. Added reference to CA-108. Added clarification for LCS/D and MS/D sets for PEST/PCB analysis. Minor changes to reflect current techniques.	LAD	10/09	10/09
07	Added additional solvent exchange procedure. Updated logbook example.	LAD	08/10	08/10

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

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1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services, Inc. laboratory personnel for the preparation of aqueous samples prior to analysis for pesticides/PCBs by GC/ECD. It includes extraction of water samples by separatory funnel, continuous liquid-liquid, and solid phase extraction methods (EPA Methods 3510, 3520, 3535A, and EPA Method 608 current revisions).

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in the extraction of aqueous samples for pesticides/PCBs analysis. Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training”.

It is the responsibility of all Katahdin personnel involved in the preparation of aqueous samples for pesticides/PCBs analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the

samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for the data.

It is the responsibility of the Supervisor to oversee that members of their group follow this SOP, that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their supervisor, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the “D” waste stream satellite accumulation area nearest the point of generation. This includes the methylene chloride waste layer generated during CLLE extraction. Special care should be taken to pour this layer off into the appropriate waste stream, leaving the sample waste to be disposed of as follows. Since Pesticide/PCB samples are at a neutral pH, SEP funnel or CLLE sample waste may be dumped into either the “N-Hi” or “N-low” satellite accumulation area. Acetone and hexane are considered flammable waste, and should be disposed of in the “O” waste stream satellite accumulation area nearest the point of generation. Acid waste generated during the cleanup of PCB samples should be disposed of in the “O” satellite accumulation area nearest the point of generation. Please refer to

the current revision of SOP CA-107 for the location of satellite waste accumulation areas

2.0 SUMMARY OF METHOD

Pesticides/PCBs are extracted from aqueous samples using methylene chloride and separatory funnel, continuous liquid-liquid apparatus or Automated Extractor System (SPE), following EPA Methods 3510, 3520, 3535A and EPA Method 608. The methylene chloride is exchanged with hexane for the final extract. Method detection limit studies must be performed annually for pesticides/PCBs using all extraction methods, if the extraction lab wishes to use either or all techniques. Method 3510 (separatory funnel) is generally preferred for pesticides/PCBs since organochlorine pesticides may dechlorinate if under elevated pH conditions for an extended period of time. (Section 3.2, Method 3510B, Rev. 2, 9/94)

3.0 INTERFERENCES

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates which are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves which have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or Teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

Prior to use, all glassware must be rinsed three times with the solvent to be used for extraction.

4.1 Separatory Funnel - 2000 mL capacity, Nalgene Teflon FEP separatory funnels with Nalgene Tefzel® screw-cap closures (or equivalent)

4.2 Concentrator tube - 10 mL, graduated

4.3 Evaporative flask - Kuderna-Danish, 500 mL capacity attached to concentrator with neck clips

4.4 Snyder column - Kuderna-Danish, three ball macro

4.5 Graduated cylinders - 100 mL, 1000 mL, or 2000 mL

4.6 Short Stem Funnels

4.7 250 mL amber collection bottles with Teflon-lined caps

4.8 12 mL and/or 16 mL glass vials with Teflon-lined caps

4.9 Continuous liquid-liquid extractors (CLLE) including body, 500 mL flat bottom boiling flask and Alhin condensers

4.10 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

4.11 Nitrogen evaporation apparatus.

4.12 Boiling chips - approximately 10/40 mesh, Teflon or selenized carborundum, 12 mesh (or equivalent). Cleaned by Soxhlet.

4.13 Water bath - eight position concentric ring bath or equivalent, equipped with a calibrated thermometer.

4.14 Vials, 60 mL with PTFE – lined screw caps.

4.15 Horizon SPE-DEX 4790 Automated Extractor System.

4.16 Atlantic DVB disks, or equivalent.

4.17 1-L amber bottles

5.0 REAGENTS

5.1 Laboratory reagent grade water - water in which an interferent is not observed at or above the PQL for any parameter of interest (carbon filtered ASTM Type II water or equivalent)

5.2 Sodium Hydroxide (10N) – Purchased from vendor, “Baker-analyzed”, or equivalent

5.3 Sodium Sulfate (ACS) - Granular, anhydrous. Bake at 400°C for 4 hours (may be done by vendor). Purify by rinsing three times with pesticide grade methylene chloride. Allow residual methylene chloride to evaporate before use. Stored in a Teflon capped glass bottle.

5.4 Sulfuric Acid Solution (1:1) - Add 500 mL concentrated sulfuric acid (certified ASC grade or better) slowly to 500 mL laboratory reagent grade water. Prepare as needed and store in a ground glass stoppered bottle.

5.5 Methylene Chloride ($MeCL_2$) - Pesticide grade or better. Lot must be verified by concentrating 300-400 mL to 1.0 mL and evaluating by GC/MS.

5.6 Acetone and Hexane - Pesticide grade or better. Lot must be verified by concentrating approximately 20-30 mL to 1.0 mL and evaluating by GC/ECD.

5.7 Pesticide/PCB Surrogate spiking solution - Prepare a solution of decachlorbiphenyl (DCB) and tetrachloro-meta-xylene (TCMX) at a concentration of 1.0 ug/mL ea in acetone. Store the solution at –10 to -20 °C in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

5.8 Pesticide Matrix Spike/Lab Control Sample spiking solution - Prepare a matrix spiking solution in pesticide grade methanol that contains all target analytes listed below:

ANALYTE	ug/mL
4,4’-DDT	0.5
4,4’-DDD	0.5
4,4’-DDE	0.5
Aldrin	0.5
Dieldrin	0.5
Endrin	0.5
Endrin Aldehyde	0.5
Endrin Ketone	0.5
Endosulfan I	0.5
Endosulfan II	0.5
Endosulfan Sulfate	0.5
alpha-BHC	0.5

ANALYTE (cont.)	ug/mL
beta-BHC	0.5
delta-BHC	0.5
gamma-BHC (Lindane)	0.5
Heptachlor	0.5
Heptachlor epoxide	0.5
Methoxychlor	0.5
alpha-Chlordane	0.5
gamma-Chlordane	0.5

5.9 PCB Matrix Spike/Lab Control Sample spiking solution - Prepare a matrix spiking solution in pesticide grade acetone that contains 5.0ug/ml ea of Aroclor® 1016/1260 mix (Restek catalog# 32039).

5.10 Store the spiking solutions at –10 to -20 °C in a Teflon sealed container. The solutions must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples are collected in 1 L amber bottles and held at 4 (±2) °C until time of extraction.

Holding time for extraction of aqueous samples for Methods 3510, 3520, and 3535 is 7 days from date of sample collection, although the analyst should be aware that actual holding times employed might be project/program specific.

7.0 PROCEDURES

The following information must be recorded in the extraction logbook.

- Extraction method
- Surrogate and spike IDs
- Lot numbers of all solvents, acids and bases, sodium sulfate, filter paper
- Nitrogen evaporation water bath temperature
- Sample pH if appicable
- Extraction and Concentration dates
- Extraction and Concentration analyst
- Sample ID or QC sample ID
- Initial and final volumes or weight
- Surrogate and spike amounts
- Any sample cleanup preformed
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)

- Prep batch start time and end time
- CLLE start time and end time
- Lot numbers of the vials the concentrated extracts are stored in.

SEPARATORY FUNNEL SAMPLE EXTRACTION

If an emulsion prevents acceptable recovery or client history indicates samples may demonstrate matrix interence, then samples should be extracted by continuous liquid-liquid extraction (CLLE).

7.1 Rinse all glassware three times with methylene chloride prior to use.

7.2 Label a 2 L Teflon separatory funnel and a 250 mL amber collection bottle clearly. Label should include laboratory sample number, matrix, analyte, and extraction date. Be sure that the detachable stopcocks are secured to the separatory funnels before adding samples.

7.3 Measure the initial volume by comparing the meniscus of the sample with the reference bottle of the same bottle type. Please refer to SOP CA-108, “Basic Laboratory Technique”, for the reference bottle verification procedure. Record the volume and any notable characteristics (e.g. color, presence of sediment, or odor) in the extraction logbook.

7.4 Transfer the contents of the sample bottle to a 2 L separatory funnel.

7.5 Transfer 1 L of laboratory reagent grade water to a 2 L separatory funnel. This serves as a method blank for the extraction batch. A method blank must be prepared for every daily extraction batch of twenty or fewer samples.

7.6 Transfer 1 L of laboratory reagent grade water to a 2 L separatory funnel for each analysis to be performed (pesticide and/or PCB). This will serve as a Laboratory Control Sample (LCS). When Pesticides and PCBs are extracted together, a LCS and LCSD set must be extracted for each analysis. An LCS is required for every daily extraction batch of twenty or fewer samples and each analysis. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.7 A matrix spike/matrix spike duplicate (MS/MSD) is to be prepared as requested by a client or, at a minimum, one pair per 20 samples or every 14 days and each analysis(refer to the logbook page, “date QC expires”). Transfer two additional 1 L aliquots of sample to 2 L separatory funnels for a matrix spike and matrix spike duplicate (MS/MSD) for each analysis. When Pesticides and PCBs are extracted together, a MS and MSD set must be extracted for each analysis. Note: Sufficient sample volume should be available without depleting all remaining sample aliquots.

7.8 Check the pH of the samples. If it is not between pH 5 and 9, adjust the pH with 10N sodium hydroxide or 1:1 sulfuric acid solution. Note the addition of NaOH or H_2SO_4 in the extraction logbook.

7.9 Using a gas-tight syringe, add 1.0 mL of surrogate spiking solution to all samples the blank, LCS/LCSD(s) and MS/MSD(s), if performed.

7.10 Using a gas-tight syringe, add 1.0 mL of pesticide or PCB matrix spiking solution to the appropriate LCS, LCSD, MS and MSD if performed.

7.11 To each empty sample bottle add 60 mLs of methylene chloride, rinse the bottle and transfer the solvent into the appropriate separatory funnel. Add 60 mL of methylene chloride directly to the blank and LCS/LCSD(s).

7.12 Ensure that each screw cap is secured tightly to the separatory funnel to prevent leaks. Shake briefly and vent in hood to release pressure. Extract the sample by shaking the funnel on mechanical shaker for 3 minutes. Allow phases to separate for at least 10 minutes. Drain the methylene chloride layer into the 250 mL amber collection bottle.

7.13 If an emulsion forms, mechanical techniques must be employed to achieve maximum separation and solvent recovery. Such means include swirling and centrifugation and draining through a small separatory funnel. In certain instances, transferring the entire sample into a continuous liquid-liquid extractor may be the only alternative. If any such techniques are used, they must be noted in the extractions logbook.

7.14 Add a second 60 mL aliquot of methylene chloride to the separatory funnel and extract for the second time (see 7.10 - 7.12). Collect the methylene chloride layer in the same 250 mL amber collection bottle.

7.15 Repeat the extraction for a third time as described in 7.13.

7.16 Proceed to Section 7.53 for extract concentration procedures.

CONTINUOUS LIQUID-LIQUID SAMPLE EXTRACTION (CLLE)

7.17 Set up the CLLE apparatus. All glassware should be rinsed three times with methylene chloride and the extract flasks properly labeled.

7.18 Add 2-3 boiling stones to the round bottom flask and approximately 500 - 600 mL of methylene chloride to the CLLE body.

7.19 Add 1 L laboratory reagent grade water to a CLLE body. This is the method blank for this extraction batch. Be sure that no water leaks into the round bottom flask. A method blank is required for every extraction batch of twenty or fewer samples.

7.20 Mark the sample level (meniscus) on the sample bottle with a wax crayon so that the volume can be measured (this may be done prior to removal from the walk-in cooler). Transfer the sample to a CLLE body, being sure that no water leaks into the round bottom flask.

7.21 Prepare an LCS for every daily extraction batch of twenty or fewer samples and each analysis (pesticide and/or PCB). Add 1 L of laboratory reagent grade water to a CLLE body. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager. When Pesticides and PCBs are extracted together, a LCS and LCSD set must be extracted for each analysis.

7.22 Mark the sample levels on the sample bottles. Transfer the samples to the CLLE bodies.

7.23 Check the pH of the samples. If it is not between pH 5 and 9, adjust the pH with 10N sodium hydroxide or 1:1 sulfuric acid solution. Note the addition of NaOH or H_2SO_4 in the extraction logbook.

7.24 Transfer two 1 L portions of a sample to CLLE bodies for each analysis for preparation of a matrix spike/matrix spike duplicate if required. An MS/MSD is required if requested by the client or per 20 samples, whichever occurs first. When Pesticides and PCBs are extracted together, a MS and MSD set must be extracted for each analysis. Note: Sufficient sample volume should be available without depleting all remaining sample aliquots.

7.25 For each sample, rinse the original sample container with approximately 30 mL of methylene chloride. Add this rinse to the CLLE body.

7.26 Determine the initial volume of the samples by comparing the grease marking where the sample meniscus was to the reference bottle located in the lab. Please refer to SOP CA-108, “Basic Laboratory Technique”, for the reference bottle verification procedure. Record the volume and any notable characteristics (e.g. color, presence of sediment, or odor) in the extraction logbook.

7.27 Add 1.0 mL of the Pesticide/PCB Surrogate Spike to each sample including the blank, LCS/LCSD and MS/MSD, if performed.

7.28 Add 1.0 mL of Pesticide or PCB Matrix Spike to the appropriate LCS/LCSD and MS/MSD pair, if performed, and stir.

7.29 Attach cooling water Allihn condensers, after first rinsing each 45/50 joint with methylene chloride. Turn on the heating mantles and allow the samples to extract for at least 18 hours, total extract time may go up to 20 hours. Turn off the mantles and let samples cool.

7.30 Proceed to Section 7. 53 for sample extract concentration procedures.

EXTRACTION WITH AUTOMATED EXTRACTOR SYSTEM (SPE)

Alternatively, samples may be extracted using the Horizon Automated Extractor System (Figure 2)

Purging the Extractor Vessels

7.31 Check and fill all necessary solvent bottles (acetone, laboratory reagent grade water and hexane) as needed. Check and empty the two waste containers as needed.

7.32 Turn on nitrogen tank to 60 psi. Turn the instrument pressure on top of the controller to 50 psi. Turn the solvent bottle pressure to 10 psi.

7.33 Turn on the Horizon controller (switch in the back).

7.34 Check the lubrication oil on the air pump. Fill as needed. Turn the air pump on.

7.35 Clean the glass sensors that are located on the back of the dispensing stems of the extractors using a Kim Wipe. This is to remove any residue that may interfere with the sensors.

7.36 Attach 19/22 adapters to 40-mL vials and attach beneath the disk holder platforms of the extractors. Assembly per owner's manual and place empty Horizon disk holder assemblies on top of the disk holder platforms. There should be roughly 1 cm separating the speedisk from the extractor downtube.

7.37 Check to be sure that all extractors have empty sample bottles loaded on top. If not, use a Horizon cap on a one liter empty bottle and firmly place the bottlenose down into the extractor.

7.38 Press *select* on the control panel to designate an extractor (1, 2, 3, 4 or "." for all), then press *enter*.

7.39 Type 8081.9, and press enter to select pesticide/PCB purge method. Once the method is loaded, start the extractors by pressing the *start* buttons on the individual extractors. The red LED will blink when the method is complete.

7.40 Repeat this process 2-3 times before using the Horizon autoextractors.

ANALYSIS OF SAMPLES WITH AUTOEXTRACTOR

7.41 Label a 60 ml vial with the sample to be extracted. Attach 19/22 adapter to vial and attach beneath the disk holder platforms of the extractors.

7.42 Place an Atlantic DVB disk (or equivalent) into a Horizon disk holder assembly and assemble per owner's manual. Place the disk holder assemble on top of the disk holder platform. There should be roughly 1 cm separating the speedisk from the extractor downtube

7.43 Mark the volume level of liquid in each sample on the outside using a grease pencil.

7.44 Add 1 L laboratory reagent grade water to 1 L amber bottle. This is the method blank for this batch. A method blank is required for every extraction batch of twenty or fewer samples.

7.45 Prepare an LCS for every daily extraction batch of twenty or fewer samples and each analysis, pesticide and/or PCB. Add 1 L of laboratory reagent grade water to a 1 L amber bottle. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required to meet client specific or program specific requirements. This information will be disseminated from the project manager or department manager.

7.46 Add 1.0 mL of Pesticide/PCB surrogate spike to each sample including the blank, LCS/LCSD and MS/MSD, if required. Recap samples and shake well.

7.47 Add 1.0 mL of pesticide or PCB matrix spike to the appropriate LCS/LCSD and MS/MSD samples. Recap and shake well.

7.48 Remove cap and add 5.0 mL of 1:1 H_2SO_4 to each sample including the blank, LCS/LCSD and MS/MSD set immediately prior to extracting the sample.

7.49 Remove the cap from each sample bottle and cover with tin foil. Screw a Horizon adapter cap over the tin foil. Invert the bottle and check for leaks.

7.50 Load the sample bottle on the holder and twist ¾ of a rotation. Stop twisting when air bubbles rise to the top of the sample bottle. Do not twist completely around. The foil may loosen and jam the valve.

7.51 Press *select* on the control panel to designate an extractor (1, 2 or "." for both), then press *enter*.

7.52 Type in 8081.3 for the method and press enter. Once the method is loaded, start the extractors by pressing the *start* buttons on the individual extractors. The red LED will blink when the method is complete. The extract will be collected in the 60 ml vial.

7.53 Sample is now ready to reduce to 10 mL final volume.

NOTE: The instrument methods stated above apply specifically to the Atlantic DVB disk. Instrument methods may need to be modified with the usage of different filters and/or to increase recoveries. See instrument logbook for current methods in use.

CONCENTRATION OF WATER SAMPLE EXTRACTS

7.54 Rinse the K-D glassware (flask, concentration tube, funnel and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride (or hexane for samples extracted with the Autoextractor) before assembling. Add two boiling chips to the K-D. Insert fluted 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride (hexane for samples extracted with the Autoextractor). Place the assembled K-D's under the funnels.

7.55 For methylene chloride extracts, add approximately 50 mL Hexane to funnel and let drain through. Since methylene chloride has a lower boiling point than Hexane, this will result in a final extract in hexane only.

Note: For Pesticide / PCB samples originating from South Carolina (see worknotes) do not add the hexane at this step. Solvent exchange will be during the nitrogen blow down procedure.

7.56 Transfer the methylene chloride or hexane extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract bottle three times with ~ 2 – 3 mLs of methylene chloride (or hexane for samples extracted with the Autoextractor). Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mLs of methylene chloride (or hexane for samples extracted with the Autoextractor) and allow to drain.

7.57 Transfer the labels from the collection bottles or round bottom flasks (from the CLLE extraction) to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride (or hexane for samples extracted with the Autoextractor).

7.58 Place the K-D in a hot water bath (85-90°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 5-6 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of hexane (methylene chloride for samples that have not gone through solvent exchange (ie. South Carolina samples). Remove the Snyder column. Wipe off any

water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL hexane.

7.59 Reduce the extracts to ≈ 1 mL using Nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (45 °C for hexane). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of hexane (methylene chloride for samples not yet solvent exchanged). The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging needle closer to the surface of the extract to expedite the concentration. Note any problems or extract losses, if they occur, in the extractions logbook. Transfer extract to a 12 or 16 mL vial. Using a reference vial for volume comparison, adjust the final extract volume to 10 mL by rinsing sides of tube with hexane and transferring rinsings to vial.

7.60 For samples that still need to be solvent exchanged, reduce the methylene chloride extract to ~ 1 mL. Add 10 mL of hexane to the concentrator tube and reduce to ~ 1 mL again on the N-evap. Adjust final extract to 10 mL by rinsing sides of tube with hexane and transferring rinsings to vial.

7.61 If at any point in the concentration procedure the concentrator tube goes dry – reextract the sample immediately.

7.62 Transfer the label from the concentrator tube to the vial. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extractions logbook the box number and “tray location” of the individual extract vials.

7.63 All sample extracts for 8082 PCB analysis must undergo a sulfuric acid wash (cleanup) prior to analysis. All sample extracts for 8081 pesticide analysis do not undergo further cleanup unless requested by the client. Therefore, all sample extracts for combined 8081/8082 analyses must be split. Prior to splitting, mix contents of vial well. One portion must be acid cleaned for 8082 analysis. The associated method blank must be split and acid-cleaned in the same fashion. PCB LCSs and matrix spikes are acid cleaned also. Pesticide LCSs and matrix spikes are not subjected to further cleanup. Please refer to Katahdin SOP CA525 (current revision), Extract Cleanup Using Sulfuric Acid, for further instructions.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration

must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

A method blank must be extracted for <u>each</u> and <u>every</u> item listed below:

- Each day of extraction (24 hours midnight - midnight)
- Each extraction method
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for <u>each</u> and <u>every</u> item listed below:

- Each extraction method
- Every extraction batch of twenty or fewer samples
- Each analysis (pesticide and/or PCB) to be performed

Refer to the current revision of the applicable Katahdin SOP for analysis of Pesticides and PCBs for quality control acceptance criteria.

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOP.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Methods 3510C and 3520C, USEPA SW-846, Third Edition, Final Update III, December 1996.

40 CFR 136, Appendix A, "Test Procedures for Analysis of Organic Pollutants," Method 608, June, 1998 edition.

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TABLE 1

SUMMARY OF METHOD MODIFICATIONS (METHOD 3510, current revision)

TOPIC	KATAHDIN SOP CA-515-07	METHOD 3510, current revision
Apparatus/Materials	1. 12 or 16 mL vials used for final extract 2. 250 mL amber bottle or flask used 3. 1.0 mL syringe 4. short stem funnels	1. 2 mL vials used for final extract 2. 250 mL Erlenmeyer flask 3. 5.0 mL syringe 4. drying column
Reagents		
Sample preservation/ handling	1. entire contents of 1 L sample bottle transferred to separatory funnel	1. one liter graduated cylinders used to transfer initial sample volume to separatory funnel
Procedures	1. extract collection in amber bottle or Erlenmeyer flask 2. extract dried using Na_2SO_4 in short stem funnels 3. no apparatus height specification for concentration on water bath 4. sample removed from water bath when volume reaches ~10 mL 5. hexane added directly to K-D body at start of concentration process	1. extract collection in Erlenmeyer flask 2. extract dried using Na_2SO_4 in drying columns 3. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4. sample removed from water bath when volume reaches 1-2 mL 5. solvent exchange via large K-D with addition of 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TABLE 2

SUMMARY OF METHOD MODIFICATIONS (METHOD 3520, current revision)

TOPIC	KATAHDIN SOP CA-515-07	METHOD 3520, current revision
Apparatus/Materials	1. short-stem funnels 2. 12 or 16 mL vials used for final extract	1. drying columns 2. 2 mL vials used for final extract
Reagents		
Sample preservation/ handling	1. entire contents of 1 L sample bottle transferred to CLLE	1. one liter graduated cylinders used to tranfer initial sample volume to CLLE
Procedures	1. CLLE for 18 $\pm$ 2 hours 2. extract dried using Na_2SO_4 in short stem funnels 3. no apparatus height specification for concentration on water bath 4. sample removed from water bath when volume reaches ~10 mL 5. hexane added directly to K-D body at start of concentration process	1. CLLE for 18-24 hours 2. extract dried using Na_2SO_4 in drying columns 3. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4. sample removed from water bath when volume reaches 1-2 mL 5. solvent exchange via macro K-D with addition of 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TABLE 3

SUMMARY OF METHOD MODIFICATIONS (METHOD 3535, current revision)

TOPIC	KATAHDIN SOP CA-515-07	METHOD 3520, current revision
Apparatus/Materials	1. Horizon SPE-DEX 4790 Automated Extractor System.	1. Empore solid-phase extraction system
Reagents		
Sample preservation/ handling	1. entire contents of 1 L sample bottle transferred to separatory funnel	1. one liter graduated cylinders used to transfer initial sample volume to separatory funnel
Procedures	1. no methanol addition 2. extraction using Horizon SPE-DEX 4790 Automated Extractor System. 3. extract dried using Na_2SO_4 in short stem funnels 4. no apparatus height specification for concentration on water bath 5. sample removed from water bath when volume reaches ~10 mL 6. hexane added directly to K-D body at start of concentration process	1. 5mL methanol added to all samples and blanks 2. extraction using Empore solid-phase extraction system 3. extract dried using Na_2SO_4 in drying columns 4. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 5. sample removed from water bath when volume reaches 1mL 6. solvent exchange via macro K-D with addition of 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

FIGURE 1

EXAMPLE OF LOGBOOK PAGE

PIP
Sep

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - AQUEOUS PESTICIDE/PCB

Extraction Method: (check one)	SW846 3520 (CLLE)		SW846 3510 (SEP) ✓		SW846 3535 (SPE)	
Analytical Method: (check one)	SW846 8081 ✓	SW846 8082 ✓	EPA 608	CLP OLM04.2	CLP OLC2.1	Other:
Standards	Surrogate ID: G606654		Spike ID: G606654		Spike ID: G606663	
Solvents	Solvent Lot # (MeCl2): H40E31		Solvent Lot # (Hexane): H22E15		Solvent Lot # (Acetone): —	
Consumables	Filter Paper Lot # K11672365		Acid Lot # 435026		$NaSO_4$ Lot # 27969003	
Nitrogen Bath Temperature: 37°C		Vial Lot #: 00097808 / 00098146				
Prep Start Time: 1400		Prep End Time: 1500		CLLE Start Time:		CLLE End Time:

Date Extracted	Ext. Init.	Sample ID	Initial Vol. mL	Surr. Vol.	Spike Vol.	Fraction Pest	Fraction PCB	Final Vol. mL	Date Conc.	Tray Location	Initials	Clean-Up GPC	Clean-Up Flor.	Clean-Up Acid Wash	Clean-Up Other	Comments
10/22/09	OB	W470360-1 W470361-1	1000	1mL	NR	✓	✓	10mL	10-23-09	PP591 F4	GN			✓		Rpost=R112304
		W470360-2			1mL					F5						Rpub=R112305
		-3								F6						
		-4	200		NR	✓				F7						PP5923A-4 brought to volume in DI
		W470361-2	1000		1mL		✓			F8				✓		
		-3								F9				✓		
																10-23-09 GN

EX-002 – Revision 1 – 10/15/09

QAEX177 0000010

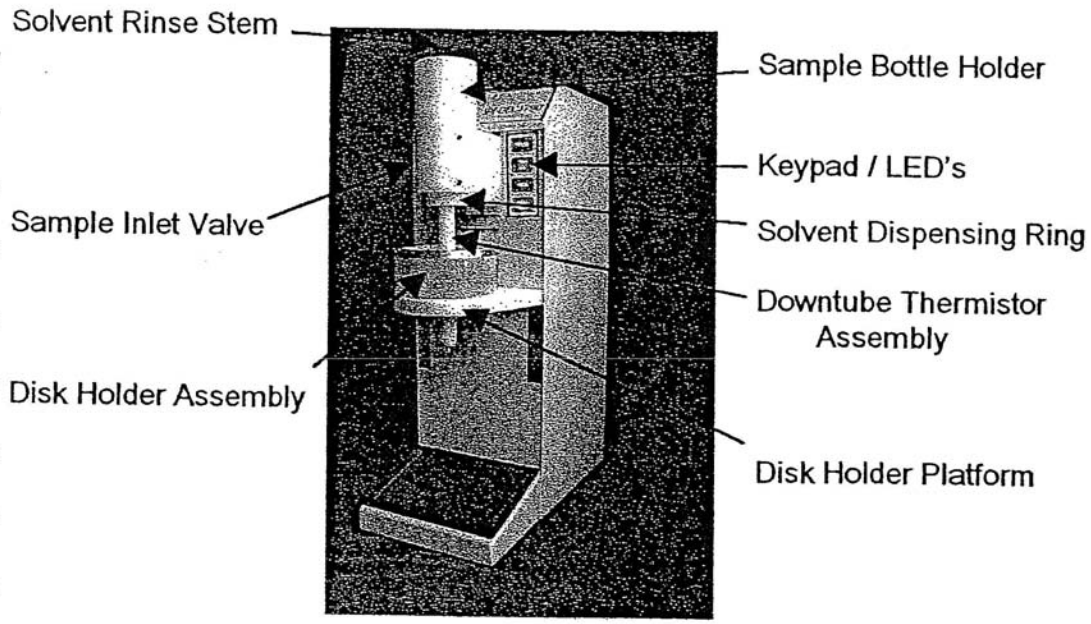
Date Extracted	Ext. Init.	Sample ID	Initial Vol. mL	Surr. Vol.	Spike Vol.	Fraction Pest	Fraction PCB	Final Vol. mL	Date Conc.	Tray Location	Initials	Clean-Up GPC	Clean-Up Flor.	Clean-Up Acid Wash	Clean-Up Other	Comments
10/22/09	OB	SC6328-1	200	1mL	NR	✓		10mL	10-23-09	F10	GN					TCLP extract brought to volume in DI
		SC6340-2								F11						
		-6								F12						
		-8								PP592 A1						
		SC6345-11								A2						
		SC6413-1								A3						
		SC6433-15a	1060				✓			A4				✓		
		-16j	1040							A5						
		SC6457-2L	1060							A6						
		-3L	970							A7						
		-4L	1040							A8						
																10-23-09 GN

EX-002 – Revision 1 – 10/15/09 QAEX177 0000011

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

FIGURE 2

HORIZON AUTOEXTRACTOR SYSTEM DIAGRAM



KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-524
Revision History
Cover Page
Page 1

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS

Prepared By: Mike Thomas Date: 7/98

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: J Benton Date: 11/15/00

QA Officer: Deborah J. Nadeau Date: 11·15·00

General Manager: Dennis P. Hufnagel Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	11.15.00	11/15/00
02	Added definitions to section 1.1. Wording changed or added to clarify sections 5, 6, 8, + 9. New figure	MRC	11.08.04	11.08.04
03	Sect. 7.1.2 - adding the step to rinse forceps also. 7.10 adding condenser temperature and output voltage of variable transformer	LAD	04/06	04/06
04	Added generated waste information. Updated spike list. Added LCSD. Reworded Sect. 7.10 and 7.11 for clarification. Updated Table 1 Replaced Figure 1	LAD	09/07	09/07
05	Changed "N-Lo" waste to "K" waste. Updated Logbook example. Sect. 7 - added wording instructing the recording of consumable lot #'s in Logbook.	LAD	07/08	07/08

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added balance criteria. Changed weight criteria to +/- 0.05g. Minor changes to section 7 to reflect current techniques. Clarified samples being GPC'd are not solvent exchanged into hexane. Updated logbook page. Added CA-108 reference for subsampling information	LAD	08/09	08/09
07	Removed targeting sample weights. Removed decanting samples prior to extraction.	LAD	08/10	08/10

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-524-07**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-524-07**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS.**

Recipient: _____ Date: _____

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure for extracting pesticides/PCBs from solids such as soils, sludges, and wastes using Method 3540. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent.

This method is applicable to the isolation and concentration of water-insoluble and slightly water-soluble organics in preparation for a variety of chromatographic procedures including methods 8081 for pesticides and 8082 for PCB's.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for pesticide/PCB analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training and Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for pesticide/PCB analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to indicate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the “D” waste stream satellite accumulation area nearest the point of generation. Acetone and hexane are considered flammable waste, and should be disposed of in the “O” waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples, used glass wool, and sodium sulfate waste should be disposed of in the soil with organics “I” waste stream

satellite accumulation area nearest the point of generation. Acid waste generated during the cleanup of PCB samples should be disposed of in the "K" satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

2.1 The solid sample is mixed with anhydrous sodium sulfate, placed in a Soxhlet extractor and extracted with methylene chloride.

2.2 The extract is then dried, concentrated, and exchanged into hexane for GC analysis. Sulfuric acid cleanup is performed on extracts for 8082 PCB analysis.

3.0 INTERFERENCES

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

4.1 Soxhlet extractor – 45/50 top joint and 24/40 lower joint.

4.1.1 500 mL flat-bottom boiling flask

4.1.2 Allihn cooling water condenser

4.2 Powder Funnels – 100 mm top diameter, 35 mm stem

4.3 Kuderna-Danish (K-D) apparatus

4.3.1 Concentrator tube - 10-mL

4.3.2 Evaporation flask - 500-mL

4.3.3 Snyder column - Three-ball macro

4.4 Nitrogen evaporation (N-EVAP) apparatus.

4.5 Boiling stones, 12 mesh silicon carbide (carborundum) – pre-purified by Soxhlet extraction in methylene chloride

4.6 Water bath - Heated, with concentric ring cover, capable of temperature control (± 5°C). The bath should be used in a hood.

4.7 Vials - Glass, 4, 12, or 16 mL with Teflon-lined screw caps

4.8 Glass wool (fiberglass) - baked at 400°C for a minimum of 4 hours or overnight.

4.9 Heating mantles - Rheostat controlled.

4.10 Disposable glass Pasteur pipets, 5 ¾”, and bulbs.

4.11 Drying oven - capable of maintaining 105°C for glassware drying.

4.12 Muffle oven – capable of maintaining 400 °C for baking glass wool and organic-free sand.

4.13 Beakers, 250 or 400 mL

4.14 Top-loading balance - capable of weighing to 0.**01** g.

4.15 Spatulas, stainless-steel

4.16 Long forceps, stainless-steel

4.17 Metal clips – for securing Soxhlets to boiling flasks

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

4.18 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

4.19 Gel Permeation Chromatograph (GPC) - J2 Scientific AccuPrep MPS™ with internal UV detection

5.0 REAGENTS

5.1 Sodium sulfate (granular, anhydrous and powdered, anhydrous) (ACS reagent grade), Na_2SO_4. Certified by the manufacturer/vendor as purified by heating at 400°C for 4 hours prior to receipt by the laboratory.

5.2 Methylene chloride - (pesticide grade or equivalent) purchased by lot, evaluated prior to use by concentration of 300 mL to 1 mL followed by GC/MS analysis.

5.3 Acetone and hexane – (pesticide grade or equivalent) purchased by lot, evaluated prior to use by concentration of 300 mL to 1 mL followed by GC/MS and GC analysis.

5.4 Organic-free sand, purified by baking at 400 °C at a minimum of 4 hours or overnight. Method blanks serve as checks on the baked sand.

5.5 Surrogate spiking solution - Prepare a solution of decachlorobiphenyl (DCB) and tetrachloro-meta-xylene (TCMX) at a concentration of 1 ug/mL in acetone.

5.6 Matrix Spike/Lab Control Sample spiking solution

5.6.1 Pesticide spike solution – prepare in pesticide grade methanol containing the analytes listed below at concentrations of 0.5 ug/mL.

4,4'-DDD	Endrin
4,4'-DDE	Endrin Aldehyde
4,4'-DDT	Endrin Ketone
Aldrin	gamma-BHC (Lindane)
alpha-BHC	Heptachlor
beta-BHC	Heptachlor Epoxide
delta-BHC	Methoxychlor
Dieldrin	alpha-Chlordane
Endosulfan I	gamma-chlordane
Endosulfan II	Endrin
Endosulfan Sulfate	Endrin Aldehyde

5.6.2 PCB spike solution – prepare Aroclor 1660 (Aroclor 1016 and 1260) in pesticide grade acetone at a concentration of 5.0 ug/mL each.

5.7 Store the solutions mentioned in sections 5.5 and 5.6 at -10 to -20 °C (±2 °C) in a Teflon sealed container. Solution must be verified by GC/ECD prior to use and must be replaced every 6 months or sooner if degradation is evident.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sediment/soil samples must be collected in a soil jar and must be maintained at 4°C (±2°C).

Holding time for extraction of sediment/soil samples for Method 3540 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

The following information must be recorded in the extraction logbook.

- Extraction method
- Surrogate and spike IDs
- Lot numbers of all solvents, acids and bases, sodium sulfate, filter paper
- Nitrogen evaporation water bath temperature
- pH if applicable
- Sonicator horns tuned
- Extraction and Concentration dates
- Extraction and Concentration analyst
- Sample ID or QC sample ID
- Initial and final volumes or weight
- Surrogate and spike amounts
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)

Samples need to be “swiped” out when removing and “swiped” in when replacing samples in storage locations to maintain the internal chain of custody. Refer to Katahdin SOP, Sample Receipt and Internal Control, current revision, for the proper procedure for removal and return samples.

7.1 Preparing the Soxhlet Extraction Apparatus

7.1.1 Rinse the Soxhlet extractors and 500 mL flat-bottom boiling flasks three times with methylene chloride. Be sure that the solvent rinses through the large vapor tube and smaller siphon tubes of the Soxhlet. Inspect these for tiny cracks. Also rinse the 24/40 lower joint.

7.1.2 Add ~ 250 mLs of methylene chloride to the 500 mL boiling flask. Add several boiling stones. Rinse the stainless steel forceps and pre-baked glass wool with Methylene chloride. Working in a hood, place a plug of the glass wool at the bottom of the Soxhlet so that the siphon tube hole is covered. Insert the 24/40 joint of the Soxhlet extractor into the 500 mL boiling flask and secure with a metal clip. Cover the top of the Soxhlet extractor with a piece of aluminum foil until ready to begin loading the sample.

7.2 Sample Handling

7.2.1 Sediment/soil samples – Do not decant any water layer on a sediment sample. Mix the sample thoroughly with the stainless steel spatula. If the sample container is full to the extent that stirring the sample is impractical, try to remove the “best representative” aliquot from the jar based on color, particle size, moisture, etc. Discard any foreign objects such as sticks, leaves, and rocks.

7.2.2 Gummy, fibrous, or oily materials not amenable to mixing should be cut, shredded, or otherwise reduced in size to allow for maximum exposure of the sample surfaces to the extraction solvent. Materials such as glass, rubber, metal, etc. may not require mixing with powdered sodium sulfate to disperse the sample. Plastic materials must be tested for degradation (melting) in methylene chloride prior to Soxhlet extraction.

7.2.3 Please refer to Katahdin Analytical Services SOP CA-108, current revision, “Basic Laboratory Techniques” for more information of subsampling.

7.3 Weigh out an approximate, greater than 30 g portion of sample into a labeled 400 mL beaker. Record sample weight to nearest 0.01 g in appropriate extraction logbook. Add between 30 to 60 g of powdered sodium sulfate, as required, to produce a “free-flowing” mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula. Keep the spatula in the sample beaker and <u>cover</u> the beaker with aluminum foil. Record sodium sulfate lot in logbook.

7.4 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare method blank, weigh out one greater than 30 g portion of purified sand in a labeled 400 mL beaker. Record sample weights to nearest 0.01 g in

appropriate extraction logbook. Add 60 g sodium sulfate and mix well. (Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.)

7.5 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one greater than 30 g portion of purified sand in a labeled 400 mL beaker. Record sample weights to nearest 0.01 g in appropriate extraction logbook. Add 30 g sodium sulfate and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB LCS's must be prepared (refer to section 5.6). If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.6 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out two approximate, greater than 30g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.01g in appropriate extraction logbook. Add 30 g sodium sulfate to each to produce a free-flowing mixture, and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB MS/MSD pairs must be prepared (refer to section 5.6).

7.7 Once all of the QC and field samples have been weighed and mixed with sodium sulfate, begin adding each to the assembled and appropriately labeled Soxhlet extractors using the stainless steel spatulas. Carefully scrape all of the mixtures from the beaker walls so that no more than 1% remains behind in the beaker. Be careful that none of solid material falls into the extract flask through the large vapor tube.

7.8 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL of the pesticide/PCBs surrogate spiking solution using a 1.0 mL gas tight syringe. Record surrogate spike volume and identification code in the extraction logbook. Thoroughly rinse syringe with solvent between each use.

7.9 To LCS/LCSD and MS/MSD add 1.0 mL of either the pesticide or PCBs matrix spike/LCS spiking solutions using a 1.0 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification codes in the extraction logbook. Thoroughly rinse syringe with solvent between each use.

7.10 Rinse the joints of the Allihn cooling condensers with Methylene Chloride, collecting the waste in a methylene chloride solvent waste container. Place each of the Soxhlet extractors in a heating mantle and lower the Allihn cooling water

condensers into the 45/50 joints of the extractors. The condensers should be set to a temperature of 15ºC. Save the pieces of aluminum foil for covering the Soxhlets when the extraction is complete. Switch on the individual heating mantles and be sure that the Rheostat of the variable transformer is set to 55% of the output voltage. Once the methylene chloride begins to boil and the Soxhlet begins to cycle (solvent will immerse the sample and collect in the Soxhlet until the level reaches that of the small siphon tube and then begin to spill over into the extract flask), re-check the apparatus' for leaks. Allow the samples to extract for 18-24 hours. Be sure the chiller/recirculator temperature is set low enough to provide enough cooling capacity for the number of extractions in the batch.

7.11 When the extraction is complete, allow the extracts to cool before dismantling. Remove the Allihn condenser and replace the aluminum foil on top of the extractor. Move the extractors to a hood and detach the extractor from the extract flask. Tilt each extractor slightly to cause any remaining solvent in the sample chamber to drain through the siphon tube into the extract flask. This will help to cool the extract flask and make the apparatus easier to dismantle. Try to drain as much solvent as possible from the extractor into the flask. **This is done by rinsing a glass tube in methylene chloride and pressing on the sample slightly so that as solvent as possible is drained into the extract flask.** Cover the flask with aluminum foil and store in the interim extract storage refrigerator unless the extracts are to be concentrated the same day.

7.12 Immediately remove the extracted soil/sodium sulfate mixtures from the extractors using a square edge spatula, and dispose of in an appropriate solid waste container. It is important to do this soon after the extractors are dismantled, as the sample mixture will tend to "freeze" into a solid mass in the Soxhlet as the solvent dries.

CONCENTRATION OF THE EXTRACTS

7.13 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add a few boiling chips to the K-D. Insert fluted 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride. Place the assembled K-D's under the funnels. Record the lot numbers of the solvent, sodium sulfate and filter papers in the extraction logbook.

7.14 If samples are to be GPC'd, refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures. Samples that undergo GPC are not solvent exchanged into hexane.

7.15 If samples are not to be GPC'd follow Steps 7.16 through 7.23 to concentrate extracts to final volume of 10.0 mLs

7.16 For a solvent exchange, (for samples not being GPC'd), add approximately 50 mL hexane to funnel and let drain through. Since methylene chloride has a lower boiling point than Hexane, this will result in a final extract in hexane only. Record the lot number of the solvent in the extraction logbook.

7.17 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mLs of methylene chloride and allow to drain.

7.18 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.19 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride, hexane, if exchange is taking place. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride, hexane, if exchange is taking place.

7.20 Reduce the extract in the concentrator tube to approximately 1-2 mL using the nitrogen blow-down apparatus to ensure all methylene chloride has been evaporated. The bath temperature must be no higher than the boiling point of the solvent (39°C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. <u>During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of hexane. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration.</u> Record the temperature of the water in the nitrogen evaporation

water bath in the extraction logbook, also note any problems or extract losses, if they occur.

7.21 Complete quantitative transfer of the extract to a vial by using hexane. Adjust the volume of the hexane extract to 10 mL in either a 12 or 16 mL vial using the appropriate "reference vial" for volume comparison.

7.22 Label the vial with lab sample number, extraction date, matrix and analysis. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extractions logbook the box number and "tray location" of the individual extract vials.

7.23 All sample extracts for 8082 PCB analysis must undergo a sulfuric acid wash (cleanup) prior to analysis, unless it has been GPC'd. All sample extracts for 8081 pesticide analysis should undergo further cleanup using the GPC unless time is a factor. All sample extracts for combined 8081/8082 analyses must be split unless GPC'd. One portion must be acid cleaned for 8082 analysis. The associated method blank must be split and acid-cleaned in the same fashion. PCB LCSs and matrix spikes are acid cleaned also. Pesticide LCSs and matrix spikes are not subjected to further cleanup. Record the lot number of the acid in the extraction logbook. Please refer to Katahdin SOP CA525 (current revision), Extract Cleanup Using Sulfuric Acid, for further instructions.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for <u>each</u> and <u>every</u> item listed below:

- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)
- Each extraction method or level
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for <u>each</u> and <u>every</u> item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples
- Each analysis (pesticide and/or PCB) to be performed

Refer to the current revision of the applicable Katahdin SOP for analysis of Pesticides and PCBs for quality control acceptance criteria.

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the

analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases “qualified” data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Method Detection Limit, Instrument Detection Limit and reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the applicable analytical SOP for other method performance parameters and requirements.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, Method 3540C, SW-846, Third Edition, Updates I, II, IIA, IIB, and III Revised December 1996, US EPA.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

LIST OF TABLES AND FIGURES

Table 1 Summary of Method Modifications
Figure 1 Example of Logbook Page

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

TABLE 1

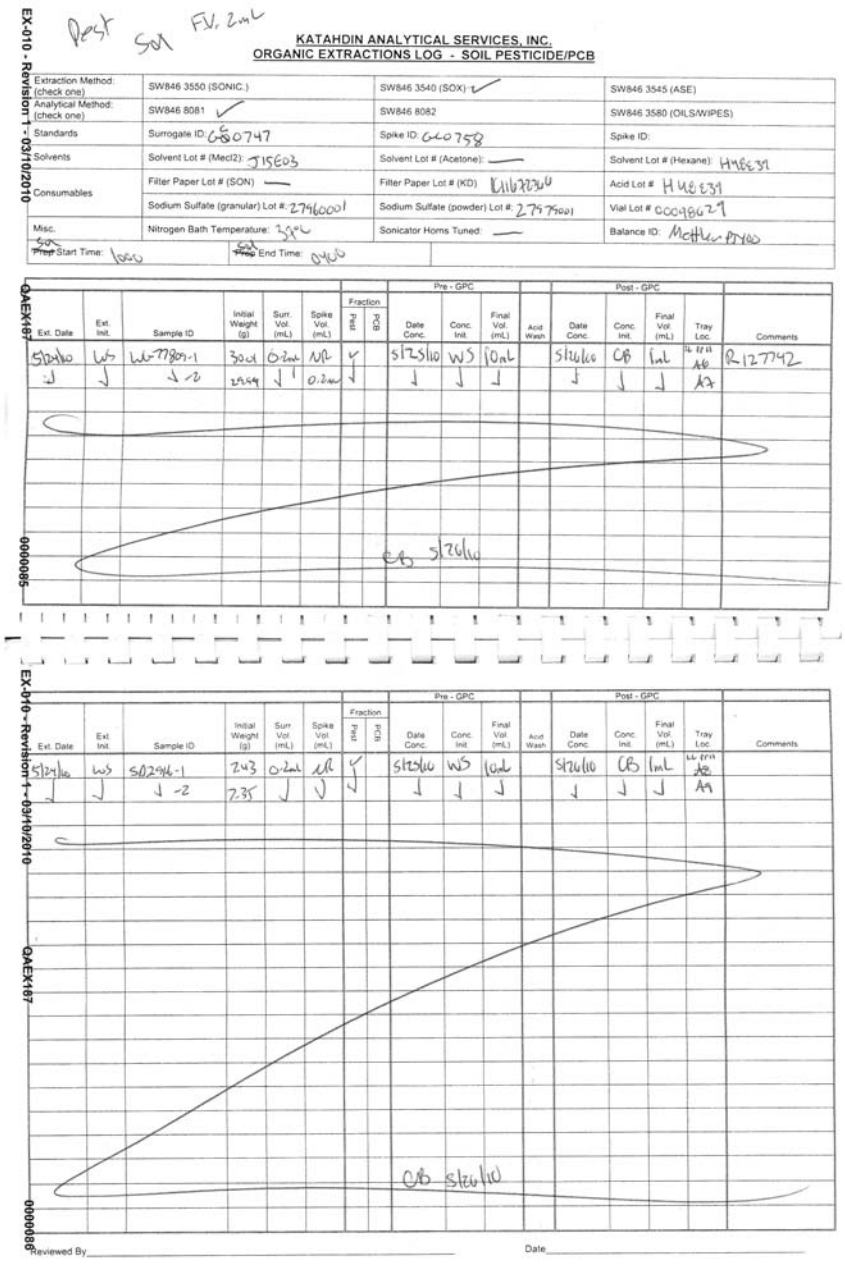
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-524-07	METHOD 3540, current revision
Apparatus/Materials	1. short stem funnels	1. drying columns
Reagents		
Sample preservation/ handling		
Procedures	1. Use 30 grams of sample and 30 grams of sodium sulfate. 2. Use 250 mL of methylene chloride 3. no apparatus height specification for concentration on water bath 4. water bath at 75-85 deg C 5. sample removed from water bath when volume reaches ~6 mL 6. Solvent exchange to hexane is performed using K-D apparatus with addition of approximately 50 mL hexane at the start of concentration process	1. Use 10 grams of sample and 10 grams of sodium sulfate. 2. Use 300 mL of methylene chloride 3. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4. water bath at 80-90 deg C 5. sample removed from water bath when volume reaches 1-2 mL 6. Solvent exchange to hexane is performed using K-D apparatus with addition of approximately 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE



Pest Soil FV. 2mL

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - SOIL PESTICIDE/PCB

Extraction Method: (check one)	SW846 3550 (SONIC.)	SW846 3540 (SOX) ✓	SW846 3545 (ASE)
Analytical Method: (check one)	SW846 8081 ✓	SW846 8082	SW846 3580 (OILS/WIPES)
Standards	Surrogate ID: GC0747	Spike ID: GC0758	Spike ID:
Solvents	Solvent Lot # (Mecl2): J15E03	Solvent Lot # (Acetone): —	Solvent Lot # (Hexane): H46E31
Consumables	Filter Paper Lot # (SON) —	Filter Paper Lot # (KD) K1127736	Acid Lot # H46E31
	Sodium Sulfate (granular) Lot #: 2746001	Sodium Sulfate (powder) Lot #: 2747501	Vial Lot # G0048627
Misc.	Nitrogen Bath Temperature: 39°C	Sonicator Horns Tuned: —	Balance ID: McHlw-PT400
~~Prep~~ Start Time: 1000	~~Prep~~ End Time: 0400		

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Fraction Pest	Fraction PCB	Pre - GPC Date Conc.	Pre - GPC Conc. Init.	Pre - GPC Final Vol. (mL)	Acid Wash	Post - GPC Date Conc.	Post - GPC Conc. Init.	Post - GPC Final Vol. (mL)	Tray Loc.	Comments
5/24/10	WS	WW-77809-1	30.01	0.2mL	NA	✓		5/25/10	WS	10mL		5/26/10	CB	1mL	~~AB~~ A8	R127742
↓	↓	↓ -2	29.99	↓	0.2mL	↓		↓	↓	↓		↓	↓	↓	A7	

CB 5/26/10

QAEX187 EX-010 - Revision 1 - 03/10/2010 0000085

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Fraction Pest	Fraction PCB	Pre - GPC Date Conc.	Pre - GPC Conc. Init.	Pre - GPC Final Vol. (mL)	Acid Wash	Post - GPC Date Conc.	Post - GPC Conc. Init.	Post - GPC Final Vol. (mL)	Tray Loc.	Comments
5/24/10	WS	SO2946-1	7.43	0.2mL	NA	✓		5/25/10	WS	10mL		5/26/10	CB	1mL	~~AB~~ A8	
↓	↓	↓ -2	7.35	↓	↓	↓		↓	↓	↓		↓	↓	↓	A9	

CB 5/26/10

QAEX187 EX-010 - Revision 1 - 03/10/2010 0000086

Reviewed By_____ Date_____

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

Prepared By: Mike Thomas Date: 7/98

Approved By:

Group Supervisor: Michael Thomas Date: 11/15/00

Operations Manager: J C Benton Date: 11/15/00

QA Officer: Deborah J. Nadeau Date: 11.16.00

General Manager: Dennis F. Hufnagel Date: 11/20/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	11.16.00	11/16/00
02	Definitions added to section 1.1. Wording was added or changed to clarify sections 4, 5, 6, 7, 8 + 9. Minor changes throughout. New figures.	MRC	11.09.04	11.09.04
03 LAD 6-26-06	Updated Sect. 7.0 to include SIM. Updated figures 2 and 3 to include current SVOA ~~spikes~~ compounds used. Updated Sect. 5.0 to include all compounds analyzed for. Updated logbook page. minor edits throughout.	LAD	04/06	04/06
04	Added waste generated information. Updated Spikes and Surrogates. Added SIM LCSD and MS/D requirements. Updated Table 1. Added GPC references. Added LCSD after LCS.	LAD	09/07	09/07
05	Updated logbook page. Added adipate compounds to Fig. 2. Added recording of consumable's lot #'s and recording the nitrogen water bath temp. in logbook	LAD	07/08	07/08

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added balance criteria. Changed all weight criteria to +/- 0.05g. Revised section 7 to reflect current techniques. Added SOP CA-108 reference for sub-sampling. Updated logbook example	LAD	08/09	08/09
07	Removed targeting sample weights. Added to weigh out a minimum 30g sample. Removed decanting samples prior to extracting. Added putting solid waste in the "I" waste stream. Updated Logbook page.	LAD	08/10	08/10

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-526-07**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-526-07**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS.**

Recipient: _____ Date: _____

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures and requirements for extracting semivolatile organic compounds from solids such as soils, sludges, and wastes using Method 3540. The Soxhlet extraction process ensures intimate contact the sample matrix with the extraction solvent.

This method is applicable to the isolation and concentration of water-insoluble and slightly water soluble organics in preparation for a variety of chromatographic procedures.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for semivolatile analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training and Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for semivolatile analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that the members of his/her group follow this SOP, to assure that their work is properly documented, and to indicate periodic review of the pertinent logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the “D” waste stream satellite accumulation area nearest the point of generation. Acetone and methanol are considered flammable waste, and should be disposed of in the “O” waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples, used glass wool, and sodium sulfate waste should be disposed of in the soil with organics “I” waste stream satellite

accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

2.1 The solid sample is mixed with anhydrous sodium sulfate, placed in a Soxhlet extractor and extracted with methylene chloride.

2.2 The extract is then dried and concentrated for subsequent 8270 Semivolatile Organics analysis.

3.0 INTERFERENCES

Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause method interferences such as discrete artifacts and/or elevated baselines in the total ion current profiles (TICPs). All of these materials routinely must be demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks. Interferences caused by phthalate esters can pose a major problem in semivolatile organics analysis because many phthalates are also target analytes. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or Teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

4.1 Soxhlet apparatus:

4.1.1 Soxhlet extractor – 45/50 top joint and 24/40 lower joint.

4.1.2 500 mL flat-bottom boiling flask

4.1.3 Allihn cooling water condenser

4.2 Powder Funnels – 100 mm top diameter, 35 mm stem

4.3 Kuderna-Danish (K-D) apparatus

4.3.1 Concentrator tube - 10-mL

4.3.2 Evaporation flask - 500-mL

4.3.3 Snyder column - Three-ball macro

4.4 Nitrogen evaporation (N-EVAP) apparatus.

4.5 Boiling stones, 12 mesh silicon carbide (carborundum) – pre-purified by Soxhlet extraction in methylene chloride

4.6 Water bath - Heated, with concentric ring cover, capable of temperature control (± 5°C). The bath should be used in a hood.

4.7 Vials - Glass, 1.8-mL capacity, with polytetrafluoroethylene (PTFE)-lined septum vials, and 12 mL with Teflon-lined caps for extracts designated for GPC cleanup.

4.8 Glass wool (fiberglass) - baked at 400°C for a minimum of 4 hours or overnight.

4.9 Heating mantles - Rheostat controlled.

4.10 Disposable glass pasteur pipets, 5 ¾” and bulbs.

4.11 Drying oven - capable of maintaining 105°C for glassware drying.

4.12 Muffle oven – capable of maintaining 400 °C for baking glass wool and organic-free sand.

4.13 Beakers, 250 or 400 mL

4.14 Top-loading balance - capable of weighing to 0.**01** g.

4.15 Spatulas, stainless-steel

4.16 Long forceps, stainless-steel

4.17 Metal clips – for securing Soxhlets to boiling flasks

4.18 Filter Paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

5.0 REAGENTS

5.1 Sodium Sulfate - anhydrous powdered and granular crystals, reagent grade, certified by the manufacturer/vendor as purified heating to 400°C prior to receipt by the laboratory.

5.2 Methylene chloride, methanol, and acetone - pesticide residue analysis grade or equivalent. Methylene chloride and acetone are evaluated, by lot, prior to use, by concentration of approximately 400 mL to 1.0 mL followed by GC/MS analysis.

5.3 Organic-free sand, purified by baking at 400 °C. Method blanks serve as checks on the baked sand.

5.4 Base/Neutral and Acid (SVOA) Surrogate Spiking Solution - Surrogate standards are added to all samples and calibration solutions. Prepare a surrogate standard spiking solution that contains the following compounds at the indicated concentrations in acetone.

Compound	Conc.
phenol-$_{d6}$	100 ug/mL
2,4,6-tribromophenol	100 ug/mL
2-fluorophenol	100 ug/mL
nitrobenzene-$_{d5}$	50 ug/mL
p-terphenyl-$_{d14}$	50 ug/mL
2-fluorobiphenyl	50 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5 SIM Surrogate Spiking Solution- Surrogate Standards are added to all samples and calibration solutions. Prepare a surrogate solution that contains the following compounds at a concentration of 2 ug/mL in acetone.

Compound	Conc. ug/mL
Fluorene-d10	2.0 ug/mL
2-Methylnaphthalene-d10	2.0 ug/mL
Pyrene-d10.	2.0 ug/mL
2,4-Dibromophenol	2.0 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.6 Base/Neutral and Acid (SVOA) Lab Control Sample / Matrix Spike Spiking Solution - Prepare a spiking solution in methanol that contains the following mixes listed in Figure 2 at a concentration of 50 ug/ml for the base/neutral compounds and 100 ug/ml for the acid compounds. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.7 Base/Neutral (SIM) Matrix Spike/ Lab Control Sample Spike Solution for SIM-SVOA. Prepare a spiking solution in methanol that contains the compounds listed in Figure 2 at a concentration of 2 ug/mL for base/neutral. Take out 1.0 mL of Base/Neutral and Acid Matrix Spike/Lab Control Spiking Solution for SVOA and dilute it to 25.0 mL in methanol. Store the solution Spiking at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.8 Base/Neutral and Acid (SVOA) Appendix IX Lab Control Sample / Matrix Spike Spiking Solution – Prepare a spiking solution in methanol that contains the compounds listed in Figure 3 at concentrations of 100 ug/ml. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sediment/soil samples must be collected in a soil jar and must be maintained at 4°C (±2°C).

Holding time for extraction of sediment/soil samples for Method 3540 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

7.0 PROCEDURES

The following information must be recorded in the extraction logbook (all that are applicable).

- Extraction method
- Surrogate and spike IDs
- Lot numbers of all solvents, acids and bases, sodium sulfate, filter paper
- Nitrogen evaporation water bath temperature
- pH
- Sonicator horns tuned?
- Extraction and concentration dates
- Extraction and concentration analyst
- Separatory funnel and sonication extraction start and end times.
- CLLE and Soxhlet extraction start and end times, also the prep start and end times.
- Sample ID or QC sample ID
- Initial and final volumes or weight
- Surrogate and spike amounts
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)

All solid samples need to be cleaned up to reduce matrix interferences, time permitting. The cleanup procedure employed is gel permeation chromatography (GPC).

Samples need to be “swiped” out when removing and “swiped” in when replacing samples in storage locations to maintain the internal chain of custody. Refer to Katahdin SOP, Sample Receipt and Internal Control, current revision, for the proper procedure for removal and return samples.

Fill out the sample preparation/extraction log with the necessary information before starting the extraction.

Prerinse all glassware three times with methylene chloride.

7.1 Preparing the Soxhlet Extraction Apparatus

7.1.1 Rinse the Soxhlet extractors and 500 mL flat-bottom boiling flasks three times with methylene chloride. Be sure that the solvent rinses through the large vapor tube and smaller siphon tubes of the Soxhlet. Inspect these for tiny cracks. Also rinse the 24/40 lower joint.

7.1.2 Add ~ 250 mLs of methylene chloride to the 500 mL boiling flask. Add several boiling stones. Using stainless steel forceps and working in a hood,

place a plug of the pre-baked glass wool at the bottom of the Soxhlet so that the siphon tube hole is covered. Insert the 24/40 joint of the Soxhlet extractor into the 500 mL boiling flask and secure with a metal clip. Cover the top of the Soxhlet extractor with a piece of aluminum foil until ready to begin loading the sample. Record the solvent lot number in the extraction logbook.

7.2 Sample Handling

7.2.1 Sediment/soil samples – Do not decant any water layer on a sediment sample. Mix the sample thoroughly with the stainless steel spatula. If the sample container is full to the extent that stirring the sample is impractical, try to remove the "best representative" aliquot from the jar based on color, particle size, moisture, etc. Discard any foreign objects such as sticks, leaves, and rocks.

7.2.2 Gummy, fibrous, or oily materials not amenable to mixing should be cut, shredded, or otherwise reduced in size to allow for maximum exposure of the sample surfaces to the extraction solvent. Materials such as glass, rubber, metal, etc. may not require mixing with powdered sodium sulfate to disperse the sample. Plastic materials must be tested for degradation (melting) in methylene chloride prior to Soxhlet extraction.

7.2.3 Refer to Katahdin SOP CA-108, current revision, "Basic Laboratory Technique" for more information on subsampling.

7.3 The following steps should be performed <u>rapidly</u> <u>to</u> <u>avoid</u> <u>loss</u> <u>of the</u> <u>more</u> <u>volatile</u> <u>extractables</u>. Weigh out an approximate, greater than 30 g portion of sample into a labeled 400-mL beaker. Record sample weight to the nearest 0.01 g in appropriate extraction logbook. Add between 30 g and 60 g of anhydrous powdered sodium sulfate as required to produce a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula. Keep the spatula in the sample beaker and <u>cover</u> the beaker with aluminum foil.

7.4 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare method blank, weigh out one greater than 30 g portion of purified sand in a labeled 400 mL beaker. Add 60 g sodium sulfate and mix well. Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.

7.5 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one greater 30 g portion of

purified sand in a labeled 400 mL beaker. Add 30 g sodium sulfate and mix well. If combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.6 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out two approximate, greater than 30 portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.01 g in appropriate extraction logbook. Add 30 - 60 g sodium sulfate to each to produce a free-flowing mixture, and mix well. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis.

7.7 Once all of the QC and field samples have been weighed and mixed with sodium sulfate, begin adding each to the assembled and appropriately labeled Soxhlet extractors using the stainless steel spatulas. Carefully scrape all of the mixtures from the beaker walls so that no more than 1% remains behind in the beaker. Be careful not to have any of the solid material fall into the extract flask through the large vapor tube.

7.8 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL of the appropriate base/neutral and acid surrogate spiking solution listed below using the pre-rinsed 1.0 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to using it for another spiking solution.

7.8.1 If the request is for SVOA, use the SVOA surrogate solution (sect. 5.4)

7.8.2 If the request is for SIM, use the SIM surrogate solution (sect. 5.5).

7.8.3 If the request is for SIM-SVOA, use both the SIM and SVOA surrogate solutions. NOTE: Separate LCS/LCSD and/or MS/MSD are needed for each analysis and should be spiked with the appropriate surrogate.

7.9 To the LCS/LCSD and the MS/MSD add 1.0 mL of the appropriate base/neutral and acid (SVOA) matrix spike/LCS spiking solution listed below using a 1.0 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent when spiking is completed.

7.9.1 If the request is for SVOA, add 1 mL of the SVOA spiking solution (sect. 5.6).

7.9.2 If the request is for SIM, add 1 mL of the SIM Spiking solution (sect. 5.7).

7.9.3 If the request is for SVOA/SIM, add 1 mL of the SVOA spiking solution and 1 mL of the SIM Spiking solution to appropriate LCS/LCSD and/or MS/MSD. (sect's 5.6 and 5.7).

7.9.4 If the request is for SVOA Appendix IX, add 1 mL of the SVOA Appendix IX spiking solution and 1 mL of the SVOA spiking solution (sect's 5.6 and 5.8).

7.10 Place each of the Soxhlet extractors in a heating mantle and lower the Allihn cooling water condensers into the 45/50 joints of the extractors. Save the pieces of aluminum foil for covering the Soxhlets when the extraction is complete. Switch on the individual heating mantles and be sure that the Rheostat of the variable transformer is set to 55-60% of the output voltage. Once the methylene chloride begins to boil and the Soxhlet begins to cycle (solvent will immerse the sample and collect in the Soxhlet until the level reaches that of the small siphon tube and then begin to spill over into the extract flask), re-check the apparatus' for leaks. Allow the samples to extract for 18-24 hours. Be sure the chiller/recirculator temperature is set low enough to provide enough cooling capacity for the number of extractions in the batch.

7.11 When the extraction is complete, allow the extracts to cool before dismantling. Tilt each extractor slightly to cause any remaining solvent in the sample chamber to drain through the siphon tube into the extract flask. This will help to cool the extract flask and make the apparatus easier to dismantle. Remove the Allihn condenser and replace the aluminum foil on top of the extractor. Move the extractors to a hood and detach the extractor from the extract flask. Try to drain as much solvent as possible from the extractor into the flask. **This is done by rinsing a glass tube in methylene chloride and pressing on the sample slightly so that as solvent as possible is drained into the extract flask.** Cover the flask with aluminum foil and store in the interim extract storage refrigerator unless the extracts are to be concentrated the same day.

7.12 Immediately remove the extracted soil/sodium sulfate mixtures from the extractors using a square edge spatula, and dispose of in an appropriate solid waste container. It is important to do this soon after the extractors are dismantled, as the sample mixture will tend to "freeze" into a solid mass in the Soxhlet as the solvent dries. Put the solid waste in the "I" waste stream.

CONCENTRATION OF EXTRACTS

7.13 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add two boiling chips to the K-D. Insert 18.5 cm filter

papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride. Place the assembled K-D's under the funnels. Record the filter paper and sodium sulfate lot numbers in the extraction logbook.

7.14 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mLs of methylene chloride and allow draining.

7.15 All samples should go through GPC cleanup except if time does not permit. Refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures.

7.16 If samples are not to be GPC'd, when time does not permit, follow Steps 7.17 through 7.22 to concentrate extracts to final volume of 1 mL.

7.17 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.18 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 4-6 mL, remove the K-D from the water bath. **Do not allow the evaporator to go dry. If the sample extract does go dry, re-extraction must occur immediately.** Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.19 Reduce the extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39°C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath

as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Record the temperature of the water in the nitrogen evaporation water bath in the extraction logbook, also note any problems or extract losses, if they occur.

7.20 When the apparent volume reaches slightly less than 1 mL, remove the concentrator tube and allow it to cool.

7.21 Complete the quantitative transfer of the extract to a 1.8 mL vial by using methylene chloride. Adjust the volume of the methylene chloride extract to 1.0 mL using the 1.8 mL reference vial for volume comparison.

7.22 Label the vial with lab sample number, extraction date, matrix and analysis. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extractions logbook the box number and “tray location” of the individual extract vials.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)
- Each extraction method or level
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

Refer to the current revision of the applicable Katahdin SOP for analysis of extractable semivolatile organics for quality control acceptance criteria.

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the applicable analytical SOP for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, Method 3540C, SW-846, Third Edition, Updates I, II, IIA, IIB, and III Revised December 1996, US EPA.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

LIST OF TABLES AND FIGURES

Table 1 Summary of Method Modifications
Figure 1 Example of Logbook Page
Figure 2 LCS/Matrix Spike Component List
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TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

TABLE 1

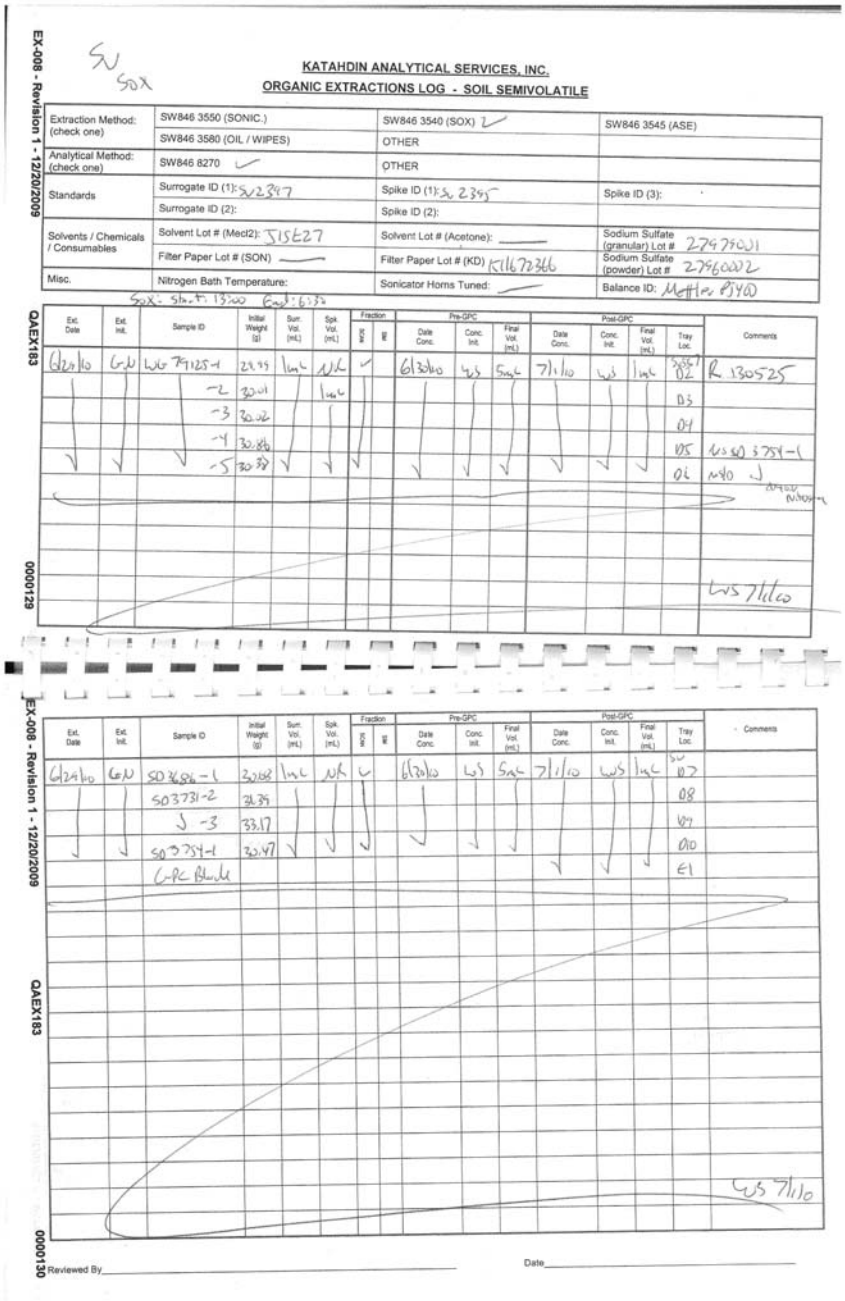
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-526-07	METHOD 3540, current revision
Apparatus/Materials	1. short stem funnels	2. drying columns
Reagents		
Sample preservation/ handling		
Procedures	1. Use 30 grams of sample and 30 grams of sodium sulfate 2. Place a plug of glass wool in soxhlet then add sample 3. Use 250 mL of methylene chloride for extraction 4. Extract the sample for 18 - 24 hours 5. Extract dried using Na_2SO_4 in short stem funnels 6. Rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride then rinse the sodium sulfate with ~ 15 mLs of methylene chloride to complete a quantitative transfer 7. no apparatus height specification for concentration on water bath 8. Water bath at 75-85 deg C 9. Sample removed from water bath when volume reaches ~6 mL	1. Use 10 grams of sample and 10 grams of sodium sulfate. 2. Place sample between 2 plugs of glass wool 3. Use 300 mL of methylene chloride for extraction 4. Extract the sample for 16 - 24 hours at 4 - 6 cycles/hour 5. Extract dried using Na_2SO_4 in drying columns 6. Wash the extractor flask and sodium sulfate column with 100 to 125 mL of extraction solvent to complete the quantitative transfer 7. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-20min 8. Water bath at 15-20 deg C above solvent boiling point 9. Sample removed from water bath when volume reaches 1-2 mL
QC - Spikes	1. Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1. Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1. Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1. Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - Accuracy/Precision		
QC – MDL		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE



EX-008 - Revision 1 - 12/20/2009

SV
SOX

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - SOIL SEMIVOLATILE

Extraction Method: (check one)	SW846 3550 (SONIC.)	SW846 3540 (SOX) ✓	SW846 3545 (ASE)
	SW846 3580 (OIL / WIPES)	OTHER	
Analytical Method: (check one)	SW846 8270 ✓	OTHER	
Standards	Surrogate ID (1): SV2397	Spike ID (1): SV 2395	Spike ID (3):
	Surrogate ID (2):	Spike ID (2):	
Solvents / Chemicals / Consumables	Solvent Lot # (MeCl2): J15E27	Solvent Lot # (Acetone): ___	Sodium Sulfate (granular) Lot # 27975001
	Filter Paper Lot # (SON) ___	Filter Paper Lot # (KD) K1167366	Sodium Sulfate (powder) Lot # 27960002
Misc.	Nitrogen Bath Temperature:	Sonicator Horns Tuned: ___	Balance ID: Mettler B340

SOX: Start: 1300 End: 6:30

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spk. Vol. (mL)	Fraction SON	Fraction WE	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	Tray Loc.	Comments
6/29/10	GN	LW 791251-1	29.99	1mL	N/A	✓		6/30/10	LWS	5mL	7/1/10	LWS	1mL	SV 02	R 130525
		-2	30.01		1mL									03	
		-3	30.02											04	
		-4	30.06											05	LWS 0 3754-1
		-5	30.07											06	MB ✓
															07/01/10
															LWS 7/1/10

0000129

EX-008 - Revision 1 - 12/20/2009

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spk. Vol. (mL)	Fraction SON	Fraction WE	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	Tray Loc.	Comments
6/29/10	GN	SO 3686-1	30.08	1mL	N/A	✓		6/30/10	LWS	5mL	7/1/10	LWS	1mL	SV 07	
		SO 3731-2	32.39											08	
		✓ -3	33.17											09	
		SO 3754-1	30.47	✓	✓	✓		✓	✓	✓	✓	✓	✓	010	
		GPC Blank												011	
															LWS 7/1/10

QAEX183

0000130

Reviewed By_____ Date_____

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

FIGURE 2

LCS/MATRIX SPIKE COMPONENT LIST

BASE/NEUTRALS	
1-Methylnaphthalene	Bis (2-chloroethoxy) methane
1,1-Biphenyl	Bis (2-chloroethyl) ether
1,2,4-Trichlorobenzene	Bis (2-Chloroisopropyl) ether)
1,2-Dichlorobenzene	Bis (2-ethylhexyl) adipate
1,3-Dichlorobenzene	Bis (2-ethylhexyl) phthalate
1,4-Dichlorobenzene	Butylbenzyl phthalate
1,4-Dioxane	Caprolactam
2,4-Dinitrotoluene	Carbazole
2,6-Dinitrotoluene	Chrysene
2-Chloronaphthalene	Dibenz (a, h) anthracene
2-Methylnaphthalene	Dibenzofuran
2-Nitroaniline	Diethyl adipate
3,3'-Dichlorobenzidine	Diethyl phthalate
3-Nitroaniline	Dimethyl phthalate
4-Bromophenylphenyl ether	Di-n-butylphthalate
4-Chloroaniline	Di-n-octyl phthalate
4-Chlorophenylphenyl ether	Fluoranthene
4-Nitroaniline	Fluorene
Acenaphthene	Hexachlorobenzene
Acenaphthylene	Hexachlorobutadiene
Acetophenone	Hexachlorocyclopentadiene
Aniline	Hexachloroethane
Anthracene	Indeno (1,2,3-cd) pyrene
Atrazine	Isophorone
Azobenzene	Naphthalene
Benzaldehyde	Nitrobenzene
Benzidine	N-Nitrosodimethylamine
Benzo (a) Anthracene	N-Nitroso-di-n-propylamine
Benzo (a) pyrene	N-Nitrosodiphenylamine
Benzo (b) fluoranthene	Phenanthrene
Benzo (ghi) perylene	p-toluidine
Benzo (k) fluoranthene	Pyrene
Benzyl alcohol	Pyridine

ACIDS		
2, 3, 4, 6-Tetrachlorophenol	2-Chlorophenol	Benzoic acid
2,4,5-Trichlorophenol	2-Methylphenol	Ethyl methanesulfonate
2,4,6-Trichlorophenol	2-Nitrophenol	Methyl methanesulfonate
2,4-Dichlorophenol	4,6-Dinitro-2-methylphenol	Pentachlorophenol
2,4-Dimethylphenol	4-Chloro-3-methylphenol	Phenol
2,4-Dinitrophenol	4-Methylphenol	
2,6-Dichlorophenol	4-Nitrophenol	

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

FIGURE 3

APPENDIX IX LCS/MATRIX SPIKE COMPONENT LIST

1,2,4,5-Tetrachlorobenzene	Hexachloropropene
1,3,5-Trinitrobenzene	Isodrin
1,4-Naphthoquinone	Isosafrole
1-Chloronaphthalene	Kepone
1-Naphthylamine	m-Dinitrobenzene
2,4-D	Methapyrilene
2-Acetyl aminofluorene	Methyl parathion
2-Naphthylamine	n-Nitrosodiethylamine
2-Picoline	n-Nitrosodi-n-butylamine
3,3-Dimethylbenzidine	n-Nitrosomethylethylamine
3-Methylcholanthrene	n-Nitrosomorpholine
4-Aminobiphenyl	n-Nitrosopyrrolidine
4-Nitroquinoline-1-oxide	n-Nitrotrosopiperidine
5-Nitro-o-toluidine	O,O,O-Triethyl phosphorothioate
7,12-Dimethylbenz(a)anthracene	o-Toluidine
a,a-Dimethylphenethylamine	Parathion
Acetophenone	p-Dimethylaminoazobenzene
Aramite	Pentachlorobenzene
Chlorobenzilate	Pentachloronitriobenzene
Diallate	Phenacetin
Dibenz(a,j)acridine	Phorate
Dimethoate	p-Phenylenediamine
Dinoseb	Pronamide
Diphenylamine	Safrole
Disulfoton	Silvex (2,4,5-TP)
Famphur	Sulfotep
Hexachlorophene	Thionazin

TITLE: **PREPARATION OF SEDIMENT/SOIL AND TISSUE SAMPLES BY ACCELERATED SOLVENT EXTRACTION USING METHOD 3545 FOR SUBSEQUENT EXTRACTABLE PESTICIDE and PCB ANALYSIS**

Prepared By: Galen Nickerson Date: 6-23-06

Approved By:

Department Manager: [signature] Date: 6-23-06

Operations Manager: Deborah J. Radeau Date: 6·23·06

QA Officer: Leslie Dimond Date: 6-23-06

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Sect. 1.4 - Added solvent and acid waste streams. Changed solvent in sect. 7.4.2 from hexane to MeCl2. Updated logbook example. Added recording of solvent, Na2SO4, hydromix, filterpaper Lot #'s as well as the temperature of the Na water bath in the logbook.	LAD	07/08	07/08
02	Updated section 4 with current materials. Updated section 7 with current techniques. Changed when Hexane is added during the solvent exchange process. Changed all weights to record to 0.05g. Updated logbook example. Added CA-108 reference for subsampling.	LAD	08/09	08/09
03	Sect. 7.2 - Removed decanting samples. Sect. 7.3 - Changed weight to approximate, greater than 15g and to record weight to 0.01g. Sect. 7.4.3 - changed rinse solvent to only methylene Chloride and removed prerinsing the sodium sulfate crystals.	LAD	12/10	12/10

TITLE: **PREPARATION OF SEDIMENT/SOIL AND TISSUE SAMPLES BY ACCELERATED SOLVENT EXTRACTION USING METHOD 3545 FOR SUBSEQUENT EXTRACTABLE PESTICIDE and PCB ANALYSIS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy _____ of document **SOP CA-537-03,** titled **Preparation of Sediment/Soil and Tissue Samples by Accelerated Solvent Extraction Using Method 3545 for Subsequent Extractable Pesticide and PCB Analysis**.

Recipient: _____Date:_____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy _____ of document **SOP CA-537-03,** titled **Preparation of Sediment/Soil and Tissue Samples by Accelerated Solvent Extraction Using Method 3545 for Subsequent Extractable Pesticide and PCB Analysis**.

Recipient: _____Date:_____

TITLE: **PREPARATION OF SEDIMENT/SOIL AND TISSUE SAMPLES BY ACCELERATED SOLVENT EXTRACTION USING METHOD 3545 FOR SUBSEQUENT EXTRACTABLE PESTICIDE and PCB ANALYSIS**

1.0 SCOPE AND APPLICATION

Method 3545 is a procedure for extracting water insoluble or slightly water soluble semivolatile organic compounds from soils, clays, sediments, sludges, waste solids and tissues samples. This Pressurized Fluid Extraction (PFE) method uses elevated temperature (100°C or 175°C) and pressure (1500 - 2000 psi) to achieve analyte recoveries equivalent to those from Soxhlet extraction, using less solvent and taking significantly less time than the Soxhlet procedure.

This method is applicable to the extraction of organochlorine pesticides and PCBs. Organochlorine pesticides and PCBs may then be analyzed by a variety of chromatographic procedures.

1.1 Definitions

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

TITLE: **PREPARATION OF SEDIMENT/SOIL AND TISSUE SAMPLES BY ACCELERATED SOLVENT EXTRACTION USING METHOD 3545 FOR SUBSEQUENT EXTRACTABLE PESTICIDE and PCB ANALYSIS**

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for pesticide and PCB analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training".

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for pesticide and PCB analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that the members of his/her group follow this SOP, to assure that their work is properly documented, and to indicate periodic review of the pertinent logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves, and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their department manager, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, "Sample Disposal,"

current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the "D" waste stream satellite accumulation area nearest the point of generation. Acetone and hexane are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples, used glass wool, and sodium sulfate waste should be disposed of in the soil with organics "I" waste stream satellite accumulation area nearest the point of generation. Acid waste generated during the cleanup of PCB samples should be disposed of in the "K" satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

Samples are prepared for extraction by weighing out a specific quantity of sample and mixing said quantity with pelletized diatomaceous earth (Hydromatrix) to remove moisture. The sample is then loaded into an extraction cell and placed on the Accelerated Solvent Extractor (ASE).

The extraction cell containing the sample is heated to the analyte-specific extraction temperature (see section 7.0), pressurized with hexane and extracted for a period of time outlined in section 7.0.

The solvent is collected from the extraction cells into 60 mL vials and allowed to cool. The solvent is then concentrated and, as needed, exchanged into solvent compatible with the cleanup or determinative step being employed.

3.0 INTERFERENCES

Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause method interferences such as discrete artifacts and/or elevated baselines in the total ion current profiles (TICPs). All of these materials routinely must be demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks. Interferences caused by phthalate esters can pose a major problem in semivolatile organics analysis because many phthalates are also target analytes. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain

phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or Teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis. Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

4.1 Pressurized fluid extraction device: Dionex Accelerated Solvent Extractor, Model number ASE 200 with appropriately sized extraction cells. The cells will accommodate 15 g of sample and are made of stainless steel which is capable of withstanding the pressure requirements necessary for this procedure.

4.2 Analytical Balance: Mettler PJ4000 balance capable of weighing to 0.01 g

4.3 Vials for the collection of extracts: Dionex 60 ml pre-cleaned, open to screw cap with PTFE-lined silicone septum.

4.4 Filter disk for extraction vessels: Dionex 1.91 cm, Type D28.

4.5 400 ml beakers

4.6 Ottawa Sand (Fisher P/N 523-3)

4.7 Kuderna-Danish (K-D) apparatus

4.7.1 Concentrator tube - 10-mL

4.7.2 Evaporation flask - 500-mL

4.7.3 Snyder column - Three-ball macro

4.8 Nitrogen evaporation (N-EVAP) apparatus.

4.9 Stainless steel spatula.

4.10 Porcelain mortar and pestle.

4.11 Boiling stones, 12 mesh silicon carbide (carborundum) – pre-purified by Soxhlet extraction in methylene chloride.

4.12 Vials - Glass, 12 mL and 4 mL capacity

4.13 500 ul syringes

4.14 Funnel powder, glass.

4.15 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

4.16 Water bath or steam bath capable of maintaining a temperature of at least 85°C.

5.0 REAGENTS

5.1 Aluminum oxide (Alumina, acid), Brockman activity I, 60-325 mesh

5.2 Drying agents

5.2.1 Pelletized diatomaceous earth (Hydromatrix) - Fisher P/N.

5.2.2 Sodium sulfate crystals, Na_2SO_4.

5.3 Extraction solvents

5.3.2 Methylene Chloride - Pesticide grade or equivalent. Lot must be verified by concentrating ≈ 300 mL to 1 mL and evaluating by both GC/MS and GC/FID analyses.

5.3.3 Hexane - Pesticide grade or equivalent. Lot must be verified by concentrating 200-300 mLs to 1 mL followed by GC/ECD analysis.

5.3.4 Acetone – Pesticide grade or equivalent. Lot must be verified by concentrating ≈ 300 mL to 1 mL followed by GC/MS, GC/ECD, and GC/FID analyses.

5.3.5 Organochlorine pesticides and PCBs may be extracted with acetone/methylene chloride (1:1,v/v) or hexane (100%).

5.3.6 Other solvent systems may be employed, provided that the analyst can demonstrate adequate performance for the analytes of interest in the sample matrix

CAUTION: For best results with very wet samples (e.g., ≥ 30% moisture), reduce or eliminate the quantity of hydrophilic solvent used.

5.4 Pre-purified nitrogen is used to purge and/or pressurize the extraction cell.

5.5 Spiking Solutions

5.5.1 Pesticide/PCB surrogate spiking solution - Prepare a solution of decachlorbiphenyl (DCB) and tetrachloro-meta-xylene (TCMX) at a concentration of 1 ug/mL in acetone. Store the solution at –10 to -20 °in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

5.5.2 Pesticide/PCB matrix spike/Lab control sample spiking solution - Prepare separate spiking solutions for Pesticides and one for PCBs in pesticide grade methanol and acetone, respectively, that contain all target analytes listed below:

Pesticide Spiking Solution

Analyte	ug/mL	Analyte	ug/mL
4,4'-DDD	0.5	Endosulfan I	0.5
4,4'-DDE	0.5	Endosulfan II	0.5
4,4'-DDT	0.5	Endosulfan Sulfate	0.5
Aldrin	0.5	Endrin	0.5
alpha-BHC	0.5	Endrin Aldehyde	0.5
beta-BHC	0.5	Endrin Ketone	0.5
delta-BHC	0.5	gamma-BHC (Lindane)	0.5
Dieldrin	0.5	Heptachlor	0.5
alpha-Chlordane	0.5	Heptachlor Epoxide	0.5
gamma-Chlordane	0.5	Methoxychlor	0.5

PCB Spiking Solution

Analyte	ug/mL
Aroclors 1016/1260	5.0

Store the solution at –10 to -20 °C in a Teflon sealed container. The solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Holding time for extraction of sediment/soil and tissue samples for Method 3545 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

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Store all extracts at 4°C (± 2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

Follow the proper procedures for maintaining Internal Chain of Custodies for samples when removing and replacing samples in storage locations. This procedure is described in KAS SOP SD-902, “Sample Receipt and Internal Control”, current revision.

The following information must be recorded in the extraction logbook (all that are applicable).

- Extraction method
- Surrogate and spike IDs
- Lot numbers of all solvents, acids and bases, sodium sulfate, filter paper
- Nitrogen evaporation water bath temperature
- pH
- Sonicator horns tuned?
- Extraction and concentration dates
- Extraction and concentration analyst
- Separatory funnel and sonication extraction start and end times.
- CLLE and Soxhlett extraction start and end times, also the prep start and end times.
- Sample ID or QC sample ID
- Initial and final volumes or weight
- Surrogate and spike amounts
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)

Rinse all glassware three times with methylene chloride.

7.1 Preparing the Accelerated Solvent Extractor

7.1.1 Before extractions can begin, the system must first go through its daily maintenance checks. To begin, turn on the system and nitrogen gas tank. Check the system pressure to ensure that the solvent pressure is at 10 psi, air is at 50 psi, and compression is at 130 psi. Next, check the solvent bottle and fill with the appropriate solvent if necessary. At this time the rinse and waste vials should be emptied and four 60 ml vials need to be placed in the rinse positions of the sample collection carousel. Record the solvent lot number in the extraction logbook.

7.1.2 The system must now be rinsed before the analysis can begin. Press the rinse button on the touch pad. The system will immediately begin its rinse cycle. This rinse procedure should be repeated at least three times before any samples are extracted to ensure that all contaminants are removed from the system.

7.1.3 To prepare extraction cells, disassemble the ASE extractor cells and clean each piece with soapy water. Next rinse each piece with acetone to ensure that all water is removed. Next, rinse the body and two screw caps three times with methylene chloride. Be sure you are using a solvent whose lot has been checked. Inspect each screw cap to ensure that the o-rings are not crushed and in need of replacement. O-rings should be replaced after approximately 50 extractions to ensure proper fit. Cell bodies should also be checked for nicks and dings at each end. If any dings are present the cell should be placed out of rotation until it can be repaired. If any instrument maintenance is necessary record the maintenance performed in the instrument maintenance logbook.

7.1.4 Once the extraction cells have been rinsed replace the bottom cell screw cap. The bottom of the cell body is the end that is imprinted with Dionex. Next, place 2 19.8 mm filter disks into the top of the cell body and push it to the bottom of the cell with the plunger that was supplied with the system. Record the lot numbers of the filter disks in the extraction logbook.

If extracting pesticides/PCBs from tissue samples weigh out 5.00 ±1.00 g of aluminum oxide (alumina, acid) into cell and place a second filter on the top of alumina. Avoiding matrix affect interference for tissue sample, the aluminum oxide is used to remove lipid. Record the lot numbers of the aluminum oxide and filters in the extraction logbook.

7.1.5 Label each extraction cell and clean, rinsed, 60 ml collection vial with the appropriate sample ID with a black marker.

7.2 Sample preparation

7.2.1 Sediment/soil samples – Do not decant or discard any water layer on a sediment sample. Mix the sample thoroughly with the stainless steel spatula. If the sample container is full to the extent that stirring the sample is impractical, try to remove the “best representative” aliquot from the jar based on color, particle size, moisture, etc. Discard any foreign objects such as sticks, leaves, and rocks.

7.2.2 Gummy, fibrous, or oily materials not amenable to grinding should be cut, shredded, or otherwise reduced in size to allow mixing and maximum exposure of the sample surfaces for the extraction. Materials such as glass, rubber, metal, etc. may not require mixing with Hydromatrix to disperse the sample. Plastic materials must be tested for degradation (melting) in methylene chloride prior to ASE extraction.

7.2.3 Tissue samples are blended prior to handling in extraction laboratory.

7.2.4 Refer to Katahdin Analytical Services SOP CA-108, current revision, “Basic Laboratory Technique” for more information regarding subsampling.

7.3 Sample Handling

The following steps should be performed rapidly to avoid loss of the more volatile extractables.

7.3.1 Weigh out an approximate, greater than 15 g portion of sample into a 400 ml beaker. For tissue sample, weigh out an approximate, greater than 5 g portion of sample into a 400 ml beaker. Record sample weight to the nearest 0.01g in the appropriate extraction logbook.

7.3.2 Add enough Hydromatrix to the sample to produce a “free-flowing” mixture, around 5g. For tissue sample, pulverize the mixture (sample-hydromatrix) using the porcelain mortar-pestle apparatus. At this point don’t add an excessive amount because more Hydromatrix can be added later in the procedure. This ensures that the sample will properly fit in the extraction cell. The amount of Hydromatrix will depend upon the moisture content of the sample (e.g., low moisture content will require less Hydromatrix). Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil. Record the Hydromatrix lot number in the extraction logbook.

7.3.3 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare a method blank, weigh out an approximate, greater than 5 g portion of Ottawa sand for tissue sample batch or an approximate, greater than 15 g portion for sediment/soil sample batch into a 400 ml beaker. Record the weight to the nearest 0.01 g in the appropriate extraction logbook. Add enough Hydromatrix to the sand to produce a “free-flowing” mixture. At this point don’t add an excessive amount because more Hydromatrix can be added later in the procedure. This ensures that the sample will properly fit in the extraction cell. Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.

7.3.4 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. If no MS/MSD is to be prepared a laboratory control sample duplicate must be prepared. To prepare an LCS (and LCSD), weigh out an approximate, greater than 15g portion of Ottawa sand into a 400 ml beaker; for tissue sample, weigh out an approximate, greater than 5 g of Ottawa sand into a 400 mL beaker. Record the weight to the nearest 0. 01 g in the appropriate extraction logbook. Add enough Hydromatrix, approximately 5g to the sand to produce a "free-flowing" mixture. At this point don't add an excessive amount because more Hydromatrix can be added later in the procedure. This ensures that the sample will properly fit in the extraction cell. Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil. **If extracting both pesticide and PCB separate LCS/D and MS/D need to be prepared.**

7.3.5 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out an approximate, greater than 15 g portion of designee sample into a 400 ml beaker. For tissue sample, weigh out an approximate, greater than 5 g portion of designee sample into a 400 mL beaker. Record the weight to the nearest 0.01g in the appropriate extraction logbook. Add enough Hydromatrix approximately 5g to the sand to produce a "free-flowing" mixture. At this point don't add an excessive amount because more Hydromatrix can be added later in the procedure. This ensures that the sample will properly fit in the extraction cell. Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.

7.3.6 Once all the QC and field samples have been weighed and mixed with a portion of Hydromatrix, find the appropriately labeled extraction cell and place the cell funnel, rinsed three times in methylene chloride, on the open end. This funnel was supplied with the system. Pour the contents of the beaker into the extraction cell. Carefully scrape all the mixtures from the beaker walls so that no more than 1% remains behind in the beaker. If there is head space remaining in the extraction cell fill the remaining space with more Hydromatrix by pouring the sample back into the beaker and mixing the new Hydromatrix into the sample. This will minimize the amount of solvent used. Again, pour the sample back into the same extraction cell and cover with a screw cap. Rinse the cell funnel with extraction solvent between each sample.

7.3.7 To all tissue samples, method blank, LCS, and MS/MSD add 0.2 mL of Pest/PCB Surrogate spiking solution using a pre-rinsed 500 uL gas tight syringe. To all sediment/soil samples and QC being extracted add 0.5 mL of

Pest/PCB surrogate spiking solution using a pre-rinsed 0.5 mL gas-tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent when spiking is complete. To LCS and MS add 0.5 mL of appropriate spike solution to each.

7.3.8 Replace all screw caps and ensure that each is properly tightened. Place each extraction cell into the ASE sample carousel. Place the corresponding collection vial into the appropriate location on the sample collection carousel. Be certain that the cell and vial are in the same number position of the carousel. In order to avoid a possible carryover, samples should be placed in order of their cleanness.

7.3.9 Press the menu button on the ASE touch pad. Choose option number one and press enter. Select method number one for Pesticide and PCB analysis, press enter. Press start on the touch pad. The extraction process will begin and takes approximately 14 minutes per sample to complete.

NOTE: Recommended extraction conditions:

<u>For Pest/PCB (saved in the instrument as method one)</u>
Oven temperature: 100°C
Pressure: 1500 - 2000 psi
Static time: 5 min (after 5 min pre-heat equilibration)
Flush volume: 60% of the cell volume
Nitrogen purge: 90 sec at 150 psi (purge time may be extended for larger cells)
Static Cycles: 1

Optimize the conditions, as needed, according to the manufacturer's instructions. In general, the pressure is not a critical parameter, as the purpose of pressurizing the extraction cell is to prevent the solvent from boiling at the extraction temperature and to ensure that the solvent remains in intimate contact with the sample. Any pressure in the range of 1500 - 2000 psi should suffice.

Once established, the same pressure should be used for all samples extracted for the same analysis type.

As stated above, the recommended conditions have been saved as methods on the instrument. If it becomes necessary to re-program conditions, follow the above guidelines.

7.3.10 Once the extraction process is complete and the instrument is idle remove the sample extracts and label with appropriate sticker. They are now ready

to be stored in the interim extract storage refrigerator unless the samples are to be concentrated the same day.

7.3.11 Remove the extraction cells from the ASE and empty their contents into the appropriate solid waste container.

7.3.12 The extract is now ready for concentration, cleanup, or analysis, depending on the extent of interferents and the determinative method to be employed. Certain cleanup and/or determinative methods may require a solvent exchange prior to cleanup and/or sample analysis.

7.4 Concentration of Extracts

7.4.1 If samples are to be GPC'd, refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures.

7.4.2 If samples are not to be GPC'd follow steps 7.4.3 through 7.4.9 to concentrate extracts to final volume of 5.0 mLs for soil/sediment samples and 2.0 mL for tissue samples

7.4.3 Before assembling, rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride. Add two boiling chips to the K-D. Insert 18.5 cm filter papers into short stem powder funnels and add ≈ 2 inches of sodium sulfate crystals. Place the assembled K-D's under the funnels. Visually examine each 60mL vial of sample. If there is excessive moisture in the sample, biphasic layering will be seen. If this occurs, add approximately 1 inch of sodium sulfate crystals to the vial. This should remove most of the moisture. Record the lot number of the sodium sulfate in the extraction logbook.

7.4.4 For a solvent exchange, no GPC, add approximately 50 mL Hexane to funnel and let drain through. Since methylene chloride has a lower boiling point than Hexane, this will result in a final extract in hexane only. Record the lot number of the solvent in the extraction logbook.

7.4.5 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. After pouring all of the extract volume through the sodium sulfate, rinse the extract vial three times with methylene chloride. Add the rinsings through the sodium sulfate to complete a

quantitative transfer. Rinse the sodium sulfate with ≈ 15 mLs of methylene chloride and allow draining.

7.4.6 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel, add one or two clean boiling stones to the K-D evaporative flask and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of hexane.

7.4.7 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of hexane. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL hexane.

7.4.8 Reduce the extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39°C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of hexane. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Record the temperature of the water in the nitrogen evaporation water bath in the extraction logbook, also note any problems or extract losses, if they occur.

7.4.9 Complete quantitative transfer of the extract to a vial by using hexane. Adjust the volume of the hexane extract to 5 mL in a 12mL vial using the appropriate “reference vial” for volume comparison. For Pesticides/PCB from tissue samples, adjust the volume of hexane extract to 2.0 mL in a 4.0 mL vial using the appropriate “reference vial” for volume comparison.

7.4.10 Label the vial with lab sample number, extraction date, matrix and analysis. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extractions logbook the box number and “tray location” of the individual extract vials.

7.4.11 All sample extracts for 8082 PCB analysis must undergo a sulfuric acid wash (cleanup) prior to analysis. Sample extracts for 8081 pesticide do not undergo further cleanup unless requested by the client. All soil/sediment sample extracts for combined 8081/8082 analyses must be split. One portion must be acid cleaned for 8082 analysis. The associated method blank must be split and acid-cleaned in the same fashion. PCB LCSs and matrix spikes are acid cleaned also. Pesticide LCSs and matrix spike samples are not subjected to further cleanup. Please refer to Katahdin SOP CA525 (current revision), Extract Cleanup Using Sulfuric Acid, for further instructions.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for <u>each</u> and <u>every</u> item listed below:

- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)
- Each extraction method or level
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for <u>each</u> and <u>every</u> item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

Refer to the current revision of the applicable Katahdin SOP for analysis of Pesticides and PCBs for quality control acceptance criteria.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the applicable analytical SOP for other method performance parameters and requirements.

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10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, Method 3545A, SW-846, Third Edition, Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, Revised February 2007, US EPA.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-803, Laboratory QA: Self Inspection System, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

LIST OF TABLES AND FIGURES

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TITLE: **PREPARATION OF SEDIMENT/SOIL AND TISSUE SAMPLES BY ACCELERATED SOLVENT EXTRACTION USING METHOD 3545 FOR SUBSEQUENT EXTRACTABLE PESTICIDE and PCB ANALYSIS**

TABLE 1

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-537-03	METHOD 3545, current revision
Apparatus/Materials		
Reagents	Katahdin receives a certificate with each lot indicating sodium sulfate crystals were dried by the manufacturer at prescribed conditions.	Section 5.3.3 Drying agents should be purified by heating at 400 °C for 4 hours…
Sample preservation/ handling	Samples are not ground prior to mixing with the drying agent.	Section 7.3 Grind a sufficient weight of the dried sample…
Procedures	Section 7.3.9 Nitrogen purge for Pesticide/PCB and DRO: 90 sec at 150 psi Oven temperature for DRO: 175°C	Section 7.8 Recommended extraction conditions for all extractions: Nitrogen purge: 60 sec at 150 psi. Oven temperature: 100 °C
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

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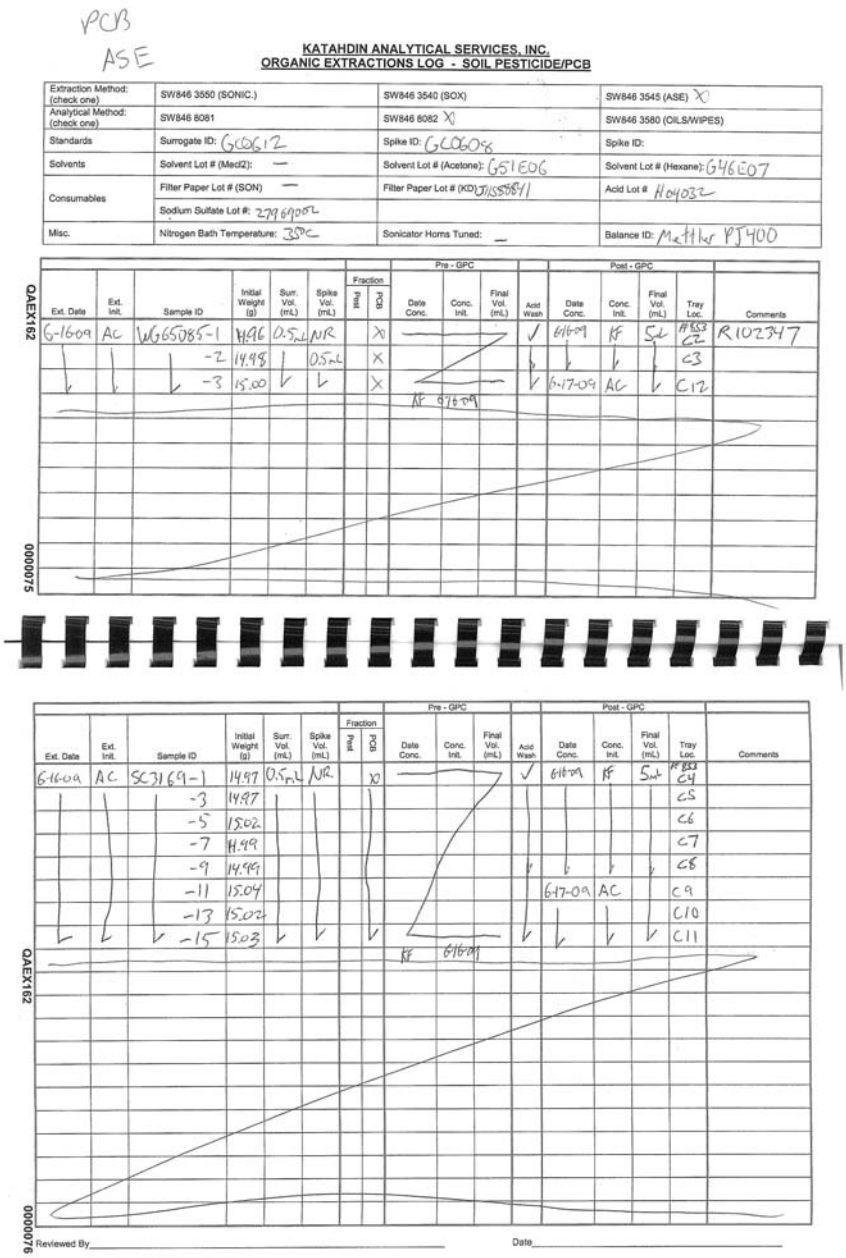
FIGURE 1

EXAMPLE OF LOGBOOK PAGE



PCB
ASE

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - SOIL PESTICIDE/PCB

Extraction Method: (check one)	SW846 3550 (SONIC.)	SW846 3540 (SOX)	SW846 3545 (ASE) X
Analytical Method: (check one)	SW846 8081	SW846 8082 X	SW846 3580 (OILS/WIPES)
Standards	Surrogate ID: GC0612	Spike ID: GC0608	Spike ID:
Solvents	Solvent Lot # (Med2): —	Solvent Lot # (Acetone): G51E06	Solvent Lot # (Hexane): G46E07
Consumables	Filter Paper Lot # (SON) —	Filter Paper Lot # (KD) J115584	Acid Lot # H04032
	Sodium Sulfate Lot #: 279690E		
Misc.	Nitrogen Bath Temperature: 35°C	Sonicator Horns Tuned: —	Balance ID: Mettler PJ400

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Fraction Pest	Fraction PCB	Pre - GPC Date Conc.	Pre - GPC Conc. Init.	Pre - GPC Final Vol. (mL)	Acid Wash	Post - GPC Date Conc.	Post - GPC Conc. Init.	Post - GPC Final Vol. (mL)	Tray Loc.	Comments
6-16-09	AC	WG65085-1	14.96	0.5mL	NR		X				✓	6/16/09	KF	5mL	#553 C2	R102347
		-2	14.98		0.5mL		X								C3	
		-3	15.00				X					6-17-09	AC		C12	
								KF 6/16/09								

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Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Fraction Pest	Fraction PCB	Pre - GPC Date Conc.	Pre - GPC Conc. Init.	Pre - GPC Final Vol. (mL)	Acid Wash	Post - GPC Date Conc.	Post - GPC Conc. Init.	Post - GPC Final Vol. (mL)	Tray Loc.	Comments
6-16-09	AC	SC3169-1	14.97	0.5mL	NR		X				✓	6/16/09	KF	5mL	#553 C4	
		-3	14.97												C5	
		-5	15.02												C6	
		-7	14.99												C7	
		-9	14.99												C8	
		-11	15.04									6-17-09	AC		C9	
		-13	15.02												C10	
		-15	15.03												C11	
								KF 6/16/09								

QAEX162

0000076

Reviewed By_____ Date_____

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

Prepared By: George Brewer Date: 11/97

Approved By:

Group Supervisor: George Brewer Date: 01/19/01

Operations Manager: John C. Burton Date: 1/22/01

QA Officer: Deborah J. Nadeau Date: 1·22·01

General Manager: Dennis F. Keegan Date: 1/22/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 3010A	Format changes, added pollution prevention, block digester; revised database references; revised and added tables.	DN	1·22·01	1/22/01
02 3010A	Added wording allowing use of digestates for ICP-MS analysis. Added use of block digester as primary heating source & adjusted volumes. Revised standard solution names & concs. in Figures 3 & 4.	DN	8·29·02	8·29·02
03	Added Uranium to spiking solutions for LCS & MS/D. Removed the Internal Custody Record for Metals Digestates figure and reference.	LAD	04/06	04/06
04	Minor changes to Section 7 to reflect current practices. Updated Figure 1 - Sample Prep Logbook. Updated Figure 2 and 3 - Spike amounts.	LAD	05/09	05/09
05	Added references. Updated Figure 2 and 3 with correct spike information. Added CA-108 reference for subsampling information.	LAD	04/10	04/10

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ____ of document **SOP CA-604-05**, titled **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ____ of document **SOP CA-604-05**, titled **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**.

Recipient: _____ Date: _____

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure utilized by Katahdin Analytical Services, Inc. personnel to solubilize metals in aqueous samples, wastes that contain suspended solids, and mobility-procedure extracts prior to analysis by inductively coupled plasma atomic emission spectroscopy (ICP) and inductively coupled plasma mass spectrometry (ICP-MS). This SOP applies to samples prepared by EPA Method 3010, with the method modifications mentioned in Table 2.

1.1 Definitions - none.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the acid digestion of aqueous samples by EPA Method 3010. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in the acid digestion of aqueous samples using EPA Method 3010 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their work in the appropriate lab notebook. Any deviations from the method or irregularities with the samples should also be recorded in the lab notebook and reported to the Supervisor or designated qualified data reviewer responsible for these data.

It is the responsibility of the Supervisor to ensure that technical personnel perform acid digestions in accordance with this SOP and to confirm that their work is properly documented through periodic review of the associated logbooks.

1.3 Safety

The acids used in this procedure are highly corrosive and reactive, and spiking standards contain toxic metals. The toxicity and reactivity of client samples are usually unknown, so samples should always be assumed to present a contact hazard. To reduce or eliminate exposure to potentially harmful chemicals, lab coats, gloves, and safety glasses or goggles must be worn whenever handling samples or reagents. Additional safety apparel, including face shields, rubber aprons, dust masks, and rubber shoe protectors, is available in the metals prep lab and should be worn whenever circumstances warrant.

Acids should be added to samples slowly and carefully while watching for reactions. This should be done under a hood, in case harmful fumes are evolved.

Hood sashes should be lowered as far as possible whenever beakers are being heated in the hood. Use caution when handling hot beakers.

Each qualified analyst or technician must be familiar with Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their supervisor, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Excess spiking solutions must be emptied into the corrosive waste carboy located in the metals prep lab for subsequent appropriate disposal in accordance with the Chemical Hygiene Plan and Safety Manual.

Sample digestates should be stored for a minimum of 60 days after digestion to allow for analysis, and reanalysis if necessary. Digestates older than 60 days may be emptied into the corrosive waste carboy in the metals prep lab for subsequent appropriate disposal in accordance with the Chemical Hygiene Plan and Safety Manual.

Any other wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Chemical Hygiene Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision.

2.0 SUMMARY OF METHOD

The aqueous sample is refluxed with nitric acid in a covered digestion vessel. Additional nitric acid is added until the color of the digestate has stabilized. After the digestate has been evaporated to a low volume, it is refluxed with hydrochloric acid and diluted to the appropriate final volume with reagent water.

Samples may be concentrated (i.e. final digestate volume less than initial sample volume) during digestion if lower detection limits are required. Volumes of reagents and spiking standards must be added in proportion to the final volume of the digestate. Because concentration of samples during digestion increases the concentrations of dissolved solids

and may exacerbate analytical interferences, concentration factors greater than 5 are not recommended.

3.0 INTERFERENCES

Interferences are discussed in the applicable analytical SOPs.

4.0 APPARATUS AND MATERIALS

4.1. 250 mL and 400 mL pre-cleaned Griffin beakers (cleaned according to the current revision of SOP CA-100, "Labware Cleaning") for digestion using a hot plate. If digestion will be performed using a block digester, 70ml graduated, polyethylene block digester tubes (with attached snap caps) will be used instead of glass beakers.

4.2 Ribbed watch glasses. If digestion is performed using a hot plate, 75 mm diameter and 100 mm diameter glass watch glasses (pre-cleaned as above) are used. If digestion is performed using a block digester, 40mm diameter disposable polyethylene watch glasses are used.

4.3 Adjustable volume automatic pipets covering the range from 10 uL to 1000 uL and disposable pipet tips; calibrated Finn pipets or Eppendorf pipets are acceptable.

4.4 Disposable graduated polystyrene specimen containers with pouring lips, 200 mL capacity.

4.5 Hot plate, block digester, or other heating source - adjustable and capable of maintaining a temperature of 90-95°C. Hot plates must be numbered for easy identification.

4.6 Device for measuring hot plate temperature. This may consist of a heat-resistant 100ml beaker containing reagent water in which a thermometer is immersed. When using a block digester, a digestion tube containing reagent water in which a thermometer is immersed may be used. The temperature of one hot plate is measured each day, on a rotating basis. The hot plate identification number and the measured temperature are recorded on the sample preparation logbook sheet.

4.7 Plastic funnels, pre-cleaned as in Section 4.1.

4.8 Filter funnel holders, capable of suspending plastic funnels above disposable specimen containers.

4.9 Polyethylene wash bottles for dispensing reagent water and 5% HNO_3.

4.10 Filter paper, Whatman No. 41 or equivalent. Filters are acid-washed immediately prior to use as follows. Place a pre-cleaned funnel in the funnel holder and put a disposable plastic specimen container under the funnel to collect the rinsates. Place a folded filter in the funnel and rinse three times with approximate 10 mL volumes of 5% HNO_3, making sure the entire surface of the filter is wetted each time and allowing each rinse to drain completely before continuing. Then rinse three times with approximate 25 mL volumes of reagent water. Discard the rinsates into the appropriate waste container. The acid-washed filter is now ready for use.

4.11 Polyethylene sample containers with screw caps or graduated polyethylene sample containers with attached snap lids, 125 mL capacity. These are not necessary when using the block digester since the final digestates are stored in the digestion tubes.

4.12 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid and 1:1 HCl.

5.0 REAGENTS

5.1 Concentrated nitric acid, HNO_3 – trace metals grade.

5.2 Concentrated hydrochloric acid, HCl – trace metals grade.

5.3 Reagent water - water that meets the performance specifications of ASTM Type II water (ASTM D1193).

5.4 Hydrochloric acid, 1:1. Add a volume of concentrated hydrochloric acid to an equivalent volume of reagent water and swirl gently to mix.

5.5 Nitric acid, 5% v/v. Add 25 mL concentrated HNO_3 to 475 mL reagent water in a 500 mL wash bottle. Cap, point the dispensing tip into a sink, and shake gently to mix.

5.6 Multi-element spiking solutions (as listed in Figure 3).

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples to be analyzed for dissolved metals should be filtered through a 0.45 um membrane filter and preserved as soon as possible after collection. Samples to be analyzed for total metals should be preserved, unfiltered, as soon as possible after collection. Aqueous samples are preserved by acidification with nitric acid to a pH of <2.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

Please refer to Katahdin Analytical Services SOP CA-108, "Basic Laboratory Technique", current revision, for information on subsampling.

7.0 PROCEDURES

7.1 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS computer spreadsheet. Print out a copy of the spreadsheet. With a permamament marker, make sample labels and attach to the polyethylene sample containers that will contain the digestates.

7.2 If using glass beakers as the digestion vessels, submerge previously cleaned beakers three times into a 10% nitric acid bath, then rinse three times with reagent water. The polyethylene digestion tubes used in conjunction with the block digester do not require acid rinsing or precleaning. Label the digestion vessels with sample numbers.

7.3 If digestion is performed using a block digester, the sample aliquot may be measured in the digestion vessel using the graduations on the digestion tubes. Measure 50 ml of well-mixed sample into a 70 ml block digestion tube. A larger sample aliquot may be used (up to 250 mL) if concentration of the sample during digestion is desired. Sample volumes larger than 50 mL may be digested in 250 mL beakers. Measure aliquot of well-mixed sample into a graduated specimen cup and transfer into a properly cleaned 250 mL beaker. Sample volumes of more than 50ml may not be digested using the 70ml block digester tubes. The volumes of reagents and spiking solutions used must be adjusted in proportion to the final digestate volume. The reagent and spiking solution volumes listed below are based on a final volume of 50 mL.

7.4 Add spike solutions to matrix spike samples and laboratory control samples (refer to Figure 3 for spiking instructions).

7.5 Use a repipetter to add 1.5 mL of concentrated HNO_3 (per 50 mL final volume) to the sample. Cover with a ribbed watch glass and place on heatsource. Heat cautiously, without boiling the sample, and evaporate to a low volume (10 - 15 mL).

<u>NOTE</u>: Do not allow any portion of the bottom of the digestion vessel to go dry during any part of the digestion. If a sample is allowed to go to dryness, low recoveries may result. Should this occur, discard the digestate and re-prepare the sample.

7.6 Cool the sample and add another 1.5 mL aliquot (per 50 mL final volume) of concentrated HNO_3. Cover and resume heating, increasing the temperature until a gentle reflux action occurs.

7.7 Continue heating, adding additional acid as necessary, until the digestate is light in color or does not change in appearance with continued refluxing.

7.8 Evaporate digestate to a low volume (10 - 15 mL).

7.9 Cool the sample and use a repipetter to add 5 mL (per 50 mL final volume) of 1:1 HCl. Cover the sample and resume heating, refluxing for an additional 15 minutes to dissolve any precipitate or residue resulting from evaporation.

7.10 Allow the sample to cool.

7.11 If the digestate contains visible particulate material, it must be filtered. Use a pre-cleaned funnel and acid-rinsed filter paper to filter the digestate into a clean graduated plastic specimen container or block digester digestion tube. Using a wash bottle, rinse the digestion vessel with reagent water and add the rinsates to the filter apparatus. After all of the liquid in the filter has drained into the specimen container or digestion tube, thoroughly rinse the filter three times with small (5-10 mL) volumes of reagent water, allowing the liquid to drain completely after each rinse.

If the digestion was performed using hot plates and the digestate does not contain particulate material, simply decant the digestate into a clean graduated specimen container (or graduated sample container with attached snap lid), rinse the beaker with reagent water, and add the rinsates to the container.

If the digestion was performed using a block digester and the digestate contains no visible particulate material, the digestate may be brought to final volume and stored in the digestion tube without decanting or rinsing.

7.12 Using the graduations on the specimen container, snap-lid container or digestion tube, dilute to the required final volume with reagent water. If a specimen container has been used, transfer the contents to the corresponding labeled polyethylene sample bottle, cap the bottle, and discard the empty specimen container. If a snap-lid container or digestion tube has been used, close and secure the snap-lid. Shake the container gently to mix. The digestate is now ready for analysis.

7.13 Review the ACCESS computer spreadsheet for accuracy. If any information is incorrect, make the necessary changes to the computer spreadsheet and print out a corrected copy. Do not discard the original copy of the spreadsheet. Record (hand write) the sample bottle ID, reagent lot numbers, spiking information, initial and final volumes, hot plate ID and hot plate temperature in the appropriate spaces on the spreadsheet. Record any method deviations, irregularities with the samples, or other pertinent observations at the bottom of the page, and sign and date the spreadsheet. Bind all copies of the spreadsheet in the sample preparation log. An

example sample preparation logbook page (ACCESS spreadsheet) is included as Figure 1.

7.14 Place each batch of digestates in a box labeled with the QC Batch ID, and put the box of digestates in the metals digestates storage area.

7.15 A condensation of the procedure described above is included in this SOP as Table 3. A controlled copy of this table may be posted in the metals preparation laboratory for reference by the analyst.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

8.1 At least one preparation blank for waters (PBW) is processed concurrently with each digestion batch of 20 or fewer samples, and is used to assess contamination resulting from the digestion procedure. The PBW consists of an aliquot of reagent water that is digested using the same reagents as those used to digest associated samples. The initial and final volumes of the PBW must be identical to those of the associated samples (i.e., if the associated samples were concentrated during digestion, the PBW must also be concentrated). Refer to the appropriate analytical SOP for PBW acceptance criteria and corrective actions.

8.2 At least one laboratory control sample for waters (LCSW) is processed concurrently with each digestion batch of 20 or fewer samples. The LCSW consists of an aliquot of reagent water that is spiked to contain all analytes of interest at known concentrations, and is digested using the same reagents as those used to digest associated samples. The initial and final volumes of the LCSW must be identical to those of the associated samples (i.e., if the associated samples were concentrated during digestion, the LCSW must also be concentrated). Directions for spiking the LCSW are contained in Figures 3 and 4. The measured analyte recoveries for the LCSW are used to assess digestion method performance. Refer to the appropriate analytical SOP for LCSW recovery acceptance criteria and corrective actions.

8.3 Matrix spiked samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. A matrix spike sample consists of an aliquot of a sample that is spiked with known amounts of all analytes of interest. Matrix spike recoveries are used to assess the effects of sample matrix on digestion and analysis performance. Directions for spiking matrix spike samples are contained in Figures 3 and 4. Refer to the appropriate analytical SOP for matrix spike recovery acceptance criteria and corrective actions.

8.4 Matrix spiked duplicate samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. Matrix spiked duplicate samples are used to assess the precision of the digestion and analysis methods.

Refer to the appropriate analytical SOP for matrix spike duplicate precision acceptance criteria and corrective actions.

NOTE: Clients may choose specific samples for matrix spike and matrix spike duplicate analysis; otherwise, the choice is left to the person performing the digestion. The sample volumes available may restrict the choice of samples used for matrix spike and duplicate digestion. Field blank samples should not be chosen for matrix spike and matrix spike duplicate analysis.

8.5 The quality control measures and frequencies described above are minimum requirements. They are summarized for reference in Table 1. Individual clients and analytical programs may impose additional QC requirements.

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOPs for method performance information.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 3010A.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

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Table 2 Summary of Method Modifications
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Figure 3 Element Concentrations in Matrix Spikes, LCSs, and Spiking Solutions: Method 3010

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TABLE 1

QC REQUIREMENTS

Analytical Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
3010	Preparation Blank for Waters (PBW)	One per prep batch of 20 or fewer samples	Refer to analytical method	Refer to analytical method
	Laboratory Control Sample for Waters (LCSW)	One per prep batch of 20 or fewer samples	Refer to analytical method	Refer to analytical method
	Matrix Spike Sample	One per prep batch	Refer to analytical method	Refer to analytical method
	Matrix Spike Duplicate Sample	One per prep batch	Refer to analytical method	Refer to analytical method
	Demonstration of analyst proficiency; accuracy and precision	One time demonstration by each analyst performing the method	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file

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TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-604-05	EPA METHOD 3010, current revision
Apparatus/Materials	1) Disposable plastic specimen cup used to measure sample volume. 2) Digestion performed in 250 mL, 400 mL Griffin beaker, or 70ml digestion tube to facilitate evaporation. 3) Ribbed watch glass used throughout digestion to reduce contamination.	1) Graduated cylinder used to measure sample volume. 2) Digestion performed in 150 mL Griffin beaker. 3) Ribbed and non-ribbed watch glasses alternated in digestion.
Procedures	1) Digestate may be analyzed for antimony and silver. 2) Sample aliquots larger or smaller than 100 mL may be used. 3) Sample evaporated to 10 - 15 mL.	1) Digestate may not be analyzed for antimony and silver. 2) Requires sample aliquot of 100 mL. 3) Sample evaporated to 5 mL.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 3

PROCEDURE CONDENSATION: EPA METHOD 3010

1. If performing digestion on a hot plate, rinse glass beakers and ribbed watch glasses 3 times in acid bath. Then rinse beakers and watch glasses 3 times with reagent water. If performing digestion with block digester, polyethylene digestion tubes do not require precleaning.
2. Label digestion vessels with sample numbers.
3. Mix sample well, measure 50 mL (or smaller or larger aliquot) into a polyethylene digestion tube. If using glass beakers, measure aliquot into graduated specimen container, and transfer to appropriate digestion vessel.
4. Add spike solutions to matrix spike samples and LCSW (refer to Figure 3 of this SOP).
5. Add 1.5 mL (per 50 mL final volume) concentrated HNO_3 to sample.
6. Cover with a ribbed watch glass.
7. Place on heating device (hotplate or block digester) and evaporate to 10 - 15 mL.
8. Cool sample and add another 1.5 mL (per 50 mL final volume) concentrated HNO_3.
9. Resume heating until gentle reflux action occurs.
10. Continue heating, adding additional HNO_3 as necessary until digestion is complete.
11. Evaporate to 10 - 15 mL.
12. Cool sample and add 5 mL (per 50 mL final volume) 1:1 HCl. Resume heating and reflux gently for 15 minutes.
13. Cool sample and filter (if necessary) or decant into a graduated polyetheyne digestion tube. Rinse beaker with reagent water and filter or decant rinsate into specimen container.
14. Dilute to appropriate final volume with reagent water.
15. Cap sample container and shake gently to mix.

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FIGURE 1

EXAMPLE PAGE FROM METALS SAMPLE PREPARATION LOGBOOK

Katahdin Analytical Services, Inc. — **Metals Preparation Benchsheet**

Reagent Information:
JT Baker HNO3: H11034 JT Baker HCL: H39027 KMG H2O2: N/A Method: 3010

LCS / Spike — LCS/Spiking Information:
- ☑ ☑ I.V. CLPP-SPK-1 (ID/Vol): MS1404 / 0.05 mL
- ☑ ☑ CLPP-SPK-INT1 (ID/Vol): MU1002I / 0.5 mL
- ☑ ☑ CLPP-SPK-INT2 (ID/Vol): MU1009 / 0.5 mL
- ☑ ☑ Uranium Spike (ID/Vol): MS1594 / 0.005 mL
- ☐ ☐ CLPP-SPK-4 (ID/Vol): N/A / mL

Hot Plate/Block ID: A
Start Time/Temp.: 930 / 95 °C
End Time/Temp.: 1500 / 95 °C
Thermometer ID/Pos.: ALC 8 / 1.4

Fisher Filter Paper: 411672325.2

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Clarity	Final Color	Final Clarity	Artifacts	Bottle
LCSWAB01ICW0	AB01ICW0	0.05	L	0.05	L	AQ	IC	AJB	02/01/2010	N/A	N/A	N/A	N/A		
PBWAB01ICW0	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010	N/A	N/A	N/A	N/A		
SD0405-001	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010						A
SD0405-001P	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010						
SD0405-001S	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010						
SD0405-002	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010						
SD0405-003	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010						
SD0405-004	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010						
SD0405-005	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010						
SD0422-001	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010						B
SD0423-001	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010						A
SD0429-001	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010						B
SD0429-002	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010						
SD0455-001	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010						
SD0455-002	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010						
SD0455-003	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010						
SD0455-004	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010						

AJB 2-1-10

Digestion performed by: AJB On: 2-1-10

Page: AB003 Revision: 00

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FIGURE 2

PREPARATION OF MATRIX SPIKES, LABORATORY CONTROL SAMPLES, AND SPIKING SOLUTIONS FOR DIGESTION OF AQUEOUS SAMPLES BY METHOD 3010

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 50 mL Final Volume (mL)
Laboratory Control Sample (LCSW) and Matrix Spike	CLPP-SPK-1	Inorganic Ventures	0.050
	CLPP-SPK-INT1	Lab Prepared (see below)	0.50
	CLPP-SPK-INT2	Lab Prepared (see below)	0.50
	1000 mg/L Uranium Standard	Inorganic Ventures	0.005

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
CLPP-SPK-INT1	1000 mg/L Se	High Purity Standards	1.0
	1000 mg/L As	High Purity Standards	1.0
	1000 mg/L Pb	High Purity Standards	1.0
	1000 mg/L Cd	High Purity Standards	2.5
	1000 mg/L Sb	High Purity Standards	1.0
	10000 mg/L K	High Purity Standards	10.0
	10000 mg/L Na	High Purity Standards	7.5
	10000 mg/L Mg	High Purity Standards	5.0
	10000 mg/L Ca	High Purity Standards	2.5
CLPP-SPK-INT2	2007ICS-1	Inorganic Ventures	10.0
	1000 mg/L Sr	High Purity Standards	5.0
	1000 mg/L Sn	High Purity Standards	5.0
	10000 mg/L Si	High Purity Standards	5.0

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FIGURE 3

ELEMENT CONCENTRATIONS IN MATRIX SPIKES, LABORATORY CONTROL SAMPLES, AND THEIR COMPONENT SPIKING SOLUTIONS FOR DIGESTION OF AQUEOUS SAMPLES BY METHOD 3010

Element	CONCENTRATION IN SOLUTION, mg/L							
	Matrix Spike	LCSW	CLPP-SPK-1	CLPP-SPK-4	CLPP-SPK-INT1	CLPP-SPK-INT2	2007 ICS-1	1000 mg/L U
Aluminum	2.000	2.000	2000					
Antimony	0.500	0.500		100	100			
Arsenic	0.500	0.500		4	10			
Barium	2.000	2.000	2000					
Beryllium	0.050	0.050	50					
Boron	0.500	0.500		50		50	500	
Cadmium	0.250	0.250		5	25			
Calcium	2.500	2.500			250			
Chromium	0.200	0.200	200					
Cobalt	0.500	0.500	500					
Copper	0.250	0.250	250					
Iron	1.000	1.000	1000					
Lead	0.500	0.500		2	10			
Magnesium	5.000	5.000			500			
Manganese	0.500	0.500	500					
Molybdenum	0.300	0.300		30		30	300	
Nickel	0.500	0.500	500					
Potassium	10.000	10.000			1000			
Selenium	0.500	0.500		5	50			
Silicon	5.230	5.230				523	230	
Silver	0.050	0.050	50					
Sodium	7.500	7.500			750			
Strontium	0.500	0.500		50		50		
Thallium	0.500	0.500		5	10			
Tin	0.500	0.500		50		50		
Titanium	1.000	1.000		100		100	1000	
Uranium	0.100	0.100						1000
Vanadium	0.500	0.500	500					
Zinc	0.500	0.500	500					

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND GFAA

Prepared By: George Brewer Date: 3/98

Approved By:

Group Supervisor: George Brewer Date: 01/24/01

Operations Manager: [illegible] C. Burto Date: 1/24/01

QA Officer: Deborah J. Nadeau Date: 1.24.01

General Manager: [illegible] Date: 1/25/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 3050B	Format changes, added pollution prevention, added MSD, added spiking instruction tables.	DN	1.24.01	1/24/01
02 3050B	Removed all references/procedures devoted to GFAA. Added use of digestates for ICP-MS analysis. Revised standard solution names + concs. in Tables 3 + 4 to reflect current practice.	DN	8.29.02	8.29.02
03 3050B	New Title to include ILMO5.3. Use of digestion block and polyethylene digestion tubes added to sections 4.0, 7.0 and Table 1. PBS changed from 1.0g water to 1.0g boiling chips. H_2O_2 addition from 3.0ml then 7.0mls to 2.0ml, 2.0ml then 7.0ml. Figures and Tables updated to reflect current practices.	LAD	03/08	03/08
04	Updated Tables 3 and 4 with current spike concentrations and volumes added. Updated logbook page. Added CA-108 reference for subsampling information.	LAD	08/09	08/09
05	Updated Tables 3 and 4 to reflect current spiking procedures.	LAD	09/10	09/10

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-605-05**, titled **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS AND ILM05.3.**

Recipient: _____Date:_____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-605-05**, titled **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS AND ILM05.3.**

Recipient: _____Date:_____

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the Katahdin Analytical Services, Inc. procedure utilized to dissolve solid matrices and solubilize metals from solid samples prior to analysis for metals by ICP-AES and ICP-MS. This SOP applies to samples prepared by EPA Method 3050, with method modifications as summarized in Table 2.

This procedure applies to all solid sample (e.g. sediments, sludges, soils, and ashes) preparations for ICP-AES and ICP-MS analyses. This method is not a total digestion technique for most samples. It is a very strong acid digestion that will dissolve almost all elements that could become "environmentally available". By design, elements bound in silicate structures are not normally dissolved by this procedure as they are not usually mobile in the environment.

1.1 Definitions

ICP-AES – Inductively Coupled Plasma Atomic Emission Spectroscopy.

ICP-MS – Inductively Coupled Plasma Mass Spectrometry.

LCSS – Laboratory Control Sample for Solids – A standard or solid reference material that has been brought through the sample preparation process.

Matrix Spike – An aliquot of a sample to which a known amount of analyte has been added before digestion.

PBS – Preparation Blank for Solids – An aliquot of reagent water that has been brought through the sample preparation process.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the acid digestion of solid samples by USEPA Method 3050 for metals analysis. Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Training".

It is the responsibility of all Katahdin technical personnel involved in the acid digestion of solid samples by USEPA Method 3050 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the procedure or irregularities with the samples should also be recorded in the lab notebook and reported to the responsible Department Manager or designated qualified data reviewer.

It is the responsibility of the Department Manager to ensure that technical personnel perform acid digestions in accordance with this SOP and to confirm that their work is properly documented through periodic review of the associated logbooks.

1.3 Safety

The acids used in this procedure are highly corrosive and reactive, and spiking standards contain toxic metals. The toxicity and reactivity of client samples are usually unknown, so samples should always be assumed to present a contact hazard. To reduce or eliminate exposure to potentially harmful chemicals, lab coats, gloves, and safety glasses or goggles must be worn whenever handling samples or reagents. Additional safety apparel, including face shields, aprons, dust masks, and shoe protectors, is available in the Metals prep lab and should be worn whenever circumstances warrant.

Acids should be added to samples slowly and carefully, while watching for reactions. This should be done under a hood, in case harmful fumes are evolved.

Hood sashes should be lowered as far as possible whenever beakers are being heated on a hot plate. Use caution when handling hot beakers.

Personnel are required to read the Katahdin Hazrdous Waste Management Plan and Safety Manual before performing this procedure, and must be familiar with the general rules for laboratory safety, personal hygiene, housekeeping, and use of protective clothing and equipment.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Excess spiking solutions must be emptied into the corrosive waste carboy located in the Metals prep lab for subsequent appropriate disposal in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual.

Sample digestates should be stored for a minimum of 60 days after digestion to allow for analysis, and reanalysis if necessary. Digestates older than 60 days may be emptied into the corrosive waste carboy in the Metals prep lab for subsequent appropriate disposal in accordance with the Katahdin Hazardous Waste Mnagement Plan and Safety Manual.

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

2.0 SUMMARY OF METHOD

A representative 1 to 2 g (wet weight) sample is digested with repeated additions of nitric acid and hydrogen peroxide. Hydrochloric acid is added to the initial digestate and the sample is refluxed. The digestate is then filtered and diluted to a final volume of 100 mL.

3.0 INTERFERENCES

Interferences are discussed in the applicable analytical SOPs.

4.0 APPARATUS AND MATERIALS

4.1 Digestion vessels. If digestion is performed using a hot plate, the appropriate digestion vessels are 100 mL pre-cleaned Griffin beakers (cleaned according to the current revision of SOP CA-100, "Labware Cleaning" and CA-602, “Glassware Preparation and Sample Preservation for Trace Element Analyses”). If digestion is performed using a block digester, the appropriate digestion vessels are new 70 mL disposable graduated polyethylene digestion tubes with attached snap lids.

4.2 Ribbed watch glasses. If digestion is performed using a hot plate, 75 mm diameter glass watch glasses (pre-cleaned as above) are used. If digestion is performed using a block digester, 40 mm diameter disposable polyethylene watch glasses are used.

4.3 Adjustable volume automatic pipets covering the range from 10 uL to 1000 uL and disposable pipet tips; calibrated Finn pipets or Eppendorf pipets are acceptable.

4.4 Disposable graduated polystyrene specimen containers with pouring lips, 200 mL capacity.

4.5 Hot plate or block digester, griddle, or other heating source - adjustable and capable of maintaining a temperature of $95^{o}C \pm 5^{o}C$. Heating sources must be numbered for easy identification.

4.6 Device for measuring hot plate temperature, consisting of a flask or digestion vessel in which the bulb of a thermometer is immersed in sand or water. The temperature of each hot plate used is measured and recordedeach day. The hot plate identification number and the measured temperature are recorded on the sample preparation logbook sheet.

4.7 Plastic funnels, pre-cleaned as in Section 4.1.

4.8 Filter funnel holders, capable of suspending plastic funnels above disposable specimen containers.

4.9 Polyethylene wash bottles for dispensing reagent water and 5% HNO_3.

4.10 Filter paper, Whatman No. 41 or equivalent. Filters are acid-washed immediately prior to use as follows. Place a pre-cleaned funnel in the funnel holder and put a disposable plastic specimen container under the funnel to collect the rinsates. Place a folded filter in the funnel and rinse three times with approximate 10 mL volumes of 5% HNO_3, making sure the entire surface of the filter is wetted each time and allowing each rinse to drain completely before continuing. Then rinse three times with approximate 25 mL volumes of reagent water, again allowing each rinse to drain completely. Discard the rinsates into the appropriate waste container. The acid-washed filter is now ready for use.

4.11 Polyethylene sample containers with screw caps or graduated polyethylene sample containers with attached snap lids, 125 mL capacity.

4.12 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid, 1:1 HNO_3, and concentrated HCl.

4.13 Analytical balance capable of reading to 0.01 gram.

4.14 Spatulas, scoops, or spoons; plastic or stainless steel, rinsed with 5% HNO_3 and reagent water. Disposable tongue depressors may be used and do not requrire to be rinsed.

5.0 REAGENTS

5.1 Concentrated nitric acid, HNO_3 – trace metals grade.

5.2 Concentrated hydrochloric acid, HCl – trace metals grade.

5.3 Reagent water - water that meets the performance specifications of ASTM Type II water (ASTM D1193).

5.4 Nitric acid, 1:1. Add a volume of concentrated HNO_3 to an equivalent volume of reagent water and swirl gently to mix.

5.5 Nitric acid, 5% v/v. Add 25 mL concentrated HNO_3 to 475 mL reagent water in a 500 mL wash bottle. Cap, point the dispensing tip into a sink, and shake gently to mix.

5.6 30% hydrogen peroxide (H_2O_2) - spectrometric grade.

5.7 Multielement spiking solutions (see Table 3 for a list of required spiking solutions).

5.8 Solid reference material – a soil containing all the elements of interest, with empirically established method-specific recoveries and acceptance limits for all analytes. Solid reference materials are purchased with documentation of analysis provided by the vendor. See Figure 4 for an example certificate of analysis for a solid reference material.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples should be collected in clean plastic or glass containers. Samples must be refrigerated (4°C ±2°C) upon receipt by the laboratory. The holding time for solid samples is 6 months from the date of sample collection.

7.0 PROCEDURE

The procedure described below is condensed for quick reference in Table 3.

SAMPLE PREPARATION

7.1 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS computer spreadsheet. Print out a copy of the spreadsheet (see Figure 2 for an example). Hand label the digestate vessels

7.2 If using glass beakers as the digestion vessels, submerge previously cleaned beakers and watch glasses three times into a 10% nitric acid bath, then rinse three times with reagent water. The polyethylene digestion tubes used in conjunction with the block digeter do not require acid rinsing or precleaning. Label the digestion vessels with sample numbers.

7.3 Weigh 1 to 2 g of well-mixed sample into a properly cleaned, labeled, and tared Griffin beaker or polyethylene digestion tube. Record (hand write) the weight of each sample on the printout of the digestion spreadsheet. Refer to Katahdin Analytical Services SOP CA-108, current revision “Basic Laboratory Technique” for more information on subsampling.

7.4 Weigh an appropriate amount of solid reference material to a clean, labeled, and tared Griffin beaker or polyethylene digestion tube to serve as a laboratory control sample.

7.5 Add spike solutions to matrix spike samples (refer to Tables 3 and 4 for spiking instructions).

7.6 Using repipetters, add 10 mL of 1:1 HNO_3, mix the slurry. Cover with a ribbed watch glass and place on heat source. Gently heat the sample to 95^oC ± 5 ^oC and reflux for 10 to 15 minutes without boiling. Remove the digestion vessel from the heat source and cool the sample.

7.7 Add 5 mL of concentrated HNO_3 to the sample, replace the watch glass, and reflux for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by HNO_3, repeat this step (addition of 5 mL of concentrated HNO_3) until no brown fumes are given off by the sample, indicating complete reaction by HNO_3.

7.8 Continue heating the sample at 95^oC ± 5^oC without boiling until the digestate has evaporated to approximately 5 to 10 mL or until two hours have elapsed, whichever occurs first. Do not allow the sample to go to dryness. Remove the digestion vessel from the heat source and cool the sample.

7.9 Add 2 mL of reagent water and 2 mL of 30% H_2O_2 to the sample, replace the watch glass, and heat gently on the heat source to start the peroxide reaction. Continue heating until effervescence subsides.

7.10 Add an additional 2 mL of 30% H_2O_2 to the sample, replace the watch glass, and heat gently on the heat source to start the peroxide reaction. Continue heating until effervescence subsides.

7.11 Add an additional 6 mL of 30% H_2O_2 in 1-mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged.

7.12 Continue heating the sample at 95^oC ± 5^oC without boiling until the digestate has evaporated to approximately 5 to 10 mL or until two hours have elapsed, whichever occurs first. Do not allow the sample to go to dryness. Remove the sample from the heat source and cool.

7.13 Add 10 mL of concentrated HCl to the digest from 7.12, replace the watch glass, and reflux at 95^oC ± 5^oC for 15 minutes. Remove the sample from the heat source and cool.

7.14 Use a pre-cleaned funnel and acid-rinsed filter paper to filter the digestate into a clean graduated polystyrene specimen container or graduated polyethylene sample container with attached snap lid. Using a wash bottle, rinse the digestion vessel with reagent water and add the rinsates to the filter apparatus. After all of the liquid in the filter has drained into the specimen container, thoroughly rinse the filter three times with small (5-10 mL) volumes of reagent water, allowing the liquid to drain completely after each rinse. Using the graduations on the specimen container or snap-lid container, dilute to 100 mL with reagent water. If a specimen container has been used, transfer the contents to the corresponding labeled polyethylene sample bottle, cap the bottle, and discard the empty specimen container. If a snap-lid

container has been used, close and secure the snap-lid. Shake the container gently to mix. The digestate is now ready for ICP-AES or ICP-MS analysis.

7.15 Review the ACCESS computer spreadsheet for accuracy. If any information is incorrect, make the necessary changes to the computer spreadsheet and print out a corrected copy. Do not discard the original copy of the spreadsheet. Record (hand write) reagent lot numbers, spiking information, and heat source temperature in the appropriate spaces on the spreadsheet. Record any method deviations, irregularities with the samples, or other pertinent observations at the bottom of the page, and sign and date the spreadsheet. Bind all copies of the spreadsheet in the sample preparation log. An example sample preparation logbook page (ACCESS spreadsheet) is included as Figure 2.

7.15 Reopen the electronic ACCESS spreadsheet for the digestion and transcribe the sample weights from the handwritten, bound copy into the electronic copy. The information in this electronic spreadsheet will later be imported into the ACCESS metals database and used to calculate sample concentrations on a weight basis.

7.16 Place each batch of digestates in a box labeled with the QC Batch ID, and put the box of digestates in the metals digestates storage area.

CALCULATIONS

7.17 Analytical results for solid samples are reported on a dry weight basis. Total solids are determined by the Wet Chemistry Group, and are recorded in spreadsheets that are electronically imported into the Access metals database. Final dry weight concentrations are calculated by the Access database as follows:

$$\text{Concentration (mg/kg dry weight)} = (C \times V) / (W \times S)$$

where: C = Measured concentration (mg/L)
V = Digestate final volume (L)
W = Sample wet weight (kg)
S = % Solids/100

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 3050 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and

standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Depatment Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

8.1 At least one preparation blank for soils (PBS) is processed concurrently with each digestion batch of 20 or fewer samples, and is used to assess contamination resulting from the digestion procedure. The PBS consists of a 1.0 g of boiling stones that is digested using the same reagents as those used to digest associated samples. Refer to the appropriate analytical SOP for PBS acceptance criteria and corrective actions.

8.2 At least one laboratory control sample for soils (LCSS) is processed concurrently with each digestion batch of 20 or fewer samples. The LCSS consists of an aliquot of a solid reference material for which the concentrations of the analytes of interest have been empirically established (solid-matrix LCSS), or an aliquot of reagent water that is spiked to contain all analytes of interest at known concentrations (aqueous-matrix LCSS). The solid reference material should normally be used as the LCSS, unless a particular client or analytical program requires that spiked reagent water be used. The LCSS is digested using the same reagents as those used to digest associated samples. Directions for spiking the aqueous-matrix LCSS are contained in Table 3. The measured analyte recoveries for the LCSS are used to assess digestion method performance. Refer to the appropriate analytical SOP for LCSS recovery acceptance criteria and corrective actions.

8.3 Matrix spike samples are processed along with each digestion batch at a minimum frequency of one per digestion batch. A matrix spike sample consists of an aliquot of a sample that is fortified with known amounts of all analytes of interest prior to digestion. Matrix spike recoveries are used to assess the biasing effects of sample matrix on digestion and analysis performance. Directions for spiking matrix spike samples are contained in Figure 2. Refer to the appropriate analytical SOP for matrix spike recovery acceptance criteria and corrective actions.

8.4 Matrix spiked duplicate samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. Matrix spiked duplicate samples are used to assess the precision of the digestion and analysis methods. Refer to the appropriate analytical SOP for matrix spike duplicate precision acceptance criteria and corrective actions.

<u>NOTE</u>: Clients may choose specific samples for matrix spike and duplicate analysis; otherwise, the choice is left to the person performing the digestion.

8.5 The quality control measures and frequencies described above are minimum requirements. Individual clients and analytical programs may impose additional QC requirements.

9.0 METHOD PERFORMANCE

Refer to the applicable instrumental analysis SOP for method performance information.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Test Methods for the Evaluation of Solid Waste," United States Environmental Protection Agency, SW-846, Third Edition, Final Update III, 12/96, Method 3050B.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

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Table 2 Summary of Method Modifications – Method 3050
Table 3 Preparation of Matrix Spikes and Spiking Solutions
Table 4 Element Concentrations in ICP-AES Matrix Spikes and Their Component Spiking Solutions
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Figure 2 Example Page from Metals Sample Preparation Logbook
Figure 3 Example Certificate of Analysis for Solid Reference Material

TABLE 1

QC REQUIREMENTS – METHOD 3050

Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
3050	Preparation Blank for Solids (PBS)	One per prep batch of 20 or fewer samples.	Refer to analytical method.	Refer to analytical method.
	Laboratory Control Sample for Solids (LCSS)	One per prep batch of 20 or fewer samples.	Refer to analytical method.	Refer to analytical method.
	Matrix Spike Sample	One per prep batch.	Refer to analytical method.	Refer to analytical method.
	Matrix Spike Duplicate Sample	One per prep batch.	Refer to analytical method.	Refer to analytical method.
	Demonstration of analyst proficiency	One-time demonstration by each analyst performing the method.	Must pass all applicable QC for method.	Repeat analysis until able to perform passing QC; document successful performance in personal training file.

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

TABLE 2

SUMMARY OF METHOD MODIFICATIONS – METHOD 3050

Topic	Katahdin SOP CA-605-02	Method 3050, current revision
Apparatus /Materials	1) Digestion performed in 100 mL Griffin beaker or 70 mL polyethylene tube. 2) Graduated disposable plastic cup or 120 mL polyethylene tube used to bring digestate to final volume.	1) Digestion performed in 250 mL Griffin beaker. 2) Volumetric flask used to bring digestate to final volume.
Procedure	1) Digestate volume reduced to 5 to 10 mL prior to filtering. 2) After filtration, the filters are rinsed three times with reagent water. 3) 30% H_2O_2 is added in two 2 mL aliquots and then six 1 mL aliquots.	1) Digestate volume reduced to 5 mL prior to filtering. 2) After filtration, the filters are rinsed twice with reagent water. 3) 30% H_2O_2 is added in one 3 mL aliquot and then seven 1 mL aliquots.

TABLE 3

PREPARATION OF MATRIX SPIKES AND SPIKING SOLUTIONS FOR DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Matrix Spike for ICP-AES	CLPP-SPK-1	Inorganic Ventures(IV)	0.10
	CLPP-SPK-INT1	Lab Prepared (see below)	1.00
	CLPP-SPK-INT2	Lab Prepared (see below)	1.00
	1000 mg/L Uranium Std.	IV or High Purity Standards	0.01

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
CLPP-SPK-INT1	1000 mg/L As,Pb,Sb,Se,Tl	High Purity Standards	1.0 each
	1000 mg/L Cd	High Purity Standards	2.5
	10000 mg/L K	High Purity Standards	10.0
	10000 mg/L Na	High Purity Standards	7.5
	10000 mg/L Mg	High Purity Standards	5.0
	10000 mg/L Ca	High Purity Standards	2.5
CLPP-SPK-INT2	1000 mg/L Mo	IV or High Purity Standards	3.0
	1000 mg/L B,Li,Sn,Sr,Ti	IV or High Purity Standards	5.0 each
	10000 mg/L Si	High Purity Standards	5.0

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

TABLE 4

ELEMENT CONCENTRATIONS IN ICP-AES MATRIX SPIKES AND THEIR COMPONENT SPIKING SOLUTIONS FOR DIGESTION OF SOLID SAMPLES BY METHOD 3050

	CONCENTRATION IN SOLUTION, mg/L							
Element	**Matrix Spike**	**CLPP-SPK-1**	**CLPP-SPK-4**	**CLPP-SPK-INT1**	**CLPP-SPK-INT2**			**1000 mg/L U**
Aluminum	2.000	2000						
Antimony	0.100		100	10				
Arsenic	0.100		4	10				
Barium	2.000	2000						
Beryllium	0.050	50						
Boron	0.500				50			
Cadmium	0.250		5	25				
Calcium	2.500			250				
Chromium	0.200	200						
Cobalt	0.500	500						
Copper	0.250	250						
Iron	1.000	1000						
Lead	0.100		2	10				
Lithium	0.500				50			
Magnesium	5.000			500				
Manganese	0.500	500						
Molybdenum	0.300				30			
Nickel	0.500	500						
Potassium	10.000			1000				
Selenium	0.100		5	10				
Silicon	5.000				500			
Silver	0.050	50						
Sodium	7.500			750				
Strontium	0.500				50			
Thallium	0.100		5	10				
Tin	0.500				50			
Titanium	0.500				50			
Uranium	0.100							1000
Vanadium	0.500	500						
Zinc	0.500	500						

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

FIGURE 1

PROCEDURE CONDENSATION – METHOD 3050

1. Prepare and print out ACCESS spreadsheet.
2. If performing digestion on a hot plate, rinse 250 mL Griffin beakers and ribbed watch glasses 3 times in acid bath. Then rinse beakers and watch glasses 3 times with laboratory reagent grade water. If perfoming digestion with block digester, polyethylene digestion tubes do not require precleaning.
3. Label digestion vessels (beakers or polyethylene sample tubes) with sample numbers.
4. Weigh 1 to 2 g of well-mixed sample into tared digestion vessels. Record sample weights.
5. Add spike solutions to matrix spike samples.
6. Add 10 mL 1:1 HNO_3 to samples and cover with watch glasses.
7. Reflux for 10 to 15 minutes at $95^o \pm 5^o$ C. without boiling. Cool samples.
8. Add 5 mL conc. HNO_3, cover beakers, and reflux for 30 minutes.
9. Repeat Step 8 as necessary until digestion is complete.
10. Reduce sample volumes to 5 to 10 mL or heat for 2 hours, whichever occurs first.
11. Cool sample and add 2 mL reagent water and 2 mL 30% H_2O_2. Heat gently until effervescence subsides.
12. Cool sample and add 2 mL 30% H_2O_2. Heat gently until effervescence subsides.
13. Cool samples and add 6 mL of 30% H_2O_2 in 1 mL aliquots. Heat gently until effervescence subsides.
14. Reduce sample volumes to 5 to 10 mL or heat for 2 hours, whichever occurs first.
15. Add 10 mL conc. HCl and reflux for 10 to 15 minutes at $95^o \pm 5^o$ C.
16. Cool sample and filter into graduated specimen container. Bring to volume with reagent water and transfer to labeled polyethylene bottle.
17. Enter sample weights into ACCESS spreadsheet.

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

FIGURE 2

EXAMPLE PAGE FROM METALS SAMPLE PREPARATION LOGBOOK

Katahdin Analytical Services, Inc. — Metals Preparation Benchsheet

Reagent Information:
JT Baker HNO3: M12022 JT Baker HCL: H64040 KMG H2O2: 6[illegible]022 Method: 3050H

LCS / Spike — LCS/Spiking Information:
- ☐ ☑ I.V. CLPP-SPK-1 (ID/Vol): MS1569 / 0.1 mL
- ☐ ☑ CLPP-SPK-INT1 (ID/Vol): MW11904 / 1.0 mL
- ☐ ☑ CLPP-SPK-INT2 (ID/Vol): MW11864 / 1.0 mL
- ☐ ☑ Uranium Spike (ID/Vol): MS1594 / ~~0.02~~ 0.01 mL ASD 7/31/09
- ☐ ☐ CLPP-SPK-4 (ID/Vol): N/A / mL

Hot Plate/Block ID: B
Start Temp.: 95 °C
End Temp.: 95 °C
Thermometer ID/Pos.: ALC 8 / 1.4
Fisher Filter Paper: K1147223B
LCSS: MS1578 Balance ID: Ohaus Galaxy 400 / Mettler AE200

REVIEWED
EM 073109
KATAHDIN ANALYTICAL METALS SECTION

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Texture	Final Color	Final Clarity	Artifacts	Bottle
LC2SZG311CS0	ZG311CS0	0.5074	g	0.10	L	SL	IC	AJB	07/31/2009	N/A	N/A	N/A	N/A	N/A	N/A
LCSSZG311CS0	ZG311CS0	0.5034	g		L	SL	IC	AJB	07/31/2009	N/A	N/A	N/A	N/A		
PBSZG311CS0	ZG311CS0	1.00	g		L	SL	IC	AJB	07/31/2009	N/A	N/A	N/A	N/A		
SC4328-001	ZG311CS0	1.79	g		L	SL	IC	AJB	07/31/2009						A
SC4328-001P	ZG311CS0	1.05	g		L	SL	IC	AJB	07/31/2009						
SC4328-001S	ZG311CS0	1.04	g		L	SL	IC	AJB	07/31/2009						
SC4348-001	ZG311CS0	1.35	g		L	SL	IC	AJB	07/31/2009						
SC4348-002	ZG311CS0	1.24	g		L	SL	IC	AJB	07/31/2009						
SC4348-003 7/31/09	ZG311CS0	1.11	g		L	SL	IC	AJB	07/31/2009						
SC4357-001 ASD	ZG311CS0	1.40	g		L	SL	IC	AJB	07/31/2009						
SC4357-~~002~~ 002	ZG311CS0	1.17	g		L	SL	IC	AJB	07/31/2009						
SC4357-~~003~~ 003	ZG311CS0	1.52	g	0.10	L	SL	IC	AJB	07/31/2009						A

ASD 7/31/09

Digestion performed by: ASD Date: 7/31/09 Page: ZG104 Revision: 00

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

FIGURE 3

EXAMPLE CERTIFICATE OF ANALYSIS FOR SOLID REFERENCE MATERIAL

ENVIRONMENTAL RESOURCE ASSOCIATES®
The Industry Standard™

M51475

DataPacK™
Lot No. D051-540

Trace Metals in Soil
Catalog No. 540

Certification

Method 3050 HNO3, H2O2, HCl Parameter	Total Concentration [1] (mg/Kg)	Certified Value [2] (mg/Kg)	Performance Acceptance Limits™ [3] (mg/Kg)
aluminum	55600*	7870	4630 - 11100
antimony	160	70.5	D.L. - 149
arsenic	316	289	234 - 344
barium	869	211	174 - 247
beryllium	60.9	54.4	45.2 - 63.6
boron	129	91.3	58.8 - 124
cadmium	114	101	82.9 - 119
calcium	9750*	3680	2970 - 4390
chromium	249	224	180 - 268
cobalt	113	101	82.7 - 119
copper	94.9	88.0	73.3 - 103
iron	24400*	15700	6610 - 24900
lead	184	158	129 - 187
magnesium	3780*	2260	1760 - 2750
manganese	703	420	343 - 497
mercury	5.32	5.18	3.42 - 6.87
molybdenum	80.2	69.6	55.5 - 83.7
nickel	137	120	99.1 - 141
potassium	33000*	3000	2200 - 3800
selenium	146	130	101 - 159
silver	127	104	68.9 - 139
sodium	15600*	1080	692 - 1470
strontium	326	113	90.5 - 135
thallium	106	94.0	72.8 - 115
tin	175	149	104 - 194
titanium	3100*	284	116 - 453
vanadium	151	111	85.1 - 137
zinc	311	272	215 - 329

Method 3050 HNO3, H2O2 Parameter	Total Concentration [1] mg/Kg	Certified Value [2] mg/Kg	Performance Acceptance Limits™ [3] mg/Kg
aluminum	55600*	7380	4440 - 10300
antimony	160	75.2	D.L. - 198
arsenic	316	284	225 - 343
barium	869	217	177 - 257
beryllium	60.9	53.6	42.7 - 64.5
boron	129	89.5	58.9 - 120
cadmium	114	103	83.6 - 122
calcium	9750*	3540	2800 - 4270
chromium	249	224	172 - 275
cobalt	113	101	82.0 - 120
copper	94.9	85.5	70.4 - 100
iron	24400*	12500	5480 - 19500
lead	184	162	132 - 192
magnesium	3780*	2160	1650 - 2670
manganese	703	415	330 - 500
mercury	5.32	5.18	3.42 - 6.87
molybdenum	80.2	68.8	52.7 - 84.9
nickel	137	119	98.5 - 140
potassium	33000*	2840	2160 - 3520
selenium	146	135	104 - 166
silver	127	107	49.8 - 164
sodium	15600*	1010	709 - 1310
strontium	326	111	89.0 - 133
thallium	106	99.3	76.8 - 122
tin	175	148	70.6 - 225
titanium	3100*	283	104 - 463
vanadium	151	104	70.5 - 138
zinc	311	275	222 - 328

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-608
Revision History
Cover Page
Page 1

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

Prepared By: George Brewer Date: 7/98

Approved By:

Group Supervisor: George Brewer Date: 01/23/01

Operations Manager: John C. Burton Date: 1/23/01

QA Officer: Deborah J. Nadeau Date: 1.23.01

General Manager: Duncan F. Hughes Date: 1/25/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 6010B	Format changes, added pollution prevention, expanded procedure and QC sections. Added tables.	DH	1.23.01	1/23/01
02 6010B	Calibration begins with analysis of S0 (cal. blank) followed by S1 (Mixed Cal. Std.) Changes to section 7.5 and Table 8 to reflect this. Made changes to element concs. in Tables 3,4,5,6 to reflect current practices.	DH	10.21.02	10.21.02
03 6010B	Added MN-IEC to standards run. Changed frequency of LRS. Changed concentration of HNO_3 in calibration blank. CRI changed from three separate solutions to one. Changed CRI vendor.	MRC	04.15.04	04.15.04
04	Updated ICV, CCV, ICB, PQL Chk std. PBW, PBS, MS & MSD acceptance criteria updated Table 1	LAD	05/06	05/06
05	Updated Tables 3,4,5,6 and 7 with current standard concentrations and prep. Updated Table 1 with current practices including NAVY audit findings. Updated sections 2, 7.2, 7.6 and Table 1 with new ICP information. Updated Table 8 with current sequence requirements.	LAD	07/07	07/07

TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added hardness definition and calculation (APP. 1)	LAD	09/07	09/07
07	Updated Summary to reflect new ICP functions. Removed ICP set-up updated tables to reflect changes in standard concentrations and preparation	LAD	11/08	11/08
08	Updates to Sections 8 and 10, Tables 1 and 2 to reflect changes from 6010B to 6010C. Added LLQC information and criteria to Sect. 8 and Table 1. Added criteria to analyze PQL standard at the beginning and END of each run.	LAD	02/09	02/09
09	Updated Sections 8, 9, 10 and Table 1 for compliance with DoD QSM version 4.1.	LAD	08/09	08/09
10	Added Table 2 - DoD QSM Ver. 4.1 QC Requirements. Minor correction to Table 1.	LAD	04/10	04/10
11	Added yttrium criteria to section 7 and Table 1.	LAD	06/10	06/10

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-608-11**, titled **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010.**

Recipient: _____Date:_____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-608-11**, titled **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010.**

Recipient: _____Date:_____

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

1.0 SCOPE AND APPLICATION

Inductively coupled plasma atomic-emission spectroscopy (ICP-AES) determines trace elements, including metals, in solution. The purpose of this SOP is to describe the procedures used by Katahdin Analytical Services, Inc. personnel to analyze aqueous and solid samples for trace metals by USEPA Method 6010 (Test Methods for Evaluating Solid Waste, Physical/ Chemical Methods, USEPA SW846).

Sample types that may be analyzed using these methods include drinking waters, ground waters, aqueous samples, TCLP, SPLP and EP Toxicity extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes. The following elements may be analyzed under this SOP: Al, Sb, As, Ba, Be, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, K, Se, Si, Ag, Na, Sn, Sr, Tl, Ti, V, and Zn.

All samples, except filtered ground water samples, analyzed under USEPA Method 6010 require digestion prior to analysis. USEPA Methods 3005, 3010, and 3050 describe appropriate digestion procedures for samples to be analyzed by ICP-AES under EPA Method 6010. Refer to current revisions of Katahdin SOPs CA-604 and CA-605, current revisions, for sample digestion procedures.

1.1 Definitions

Analytical Spike - An aliquot of a sample to which a known amount of analyte has been added before analysis and after digestion, if digestion is required.

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

CRI - Contract Required detection limit sample for ICP - A low concentration standard used to verify calibration accuracy near the low end of the calibration range.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy.

ICP-AES - Inductively Coupled Plasma Atomic Emission Spectroscopy.

ICS - Interference Check Sample - Two standards (ICSA and ICSAB) used to verify the effectiveness of interelement correction and background correction. Solution ICSA contains only interferents (Al, Ca, Fe, and Mg) at high concentrations (200 to

500 mg/L); solution ICSAB contains interferents at the same concentrations as well as analytes at low (20 mg/L or less) concentrations.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 99% confidence.

LOD – Limit of Detection – An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix-specific and is used for DoD QSM acceptance criteria.

LOQ – Limit of Quantitation.- The minimum concentration of a target analyte that produces a quantitative result within specified limits of precision and bias.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

LRS - Linear Range Standard - A high-concentration standard used to determine the upper reporting limit of the ICP calibration.

PB - Preparation Blank - Reagent water that has been brought through the sample preparation process.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

Serial Dilution - The dilution of a sample by a factor of five. When corrected by the dilution factor, the measured analyte concentrations of the diluted sample should agree with those of the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

Hardness – The sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in mg/L.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in ICP analysis by EPA Method 6010. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in ICP analysis by Method 6010 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

Samples, sample digestates, standards, and other reagents used in ICP analysis may contain high concentrations of acids and toxic metals. Safety glasses should be worn when changing or adjusting argon tanks.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes from ICP analysis should be disposed of in a manner appropriate to the hazards they present. Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Analytical Environmental Health and Safety Manual I and SOP SD-903, “Sample Disposal,” current revision. Expired

standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD

This method describes multielemental determinations by ICP-AES using simultaneous optical systems and radial and axial viewing of the plasma. The basis of the method is the measurement of atomic emission from sample atoms entrained in an argon plasma by optical spectroscopy. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where thermal excitation of entrained atoms and ions occurs. Characteristic atomic-line and ionic-line emission spectra are produced by a radio-frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating and the intensities of the emitted lines are monitored by a solid state charge injection device (CID) camera system. Photocurrents from the CID camera system are measured by a computer system. Element concentrations of unknown samples are quantitated by comparison of sample emission intensities to emission intensities of standards of known concentration. A background correction technique is used to compensate for variable background contribution to the determination of trace elements. Background is measured adjacent to the analyte lines on samples during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, has been determined by the complexity of the spectrum adjacent to the analytical line. The position used must be relatively free of spectral interference and must reflect the same change in background intensity as occurs at the analyte wavelength. Physical interferences are corrected through the use of an internal standard (yttrium) that is automatically added to all samples and standards prior to nebulization. The possibility of additional interferences (noted in section 3) must be recognized and appropriate corrections applied.

3.0 INTERFERENCES

Several types of interference effects may contribute to inaccuracies in the determination of trace elements. They can be summarized as spectral interferences, physical interferences, and chemical interferences.

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; and 4) background from stray light from the line emission of high concentration elements. The first of these effects is compensated by utilizing the computer correction of raw data, requiring the monitoring and measurement of the interfering element (interelement correction). The second effect is controlled by choosing analytical wavelengths that are free from overlapping molecular emission spectra. The third and fourth effects are usually compensated by a background correction adjacent to the analyte line. Uncorrected spectral interferences may be detected through examination of serial dilution and matrix spike data.

Physical interferences are generally considered to be effects associated with sample nebulization and transport processes. Such properties as changes in viscosity and surface tension can cause significant inaccuracies, especially in samples that may contain high dissolved solids and/or acid concentrations. Matrix matching of standards and samples and the use of a peristaltic pump may lessen these interferences. If these types of interferences are operative, they must be reduced by dilution of the sample and/or utilization of standard addition techniques. Another problem that can occur from high dissolved solids is salt buildup at the tip of the nebulizer. This affects aerosol flow rate causing instrumental drift. Regular cleaning of nebulizer tips and dilution of samples with high dissolved solids contents are used to control this problem. Physical interferences are also corrected by this laboratory through the use of an internal standard. Uncorrected physical interferences may be detected through examination of serial dilution and matrix spike data. Instrument drift caused by the salting up of nebulizer tips may also be detected by looking for oriented drift in calibration verification standards analyzed regularly throughout the run.

Chemical interferences are characterized by molecular compound formation, ionization effects, and solute vaporization effects. Normally these effects are not pronounced with the ICP technique; however, if observed they can be minimized by careful selection of operating conditions (i.e., incident power, observation position, etc.), by matrix matching, and by standard addition procedures. These types of interferences can be highly dependent on matrix type and the specific analyte element. Uncorrected chemical interferences may be detected through examination of serial dilution data.

4.0 APPARATUS AND MATERIALS

4.1 Computer-controlled inductively-coupled plasma atomic emission spectrometer (plasma viewed radially or axially) equipped for internal standardization, and capable of performing automatic background correction and interelement correction. For more information refer to the current revision of Katahdin SOP CA-632, “Operation and Maintenance of the Thermo ICAP 6500 ICP Spectrophotometer”.

4.2 Computer-controlled autosampler.

4.3 Argon gas supply – high purity.

4.4 Volumetric glassware of suitable precision and accuracy.

4.5 Automatic pipets of suitable precision and accuracy. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

Refer to the appropriate instrument-specific SOP for additional required equipment.

5.0 REAGENTS

5.1 Hydrochloric acid, concentrated (HCl) – spectroscopic grade.

5.2 Nitric acid, concentrated (HNO_3) – spectroscopic grade.

5.3 Reagent water, trace metals free.

5.4 Calibration blank – reagent water containing HCl (5% v/v) and HNO_3 (5% v/v). Calibration blank solution is prepared in large volumes (up to 20 liters) and stored in a carboy. Calibration blank solution is used in establishing the analytical curve, and in all initial and continuing calibration blank determinations. This solution is also used to flush the system between standards and samples. Intermediate and working standards are prepared by diluting stock standards and intermediate standards with calibration blank solution so that all standards and blanks are acid matrix-matched to sample digestates.

5.5 Single element and multielement stock standard solutions – purchased standards prepared from high purity salts or metals, and supplied by the vendors with certificates of purity and analysis. Refer to Tables 3 and 4 for a listing of stock standards required, and to Table 8 for element concentrations in stock standards.

5.6 Intermediate standard solutions – laboratory-prepared multielement standards that are used in the subsequent preparation of working standards. Refer to Table 5 for a listing of intermediate standards required and for preparation instructions. Refer to Table 7 for element concentrations in intermediate standards.

5.7 Working standard solutions – laboratory-prepared multielement standards that are used to calibrate the instrument and to perform all necessary QC checks. Refer to Table 4 for a listing of working standards and for preparation instructions. Refer to Table 6 for element concentrations in working standards.

5.8 5 mg/L yttrium internal standard solution – add 0.5 mL 10000 mg/L yttrium stock standard to a 1000 mL volumetric flask half filled with calibration blank solution. Bring to volume with calibration blank solution.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples to be analyzed for trace metals by ICP should be collected and preserved as described in the following table.

Matrix	Container[1]	Volume / Weight	Preservation / Treatment	Holding Time
Aqueous (total)	P, G	250 mL	HNO_3 to pH < 2	6 months
Aqueous (dissolved)	P, G	250 mL	Filter, HNO_3 to pH < 2	6 months
Solid	P, G	10 g	Cool, 4°C	6 months

[1] P = polyethylene or, G = glass

7.0 PROCEDURES

7.1 Begin by following the startup and calibration instructions provided in the current revision of Katahdin SOP CA-632, "Operation and Maintenance of the Thermo ICAP 6500 ICP Spectrophotometer"

7.2 Analysis must proceed in the sequence described in Table 9 to ensure that all necessary quality control samples are analyzed at the appropriate frequencies. A minimum of two replicate integrations is required for all standards and samples. Analysis always begins with the analysis of a calibration blank solution (S0) followed by analysis of a multielement calibration standard (S1 in Table 4) to calibrate the instrument. The system is flushed with calibration blank for two minutes between each sample and standard, and each sample and standard is aspirated for one minute prior to the beginning of emission measurements.

7.3 Analysis continues with analysis of the initial calibration verification standard (ICV) and the initial calibration blank (ICB) to verify the accuracy of the calibration. Refer to Section 8 and Table 1 for additional information.

7.4 A continuing calibration verification standard (CCV) and a continuing calibration blank (CCB) must be analyzed at the beginning of the run, after every ten samples, and at the end of the run to verify the continued accuracy of the calibration. Refer to Section 8 and Table 1 for additional information.

7.5 Interference check standard solutions (ICSA and ICSAB) must be analyzed at the beginning, end, and at periodic intervals (4-6 hours, 30-40 analytical samples) throughout the sample run to verify the accuracy of the IEC factors. Refer to Section 8 and Table 1 for additional information.

7.6 A practical quantitation limit standard (PQL) must be analyzed at the beginning of each run to determine the accuracy of the calibration at the reporting limit. Refer to Section 8 and Table 1 for additional information.

7.7 All sample analytical results for a particular element that are bracketed (preceded or followed) by failing results in a QC sample (ICV, ICB, CCV, CCB, ICSA, or ICSAB) for that element must not be reported. The sample must be reanalyzed for the element in question.

7.8 All samples that exceed the linear dynamic range must be diluted and reanalyzed. This includes samples with interfering elements that exceed the calibration ranges, because accurate quantitation of interfering elements is necessary for reliable interelement correction. For example, if a sample has been submitted to the laboratory for lead analysis, and the measured aluminum concentration of that

sample exceeds the calibration range for aluminum, it must be diluted sufficiently to bring aluminum within the linear dynamic range and the lead result must be reported from that dilution analysis.

7.9 If dilutions of digested samples are performed, the measured element concentrations must be multiplied by the dilution factor prior to reporting. This is accomplished automatically by entering the dilution factor in the autosampler table prior to initiation of analysis.

7.10 All analyses are performed using yttrium as an internal standard to compensate for enhancement or depression of the analytical signal due to matrix effects. Yttrium solution is pumped at a constant rate through one channel of the peristaltic pump. Samples and standards are pumped through a second channel of the pump. The tubing carrying the internal standard is connected to the tubing carrying samples and standards downstream from the pump, and mixing of the two streams is accomplished in a mixing coil downstream from the connection, prior to nebulization. For each sample or standard, the computer that controls the spectrometer divides the detected emission signal for each element by the detected yttrium emission signal prior to quantitation, thus normalizing all emission signals to that of yttrium. The yttrium recovery must be within ± 20% of the counts of the initial calibration blank. If the recovery is outside of this, the sample must be diluted and reanalyzed.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 6010 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases “qualified” data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information

will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument. This determination requires seven replicate analyses of a reagent water spiked at 3-5 times the anticipated detection limit for each analyte, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed on each instrument. This determination requires at least seven replicate digestions and analyses of a reagent water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the seven replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.3 Limits of Detection (LOD) are used when evaluating data using DoD QSM. The LOD is established by spiking a quality system matrix at 2-3 times the detection limit for a single analyte standard and 1-4 times the detection limit for a multi-analyte standard. The LOD must be verified quarterly. For more information on performing LOD determinations, refer to the current revision of Katahdin SOP QA-806.

8.4 Limits of Quantitation (LOQ) are used when evaluating data using DoD QSM. The LOQ must be above the LOD.

8.5 A Lower Limit of Quantitation Check (LLQC) sample must be prepared and analyzed annually or on an as-needed basis to confirm the laboratory's Practical Quantitation Limits (PQLs). The LLQC sample is equivalent to the PQL standard (Section 8.10) but is carried through the entire sample preparation and analysis process. Element recoveries for the LLQC sample must fall within 70% to 130% of the expected concentrations to confirm the previously established PQLs.

8.6 The upper limit of the linear dynamic range (LDR) must be established for each wavelength utilized. It must be determined from a linear calibration prepared in the normal manner using the established analytical operating procedure for the instrument. The LDR should be determined by analyzing succeedingly higher standard concentrations of the analyte until the observed analyte concentration differs by no more than 10% from the stated concentration of the standard. Determined LDRs must be documented and kept on file. The LDR which may be

used for the analyses of samples should be judged by the analyst from the resulting data. Determined sample analyte concentrations that are greater than the determined upper LDR limit must be diluted and reanalyzed. The LDRs should be verified **every six months** or whenever, in the judgment of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be redetermined.

8.7 The alkali and alkaline earth metals may have non-linear response curves due to ionization and self-absorption effects. These curves may be used for quantitation of samples if the effective range is checked and if the second order curve fit has a correlation coefficient of 0.998 or better. Third order fits are not acceptable. Non-linear response curves must be revalidated and recalculated every six months.

ANALYTICAL RUN QC SAMPLES

8.8 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared by combining compatible elements from a standard source different than that of the calibration standard and at concentrations within the linear working range of the instrument. The results of the ICV must fall within 90% to 110% of the expected values. If the ICV fails, result for the failing elements may not be reported from the run unless the ICV recovery is greater than 110% and the sample result is less than the PQL.

No results may be accepted for failing elements if DoD QSM acceptance criteria are being used.

8.9 Continuing Calibration Verification (CCV) solutions are analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standards used for calibration at concentrations near the mid-point of the calibration curve. Results of the CCVs must fall within 90% to 110% of the expected values. If a CCV fails, results for the failing elements may not be reported from the run unless the CCV recovery is greater than 110% and the sample result is less than the PQL (less than reporting limit for DoD QSM). Also, for failing elements, all samples analyzed after the last passing CCV must be reanalyzed.

8.10 Calibration blank solution is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the Practical Quantitation Level (PQL) for each element. If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed, with the following exception. If the result for a CCB or ICB is greater than the PQL, sample results that are less than the PQL or greater than or equal to ten times the measured CCB

concentration may be reported. Also, for failing elements, all samples analyzed after the last passing CCB must be reanalyzed, with the exception noted above.

If DoD QSM acceptance criteria are being used, the absolute values of results of ICBs and CCBs must be less than the Limit of Detection (LOD). If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed.

8.11 Interference check solutions ICSA and ICSAB (refer to Section 1.1) are analyzed at the beginning of each run to verify interelement correction factors and background correction. ICSA contains interferent elements (Al, Ca, Fe, and Mg) only, at concentrations of 200 mg/L to 500 mg/L. Results for interfering elements in the ICSA must fall within 80% to 120% of the expected values. Results for unspiked elements in ICSA must fall within ± PQL if the PQL is greater than 0.01 mg/L, within ± 2xPQL if the PQL is less than or equal to 0.01 mg/L. If DoD QSM acceptance criteria are being used, the absolute value of unspiked elements must be less than the LOD. ICSAB contains interferent elements at concentrations of 200 mg/L to 500 mg/L, and analytes at concentrations of 20 mg/L or less. Results for all elements (interferents and analytes) in ICSAB must fall within 80% to 120% of the expected values. If the ICSA or ICSAB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICSA or ICSAB has been analyzed.

8.12 A Practical Quantitation Limit (PQL) Check Standard or low level continuing calibration verification (LLCCV) is analyzed at the beginning (after the ICV and ICB samples) and at the end of each run. Element concentrations in this solution are at the laboratories practical quantitation limit. Element recoveries for the PQL check Standard must fall between 70-130% of the expected values. If the PQL Check Standard fails, the results for the failing elements may not be reported from the run, unless the PQL Check Standard recovery is greater than 130% and the samples results are less than the PQL.

If DoD QSM acceptance criteria are being used, recoveries must fall between 80-120%. If the PQL Check Standard fails, the results for the failing elements may not be reported from the run.

PREPARATION BATCH QC SAMPLES

8.13 Each digestion batch of twenty or fewer samples will contain a preparation blank and a laboratory control sample. Each batch will also contain one or more of the following QC samples: laboratory control sample duplicate, sample duplicate, matrix spike sample or matrix spike sample duplicate.

8.14 A preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the

Practical Quantitation Level (PQL) for each element. For DoD QSM acceptance criteria the results must be less than ½ the PQL except for common contaminants which must be less than the PQL. If a preparation blank fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL (greater than ½ PQL for DoD), associated sample results that are less than the PQL (less than ½ PQL for DoD) or greater than or equal to ten times the measured preparation blank concentration may be reported.

8.15 A laboratory control sample (LCS), consisting of spiked reagent water or a solid reference material carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. Results for laboratory control samples must fall within 80% to 120% of the expected value, unless vendor-supplied limits (for solid reference materials) or laboratory-generated statistical limits are available. If a laboratory control sample fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested with the following exception. If the LCS fails high, sample results less than the PQL may be reported.

If DoD QSM acceptance criteria are being used, recovery for solid matrix samples must fall between 80% to 120% except for Ag, which must fall between 75% and 120%. Results may not be reported without a valid LCS and will be qualified and explained if reanalysis cannot be performed.

SAMPLE MATRIX QC SAMPLES

8.16 Matrix spiked duplicate samples are prepared at a minimum frequency of one per digestion batch. The recovery for each element in a spiked sample or spiked duplicate sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If one or both spike recoveries fail, the associated sample result must be flagged on the report of analysis. If DoD QSM acceptance criteria are being used, recoveries must be the same as stated for laboratory control samples.

The relative percent difference between sample duplicate, matrix spiked duplicate or LCS duplicate, is calculated as follows:

$$\text{RPD (\%)} = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where: D_1 = sample result
D_2= duplicate sample result

A control limit of 20% RPD is applied to duplicate analysis if the original sample result is greater than 50X the IDL. If the matrix spike duplicate analysis fails, the associated sample result must be flagged on the report of analysis.

8.15 A serial dilution is analyzed to check for chemical or physical interferences. If the analyte concentration of a sample is sufficiently high (minimally, 50 x IDL or 50 x LOQ if using DoD QSM acceptance criteria), the measured concentration of a serial dilution (1:5 dilution) of the sample should agree within 90% to 110% of the original determination. The percent difference between the original sample and the serial dilution should be calculated as follows:

$$\text{Difference (\%)} = \frac{|L-S|}{S} * 100\%$$

where: L = Serial dilution result (corrected for dilution)
S = Original sample result

If the serial dilution analysis fails, a matrix interference should be suspected. The associated sample result should be flagged on the report of analysis or the sample should be reanalyzed at dilution to eliminate the interference.

For DoD QSM samples a Post-digestion Spike (PDS) addition must be performed if the serial dilution is not within acceptance criteria.

8.16 Post-digestion Spike (PDS) additions must be performed for DoD QSM samples if the serial dilution is not within acceptance criteria or if the analyte concentrations in all samples are less than 50x the LOD. The spike addition should produce a concentration that is between 10 and 100x the LOQ. The recovery of the PDS must be within 75-125%. If the PDS fails, all samples must be run by method of standard additions or appropriately flagged.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) and the limit of detection (LOD) are defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs and LODs are determined prior to sample analysis per type of instrument and filed with the Inorganic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, for procedures on determining the MDL.

Refer to the current revision of Method 6010 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 6010C.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09
The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, current revision.

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TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 1

QC REQUIREMENTS

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
USEPA 6010	Initial Calibration, minimum 1 point plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient ® ≥ 0.998	Recalibrate
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within ± 10% of true value.	1) Do not use results for failing elements unless the ICV > 110% and the sample < the PQL. 2) Investigate and correct 3) DoD: No samples may be run until calibration is verified
	Initial Calibration Blank (ICB)	Immediately after the ICV.	Absolute value of ICB < PQL.	1) Do not use results if ≥ PQL and 10x< CCB level. 2) Investigate and correct problem.
	Continuing Calibration Verification (CCV)	At beginning of run, after every 10 samples, and at end of run.	Recovery within ± 10% of true value.	1) Do not use results for failing elements unless the CCV > 110% and the sample < the PQL. 2) Investigate and correct problem.
	Continuing Calibration Blank (CCB)	After every 10 samples and at end of the run.	Absolute value of CCB < PQL.	1) Do not use results if ≥ PQL and < 10x CCB level. 2) Investigate and correct problem.
	Practical Quantitation Level Check Standard (PQL) (LLCCV)	At beginning and end of run.	Recovery within ± 30% of true value.	1) Do not use results for failing elements unless the ICV > 110% and the sample < the PQL. 2) Investigate and correct problem.
	Interference Check Solution A (ICSA)	At beginning and end of run.	For Al, Ca, Fe, and Mg, recovery within ± 20% of true value. For analytes not spiked, ± PQL, or, if PQL ≤ 0.01 mg/L, ± 2x PQL.	1) Do not use results for failing elements. 2) Investigate and correct problem.
	Interference Check Solution AB (ICSAB)	At beginning and end of run.	Recovery of each analyte within ± 20% of true value.	1) Do not use results for failing elements. 2) Investigate and correct problem.
	Preparation Blank (PBW/PBS)	One per digestion batch of 20 or fewer samples.	Less than PQL.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration ≥ PQL and <10x the blank concentration.
	Laboratory Control Sample (LCSW/LCSS)	One per digestion batch of 20 or fewer samples.	Recovery within ± 20% of true value, unless vendor-supplied or statistical limits have been established.	1) Investigate source of problem. 2) Redigest and reanalyze all associated samples. 3) DoD: Flag specific analytes if samples cannot be reanalyzed.
	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery ± 25% of true value, if sample < 4x spike added.	1) Flag results.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 1 (cont)

QC REQUIREMENTS

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
USEPA 6010 (cont.)	Matrix Spike Duplicate Sample (P) or sample duplicate	One per digestion batch of 20 or fewer samples.	Recovery ± 25% of true value, if sample < 4x spike added. RPD ≤20% for duplicate spikes and sample duplicates.	1) Flag results.
	Serial Dilution (L)	One per digestion batch.	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result. Flag result or dilute and reanalyzed sample to eliminate interference	Perform post digestion spike addition (PDS)
	Post-Digestion Spike Sample (A)	When dilution test fails or analyte concentration in all samples <50x LOD	Recovery within ± 25%.	Run associated samples by method of standard addition or flag results.
	Internal Standard	Every sample	± 20% (compared to the initial calibration blank)	Dilute sample and reanalyze.
	Instrument Detection Limit (IDL) Study	Quarterly.	IDL < MDL PQL > 2-3 * the IDL	1) Repeat IDL study. 2) Raise PQL.
	Method Detection Limit (MDL) Study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		
	Lower Limit of Quantitation Check (LLQC) Sample	Digest and analyze annually or as needed to confirm PQLs	70% - 130% of true value	Re-evaluate PQLs
	Linear Range Study	Every six months	Run succeedingly higher stds until recovery not within ± 10%. Use highest passing concentration as upper limit of linear range.	Only accept data to highest passing concentration until next linear range study.
	Limit of Detection (LOD) Determination	Quarterly	LOD = 1-4X MDL	Repeat LOD Determination
	Limit of Quantification (LOQ) Determination	Quarterly	LOQ > LOD	

TABLE 2

DoD QSM VERSION 4.1 QC REQUIREMENTS

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.	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	Refer to current revision of SOP QA-806				
LOQ establishment and verification	Refer to current revision of SOP QA-806				
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.	
Initial calibration (ICAL) for all analytes ICP: minimum one high standard and a calibration blank	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, r ≥ 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.

TABLE 2 (cont)

DoD QSM VERSION 4.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing calibration verification (CCV)	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.	After every 10 field samples and at the end of the analysis sequence.	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	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.
Method blank	One per preparatory batch.	No analytes detected > ½ RL (> RL for common lab contaminants) and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For negative blanks, absolute value < LOD.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	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. For negative blanks, absolute value < LOD.	Correct problem. Re-prep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	
Interference check solutions (ICS)	At the beginning of an analytical run.	ICS-A: Absolute value of concentration for all non-spiked analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within ± 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.	

TABLE 2

DoD QSM VERSION 4.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
LCS containing all analytes to be reported	One per preparatory batch.	Water and Soil: Recovery must be within + 20% of the true value	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.	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.
Matrix spike (MS)	One per preparatory batch per matrix	For matrix evaluation, recovery must be within + 20% of the true value.	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.	MSD: For matrix evaluation, recovery must be within + 20% of the true value. 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	One per preparatory batch.	If sample concentrations > 50 x LOQ, then the five-fold dilution must agree within ± 10% of the original measurement.	Perform post-digestion spike (PDS) addition.	Flagging criteria are not appropriate.	Only applicable for samples with concentrations > 50 x LOQ.
Post-digestion spike (PDS) addition	When dilution test fails or analyte concentration in all samples < 50 x LOD.	Recovery within 75-125%.	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.
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.	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-608-11	Method 6010, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures		
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		
QC - Calibration Blanks	Acceptance criteria employed for 6010: ± PQL	Acceptance criteria stated in 6010: less than 10% of PQL

TABLE 4

PREPARATION OF CALIBRATION AND QUALITY CONTROL STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Calibration Standard (STD1 or S1)	ICP- intermediate Standard	Lab Prepared (see Table 5)	10.0
	QCS 26	High Purity Standards	1.0
Initial Calibration Verification (ICV)	QCP-CICV-3	Inorganic Ventures	0.96
	1000 mg/L Si	Inorganic Ventures	0.98
	1000 mg/L Al	High Purity Standards	0.96
	IV-28	Inorganic Ventures	0.4
	1000 mg/L Sn	Inorganic Ventures	0.04
Interference Check Sample A (ICSA)	CLPP-ICS-A	Inorganic Ventures	10.0
Interference Check Sample AB (ICSAB)	CLPP-ICS-A	Inorganic Ventures	10.0
	CLPP-ICS-B4	Inorganic Ventures	1.0
	ICSAB-INT	Lab Prepared (see Table 5)	5.0
Continuing Calibration Verification (CCV)	ICP intermediate standard	Lab Prepared (see Table 5)	5.0
	QCS 26	High Purity Standards	0.5
Practical Quantitation Limit Sample (PQL)	PQL-INT	Lab Prepared (see Table 5)	1.0

TABLE 5

PREPARATION OF INTERMEDIATE STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
PQL-INT	1000 mg/L B,Li,Sn,Sr, W, U	H-P or IV	1.0 each
	10000 mg/L K, Na	H-P or IV	1.0 each
	1000 mg/L Ni	High Purity Standards	0.4
	1000 mg/L Co	High Purity Standards	0.3
	1000 mg/L Cu,V,Zn	High Purity Standards	0.25 each
	1000 mg/L Si	High Purity Standards	2.0
	1000 mg/L Cr,Ti,Tl,Ag	High Purity Standards	0.15 each
	1000 mg/L Cd,Se, Mo	High Purity Standards	0.1 each
	10000 mg/L Al	High Purity Standards	0.3
	1000 mg/L As,Sb	High Purity Standards	0.08 each
	1000 mg/L Ba,Be,Mn,Pb	High Purity Standards	0.05 each
	10000 mg/L Ca,Mg	High Purity Standards	0.05 each
	10000 mg/L Fe	High Purity Standards	0.1
ICSAB-INT	10000 mg/L K,Na	H-P or IV	4.0 each
	10000 mg/L B, Li, Mo,Sr,Sn,Ti, W, U	High Purity Standards	1.0 each
	1000 mg/L Si	High Purity Standards	4.0

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 6

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L								
	STD1	ICV	PQL	ICSA	ICSAB	CCV	AL_IEC	FE_IEC	MN_IEC
Aluminum	25	10	0.3	500	500	12.5	500		
Antimony	1	0.4	0.008		0.6	0.5			
Arsenic	1	0.4	0.008		0.1	0.5			
Barium	1	0.4	0.005		0.5	0.5			
Beryllium	1	0.4	0.005		0.5	0.5			
Boron	1	0.4	0.1		0.5	0.5			
Cadmium	1	0.4	0.01		1.0	0.5			
Calcium	25	10	0.05	500	500	12.5			
Chromium	1	0.4	0.015		0.5	0.5			
Cobalt	1	0.4	0.03		0.5	0.5			
Copper	1	0.4	0.025		0.5	0.5			
Iron	25	10	0.1	200	200	12.5		200	
Lead	1	0.4	0.005		0.05	0.5			
Lithium	1	0.4	0.1		0.5	0.5			
Magnesium	25	10	0.05	500	500	12.5			
Manganese	1	0.4	0.005		0.5	0.5			10
Molybdenum	1	0.4	0.01		0.5	0.5			
Nickel	1	0.4	0.04		0.5	0.5			
Potassium	25	13.6	1		20	12.5			
Selenium	1	0.4	0.01		0.05	0.5			
Silicon	1	0.4	0.2		2	0.5			
Silver	1	0.4	0.015		0.2	0.5			
Sodium	25	10	1		20	12.5			
Strontium	1	0.4	0.1		0.5	0.5			
Thallium	1	0.4	0.015		0.1	0.5			
Tin	1	0.4	0.1		0.5	0.5			
Titanium	1	0.4	0.015		0.5	0.5			
Tungsten	1	0.4	0.1		0.5	0.5			
Uranium	1	0.4	0.1		0.5	0.5			
Vanadium	1	0.4	0.025		0.5	0.5			
Zinc	1	0.4	0.025		1.0	0.5			

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 7

ELEMENT CONCENTRATIONS IN INTERMEDIATE STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L			
	ICP Intermed STD		PQL-INT	ICSAB-INT
Aluminum	240		30	
Antimony			0.8	
Arsenic			0.8	
Barium			0.5	
Beryllium			0.5	
Boron			10	10
Cadmium			1.0	
Calcium	240		5.0	
Chromium			1.5	
Cobalt			3.0	
Copper			2.5	
Iron	240		10	
Lead			0.5	
Lithium	10		10	10
Magnesium	240		5.0	
Manganese			0.5	
Molybdenum			1.0	10
Nickel			4.0	
Potassium	150		100	400
Selenium			1.0	
Silicon	250		20	40
Silver			1.5	
Sodium	240		100	400
Strontium	10		10	10
Thallium			1.5	
Tin	10		10	10
Titanium			1.5	10
Tungsten	10		10	10
Uranium	10		10	10
Vanadium			2.5	
Zinc			2.5	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 8

ELEMENT CONCENTRATIONS IN STOCK STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L					
	IV-28	QCS-26	2007 ICS-1	CLPP-ICS-A	CLPP-ICS-B4	QCP-CICV-3
Aluminum	100	100		5000		
Antimony	100	100			60	
Arsenic	100	100			10	500
Barium	100	100			50	
Beryllium	100	100			50	
Boron	100	100	500			
Cadmium	100	100			100	250
Calcium	100	100		5000		
Chromium	100	100			50	
Cobalt	100	100			50	
Copper	100	100			50	
Iron	100	100		2000		
Lead	100	100			5	500
Lithium	100					
Magnesium	100	100		5000		
Manganese	100	100			50	
Molybdenum	100	100	300			
Nickel	100	100			100	
Potassium	1000	1000				
Selenium	100	100			5	500
Silicon	50	50	230			
Silver	100	100			20	
Sodium	100	100				
Strontium	100					
Thallium	100	100			10	500
Tin						
Titanium	100	100	1000			
Vanadium	100	100			50	
Zinc	100	100			100	

TABLE 9

REQUIRED ANALYTICAL SEQUENCE

Sequence Number	Standard/Sample	Purpose
1	Blank (Calibration Blank)	Initial calibration
2	S1 (Calibration Standard)	Initial calibration
3	ICV (Initial Calibration Verification)	Check calibration accuracy
4	ICB (Initial Calibration Blank)	Check calibration accuracy
5	PQL (Practical Quantitation Level Sample)	Check calibration accuracy near PQL, repeat before final CCV, CCB
6	ICSA (Interference Check Solution A)	Verify accuracy of IEC factors, repeat before final CCV, CCB
7	ICSAB (Interference Check Solution AB)	Verify accuracy of IEC factors, repeat before final CCV, CCB
8	CCV (Continuing Calibration Verification)	Check calibration stability
9	CCB (Continuing Calibration Blank)	Check calibration stability
10-19	Analyze up to 10 samples	
20	CCV (Continuing Calibration Verification)	Check calibration stability
25	CCB (Continuing Calibration Blank)	Check calibration stability
…	Continue analyzing sequences of up to 10 samples, followed by a CCV and a CCB	

ATTACHMENT 1

HARDNESS BY CALCULATION

As referenced in "Standard Methods for the Examination if Water and Wastewater," Methods 2340 A & B, Hardness Introduction and Hardness by Calculation, American Public Health Association, 18th Edition, Revised 1992, total hardness is the sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in milligrams per liter.

Once the calcium and magnesium concentrations have been determined by EPA methods 6010, 6020, 200.7 or 200.8, the total hardness of an aqueous sample may be calculated as follows:

$$\text{Total Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L) + 4.118\ (Mg, mg/L)$$

The calcium hardness of an aqueous sample may also be calculated as follows:

$$\text{Calcium Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L)$$

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-720
Revision History
Cover Page
Page 1

TITLE: TOTAL SUSPENDED SOLIDS (NON-FILTERABLE RESIDUE) BY EPA METHOD 160.2 AND STANDARD METHODS 2540 D

Prepared By: Betsy Carbone Date: 12/97

Approved By:

Group Supervisor: Keith Tangney Date:

Operations Manager: John C. Burton Date: 1/24/01

QA Officer: Deborah J. Nadeau Date: 1.22.01

General Manager: Dennis P. Hughes Date: 1/25/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Format changes, added pollution prevention, added SM 2540D. Minor changes to sections 7+8 and Tables.	DN	1.22.01	1.22.01
02	Changed Title to reflect referenced methods. Added KIMS data entry. added new logbook. Revised text for clarity. Added Wording to Sect. 8	LAD	031605	031605
03	added definitions to section 1.	LAD	02/08	02/08
04	Added 1L sample volume required for lower PQL. Corrected balance requirement typo. Added method modification to Table 2.	LAD	06/08	06/08
05	Added requirement to record oven and thermometer IDS in logbook. Added references to section 18.	DN	08/09	08/09

TITLE: TOTAL SUSPENDED SOLIDS (NON-FILTERABLE RESIDUE) BY EPA METHOD 160.2 AND STANDARD METHODS 2540 D

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Updated or added references to section 10 and Table 1.	LAD	06/10	06/10

TITLE: TOTAL SUSPENDED SOLIDS (NON-FILTERABLE RESIDUE) BY EPA METHOD 160.2 AND STANDARD METHODS 2540 D

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Recipient: _____Date:_____

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STANDARD OPERATING PROCEDURE

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TITLE: TOTAL SUSPENDED SOLIDS (NON-FILTERABLE RESIDUE) BY EPA METHOD 160.2 AND STANDARD METHODS 2540 D

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure used by Katahdin Analytical Services, Inc. technical personnel to measure the concentration of total suspended solids (TSS) found in drinking and surface waters and domestic and industrial wastes as described in EPA Method 160.2 and SM 2540 D.

The procedure is applicable to measurement of TSS to a Practical Quantitation Limit (PQL) of 4 mg/L when 250 mL of sample is used for analysis.

1.1 Definitions

Total Suspended Solids – the portion of total solids retained by a standard glass fiber filter.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

LCS - Laboratory Control Sample - A standard or solid reference material of known value that has been brought through the sample preparation an analysis process. The LCS is used to assess the accuracy of the method.

MB – Method Blank - Reagent water that has been brought through the sample preparation and analysis process. The MB is used to assess contamination.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the MDL.

MDL - Method Detection Limit - The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in the analysis of Total Suspended Solids by EPA Method 160.2 and SM 2540 D. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in analysis of TSS by 160.2 and 2540 D to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the

lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, “Sample Disposal,” current revision.

2.0 SUMMARY OF METHOD

A well-mixed sample is filtered through a glass fiber filter, and the residue retained on the filter is dried to constant weight at 103-105°C. The filtrate from this procedure may be used to determine total dissolved solids (filterable residue). See Katahdin SOP CA-719, current revision.

TITLE: TOTAL SUSPENDED SOLIDS (NON-FILTERABLE RESIDUE) BY EPA METHOD 160.2 AND STANDARD METHODS 2540 D

3.0 INTERFERENCES

Filtration apparatus, filter material, pre-washing, post-washing, and drying temperature are specified because these variables have been shown to affect the results.

Samples high in total dissolved solids (filterable residue), such as saline waters, brines, and some wastes, may be subject to a positive interference from entrained dissolved solids. Washing of the filter to remove any dissolved solids entrained in the filter and suspended solids minimizes this potential interference.

Because excessive residue on the filter may form a water-entrapping crust, limit the sample size to that yielding no more than 200 mg of residue.

4.0 APPARATUS AND MATERIALS

4.1 Vacuum pump - capable of generating vacuum at 20-25 psi.

4.2 Oven - capable of maintaining a temperature between 98-105°C.

4.3 Glass fiber filter discs without organic binders, 47 mm diameter, nominal pore size 2.0 um or less. Gelman type A/E, Millipore type AP40, Whatman type 934-AH, or equivalent are acceptable.

4.4 Plastic filter funnel with filter support grid and magnetic closure, 300 mL capacity.

4.5 Filter flask with side arm, 500 mL capacity.

4.6 Analytical balance - capable of weighing to 0.1 mg; balance must be calibrated in accordance with Katahdin SOP, CA-102, Balance Calibration, before each measurement. Weights used are 5.0 g, 2.0 g, 0.2 g and readings should be recorded in the TSS Logbook (Figure 1).

4.7 Desiccator with conditioned indicating desiccant (desiccant is conditioned by drying at 210°C for one hour)

4.8 Graduated cylinders – 10, 25, 50, 100, 250 mLs.

4.9 Aluminum tins to hold filters for drying

5.0 REAGENTS

5.1 Laboratory reagent grade water: Water, which has been treated by the Culligan Water System and the Millipore Mill-Q™ Water System.

5.2 Laboratory Control Sample (LCS) solution - The LCS is a purchased non-filterable residue standard that is prepared according to the manufacturer's directions. The prepared standard has a certified reference value that is calculated for each standard from the measured weight. The LCS solution must be shaken before each use to suspend settled particulates.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples are collected in plastic or glass sample containers and to minimize microbiological decomposition of solids, stored at 4 (±2) °C until time of analysis

The holding time for this test is 7 days from the date of sampling. To obtain a PQL of 4 mg/L, 250 mL of sample is required.

7.0 PROCEDURES

7.1 Prepare glass filters as follows. Place the glass fiber filter on the filter funnel with the wrinkled side up. While vacuum is applied, wash the filter with three successive 20 mL volumes of laboratory reagent grade water. Remove all traces of water by continuing to apply vacuum after water has passed through. Remove filter and dry in oven at 103-105°C for one hour. After drying, place in desiccator until needed. Weigh the filter immediately before use. Record initial filter weight in TSS Logbook (Figure 1). Handle filter with forceps only.

7.2 Assemble filtering apparatus with a prepared, preweighed filter. Shake sample vigorously and quantitatively transfer 250 mL of sample, or another measured aliquot less than 250 mL, to the filter funnel using a graduated cylinder. Begin suction. Remove all traces of water by continuing to apply vacuum after sample has passed through. Record the initial sample volume in TSS Logbook.

Note: For clients who require a PQL of 1.0 mg/L, 1L of sample must be filtered.

Note: If the measured volume does not filter completely, the sample must be discarded and reanalyzed with a fresh filter. For these cases, it is advisable to prepare several extra filters ahead of time and store them in the desiccator.

Note: Since excessive residue may form a water trapping crust, samples in excess of 200 mg of residue must be rerun at a dilution.

7.3 With suction on, wash graduated cylinder, filter, non-filterable residue and filter funnel three times with approximate 10-mL volumes of laboratory reagent grade water, allowing complete draining between washings.

Note: If the sample filtrate is to be analyzed for total dissolved solids (TDS), remove the TDS aliquot from the filter flask before rinsing the filter.

7.4 Remove filter from filter funnel, and place in an aluminum tin. Place in the oven at 103-105°C for at least one hour. Record initial and final times and temperatures in TSS Logbook. Record the oven ID and thermometer ID in the TSS logbook.

7.5 Cool in desiccator to room temperature and weigh. Repeat cycle of heating, cooling, and weighing until a constant weight is obtained or until the difference between successive weighings is 0.5 mg or 4%, whichever is less. For each sample, method blank, and LCS, record the final two weights in the TSS logbook.

7.6 The initial sample volume and initial and final sample weights are entered manually into the Katahdin Information Management System (KIMS) for calculation and reporting. After the data are entered, a batch sheet (Figure 2) is automatically printed by KIMS. Refer to the current revision of Katahdin SOP CA-732 ("Wet Chemistry Data Entry and Review Using Katahdin Information Management System") for further information.

The final concentration of TSS is calculated by the KIMS using the following formula:

$$\text{TSS (mg/L)} = \frac{[\text{Wt of filter + residue (g)}] - [\text{Wt of filter (g)}]}{\text{Sample Volume (mL)}} * \frac{1000\ \text{mg}}{\text{g}} * \frac{1000\ \text{mL}}{\text{L}}$$

7.7 After completion of each test, the TDS Logbook must be signed and dated by the person performing the test. All unused lines are to be "z-ed" out and initialed and dated.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

See below and refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The Department Manager, Operations Manager, General Manager and/or Quality Assurance Officer may be consulted to evaluate data. Due to the 7-day hold time associated with this method, samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be reported after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work

performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 The analytical balance must be calibrated in accordance with Katahdin SOP, CA-102, Balance Calibration, before each measurement. Weights used are 5.0 g, 2.0 g, 0.2 g and readings should be recorded in the TSS logbook.

Acceptance Criteria:

ASTM Class 1 Weight	Acceptance Range
5.0 g	4.9998g - 5.0002g
2.0 g	1.9998g - 2.0002g
0.2 g	0.1998g - 0.2002g

8.2 A method blank, consisting of a blank filter with 250 mL of laboratory reagent grade water filtered through it, must be run for every batch of 20 or fewer samples.

Acceptance Criteria: Measured TSS < Practical Quantitation Limit
Corrective Action for Non-Conformance: Evaluate sample results. Report results <PQL. Report results >10x blank contamination with narration. Reanalyze all other samples. If samples cannot be reanalyzed, a Corrective Action Report (CAR) must be initiated and data appropriately qualified.

8.3 A duplicate sample must be run for every 10 samples.

Calculate the mean (X) and relative percent difference (RPD) for every duplicate run as follows:

$$X = \frac{X_1 + X_2}{2}$$

$$RPD = \frac{X_1 - X_2}{X} \times 100$$

where: X_1 = non-filterable residue (TSS) measurement for sample
X_2 = non-filterable residue (TSS) measurement for sample duplicate

Acceptance Criteria: Relative Percent Difference (RPD) $\leq$20%.

Corrective Action for Non-Conformance: Reanalysis of sample in duplicate; if RPD is again >20%, report original result with the appropriate flag or narration.

8.4 One Laboratory Control Sample (LCS) must be prepared and analyzed with every sample batch of twenty or fewer samples.

Acceptance criteria: 60-140% recovery of the true value for USEPA Method 160.2. These nominal acceptance criteria may change dependent on the actual true value of the LCS. The acceptance criteria for the LCS for SM 2540 D are 90-110% recovery of the true value. Some commercially available LCSs have true values so low that 90-110% acceptance criteria are not reasonable or achievable. The acceptance criteria should be evaluated with each new lot of LCS. If criteria are exceeded, samples must be evaluated and/or reanalyzed. If samples cannot be reanalyzed, a Corrective Action Report (CAR) must be initiated and data appropriately qualified.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined and filed with the Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revisions of USEPA Method 160.2 and SM 2540 D for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Method 160.2, Residue, Non-Filterable, "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020, Revised March, 1983.

Method 2540 D. Total Suspended Solids Dried at 103-105°C, "Standard Methods for the Examination of Water and Wastewater", 18th edition, 1992, APHA-AWWA-WPCF.
Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

TITLE: TOTAL SUSPENDED SOLIDS (NON-FILTERABLE RESIDUE) BY EPA METHOD 160.2 AND STANDARD METHODS 2540 D

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

LIST OF FIGURES & TABLES

TABLE 1 QC Requirements
TABLE 2 Summary Of Method Modifications
FIGURE 1 Example of TSS Logbook Page
FIGURE 2 Example of TSS KIMS Batch Sheet

TITLE: TOTAL SUSPENDED SOLIDS (NON-FILTERABLE RESIDUE) BY EPA METHOD 160.2 AND STANDARD METHODS 2540 D

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Total Suspended Solids, TSS (Non-Filterable Residue) / USEPA Method 160.2 and Standard Methods 2540 D	Method blank	One per prep batch of 20 or fewer samples	Measured TSS <PQL	1) Investigate source of contamination 2) Evaluate sample results. Report results <PQL. Report results >10x blank contamination with narration. 3) Reprep a blank and all remaining samples.
	LCS	One per prep batch of 20 or fewer samples	60-140% (EPA 160.2) 90-110% (SM 2540D)	1) Investigate source of problem. 2) If the LCS recovery is high report sample results <PQL with narration. 3) Reprep a blank and all remaining samples.
	Sample duplicate	One duplicate every ten samples	RPD $\leq$20	1) Investigate problem and reanalyze sample in duplicate 2) If RPD still >20, report original result with notation.
	Initial Demonstration of Performance: Precision and accuracy study using 4 replicate analyses LCS	Initially once per analyst, then annually per analyst.	All recoveries within method QC acceptance limits	Investigate source of problem; rerun P & A study.
	MDL Study and/or LOD/LOQ Verification.	Refer to KAS SOP QA-806, “Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications”, current revision.		

TITLE: TOTAL SUSPENDED SOLIDS (NON-FILTERABLE RESIDUE) BY EPA METHOD 160.2 AND STANDARD METHODS 2540 D

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-720-06	USEPA method 160.2 or SM 2540 D, current revisions
Apparatus/Materials	None	
Reagents	None	
Sample preservation/ handling	None.	
Procedures	7.1 ……Remove filter and dry in oven at 103-105°C for one hour. After drying, place in desiccator until needed. Weigh the filter immediately before use.	SM2540 D, 3.a. ……Repeat cycle of drying or igniting, cooling desiccating and weighing until a constant weight is obtained or until weight change is less than 4% of the previous weighing or 0.5 mg.
QC - Spikes	None	
QC - LCS	None	
QC - Accuracy/Precision	None.	
QC - MDL	None	

TITLE: TOTAL SUSPENDED SOLIDS (NON-FILTERABLE RESIDUE) BY EPA METHOD 160.2 AND STANDARD METHODS 2540 D

FIGURE 1

EXAMPLE OF TSS LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES, INC.

TOTAL DISSOLVED SOLIDS/TOTAL SUSPENDED SOLIDS

Total Disolved Solids: EPA 160.1____ Total Dissolved Solids: SM 2540C____ PQL: 10mg/L

Total Suspended Solids: EPA 160.2____ Total Suspended Solids: SM 2540D✓ PQL: 4mg/L

Aqueous Solids LCS Sand ID/Lot# W9018 TV-831 exp 4/12

ASTM Class Weights			Oven Temperatures (C)		
Balance ID: METTLER AS200			Oven ID: 107N0042 Filter		
True WT (g)	Initial WT (g)	Final WT (g)	In Temp. 103	Date: 6/10/10	Time: 1428
0.2000	0.2002	0.2000	Out Temp. ↓	Date: 6/11/10	Time: 830
2.0000	2.0000	2.0000	In Temp.	Date: ↓	Time: 950
5.0000	5.0000	5.0000	Out Temp. ↓	Date: ↓	Time: 1050
10.0000					
100.0000					

Notes: W678483

KAS Sample ID	Sample Volume (ml)	Time of Initial Aliquot	Filter or Dish ID	Filter or Dish Wt. (g)	Dry 1 Dish/Filter + Sample Residue Wt. (g)	Dry 2 Dish/Filter + Sample Residue Wt. (g)	Time of Final Weighing (Analysis)
Blank WG	100	1400	25	1.1025	1.1025	1.1025	1146
LCS WG	100	1402	26	1.1261	1.2090	1.2088	1147
SD 3272-3A	250	1404	27	1.0997	1.1000	1.0999	1148
DUP WG -3A	250	1406	28	1.0893	1.0897	1.0896	1149
SD 3241-3A	250	1408	29	1.1241	1.1248	1.1248	1150
SD 3277-1A	220	1410	30	1.1254	1.1276	1.1275	1151
SD 3287-1A	990	1412	31	1.0995	1.0997	1.0997	1152
↓ -2A	960	1414	32	1.1539	1.1749	1.1749	1153
SD 3258-1A	250	1416	33	1.1420	1.1658	1.1658	1154
↓ -2A	260	1418	34	1.1106	1.1146	1.1146	1155
SD 3298-1A	960	1420	35	1.1308	1.1310	1.1310	1156
↓ -2A	980	1422	36	1.1026	1.1076	1.1076	1157
SD 3295-1A	250	1424	37	1.0699	1.0716	1.0714	1158
DUP WG -1A	250	1426	38	1.0770	1.0789	1.0788	1159
SD 3296-1A	160	1428	39	1.0991	1.1173	1.1172	1200

Analyst: [signature] Date: 6-10-10 / 6-11-10

Checked By: Date:

WL-012 - Revision 1 - 12/10/2009 QAWL576 0000083

TITLE: TOTAL SUSPENDED SOLIDS (NON-FILTERABLE RESIDUE) BY EPA METHOD 160.2 AND STANDARD METHODS 2540 D

FIGURE 2

EXAMPLE OF KIMS TSS BATCH SHEET

WET CHEMISTRY BATCH REPORT
Feb 22 2008, 04:27 pm
Batch: WG48593

Parameter: Solids-Nonfilterable Residue

Date Analyzed: 21-FEB-08

Analyst Initials: JF

Prep Date: 20-FEB-08

Prep Method: SM 2540D

Prep Chemist: JF

Sample	Samp Type	Method	Tare Amt.	Final Amt.	Rpt. DF	Result	Rpt Result	TS(%)	PQL	MDL	Adj PQL	RPD	%Rec
SB0845-1	SAMP	SM 2540D	0.1271000g	0.1351000g	25	800	800 mg/L	NA	4	26	100		
SB0846-1	SAMP	SM 2540D	0.1279000g	0.1362000g	2.9	97.64706	98. mg/L	NA	4	3	12		
SB0862-1	SAMP	SM 2540D	0.1244000g	0.1246000g	1	.8	U4.0 mg/L	NA	4	1.026	4		
SB0867-1	SAMP	SM 2540D	0.1257000g	0.1259000g	1	.2	U1.0 mg/L	NA	1	.26	1		
SB0867-2	SAMP	SM 2540D	0.1262000g	0.1309000g	1	4.7	4.7 mg/L	NA	1	.26	1		
SB0871-1	SAMP	SM 2540D	0.1266000g	0.1405000g	1	55.6	56. mg/L	NA	4	1.026	4		
WG48593-1	MBLANK	SM 2540D	0.1261000g	0.1260000g	1	-.1	U1.0 mg/L	NA	4	1.026	4		
WG48593-2	LCS	SM 2540D	0.1280000g	0.1353000g	2.5	73	73. mg/L	NA	4	2.6	10		97
WG48593-3	DUP	SM 2540D	0.1248000g	0.1327000g	25	790	790 mg/L	NA	4	26	100	1	

Comments:

WG48593-1 SB0845-1
WG48593-2 SB0845-1
WG48593-3 SB0845-1

Entered by: _____ Date: 2-22-08 Accepted by: CP Date: 2/22/08

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II LLOYD KAHN METHOD

Prepared By: Greg Lull Date: 7/2002

Approved By:

Group Supervisor: Keith Tanguay Date: 091102

Lab Operations Mgr: Joel C. Burton Date: 9/11/02

QA Officer: Deborah J. Nadeau Date: 9.11.02

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Sect. 1-clarified CCB, Sect. 2-clarified phosphoric acid conc. Sect. 7- reworded software and instr. start up, added TS to calc., removed steps not currently in practice. Sect. 8 -updated LCS and CCV information. Updated Table 1	LAD	01/07	01/07
02	Added method SW846 9060 to title and Table 2. Added 9060 quadruplicate sample analysis to sect. 7 and Table 1. Removed D.I. water from MB definition 5.3-added TIC std. info. Sect. 7.0 - rewrote for clarity on instrument and software instructions. Fixed typos- sect 2.	LAD	06/08	06/08
03	Minor changes to reflect current equipment, practices and techniques.	LAD	08/09	08/09
04	Added 9060 QC requirements to Table 1. Added method modifications to Table 2. Minor edits to section 7 to reflect current practice, remove redundancy and for clarification. Updated and/or added references to section 7, 9 and 10.	LAD	06/10	06/10

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

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Recipient: _____Date:_____

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

1.0 SCOPE AND APPLICATION

This SOP describes the procedures used by Katahdin Analytical Services, Inc. technical personnel to determine Total Organic Carbon (TOC) in solids in accordance with EPA Region II Lloyd Kahn method.

This method is applicable to sediment, sludges, and soil samples. The detection limit for this method is 100 µg C and a method PQL of 400 ug/g.

1.1 Definitions/Acronyms

TC – Total carbon

IC – Inorganic Carbon

TOC – Total Organic Carbon

Method Blank – An empty sample boat with no reagents added that is carried through the entire analytical procedure in the same manner as a sample.

LCS/ICV - Laboratory Control Sample/ Initial Calibration Verification. One LCS/ICV per batch is prepared from a separate source from the CCV and calibration curve standards. LCS/ICV verifies the calibration curve.

CCV - Continuing Calibration Verification. The CCV is made from the same source as the calibration. One CCV is run every ten samples.

CCB - Continuing Calibration Blank. The CCB is an empty sample boat with no reagents added. One CCB is run every ten samples.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of TOC in solids by the Lloyd Kahn Method. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in analysis of TOC in solids by the Lloyd Kahn method to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

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It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method have not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their department manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in adherence with the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, Sample Disposal, current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP SD-903.

2.0 SUMMARY OF METHOD

Total Carbon (TC) is measured utilizing a carbonaceous analyzer with a boat sampling module and 900°C furnace attached. The resulting combustion converts carbon-to-carbon dioxide (CO_2) in the presence of oxygen. The amount of CO_2 derived from a sample is directly proportional to the concentration of carbonaceous material in the sample and is then measured by a non-dispersive infrared detector (NDIR).

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To determine **Total Organic Carbon** (TOC), however, carbonate and bicarbonate ions contributing to the TC result must be accounted for. This is achieved by adding 1:1 phosphoric acid to the sample and combusting it at 103° C for 10 minutes to remove any **Inorganic Carbon** (IC) before analyzing the sample. The **Total Carbon** result then equals the **Total Organic Carbon.**

3.0 INTERFERENCES

Volatile organics in the sediment may be lost in the decarbonation step resulting in a low bias.

4.0 APPARATUS AND MATERIALS

4.1 Shimadzu model TOC-Vcph with NDIR.

4.2 SSM-5000A 900°C furnace with boat sampling module.

4.3 Mettler AE 100 balance (accurate to 0.1 mg) or equivalent.

4.4 Ceramic boats.

4.5 Drying oven capable of maintaining 103-105°C

4.6 Oxygen gas

5.0 REAGENTS

5.1 Dextrose Solid (40% Carbon by weight = 400,000 ug/g C)

Calibration Standards:

0.0 mg - Calibration Blank
1.0 mg - 400ugC
5.0 mg - 2000ugC
10.0 mg - 4000ugC
40.0 mg - 16000ugC
60.0 mg - 24,000ugC

(Upper range limit for TC is 30,000ugC)

5.2 Dextrose Solid (40% Carbon by weight = 400,000 ug/g C) used as LCS run at 16000-20000ugC (40-50mg). Standard must be a source different from CCV source.

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5.3 Sodium Carbonate, anhydrous (11.3% Carbon by weight = 113,000 ug/gC) Calibration Standards:

0.0 mg - Calibration Blank
3.5 mg - 400ugC
17.7 mg - 2000ugC
35.3 mg - 4000ugC
70.7 mg - 8000ugC
212.0 mg - 24,000ugC

(Upper range limit for IC is 25,000 ugC)

5.4 1:1 Phosphoric acid / DI water solution

5.5 Sodium Hydrogen Carbonate (14.28% Carbon by weight = 142,857ug/gC) used as LCS run at 4000ugC level (28 mg).

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Collect sediments in a glass jar with Teflon or aluminum foil. Cool and maintain at 4° (±2) C. Analyze within 14 days for Lloyd Kahn and 28 days for SW846 9060.

7.0 PROCEDURES

SET UP AND CALIBRATION

7.1. Turn on TOC-Vcph analyzer, SSM-5000Afurnace, and oxygen supply and connect to the TOC software program.

7.2. Start the TOC-V software program by double clicking the TOC-V icon on the desktop and selecting sample table editor. Enter user name, passwords and press **OK**. Click the “new” icon followed by “sample run”. Choose SSM-5000A from the pull down menu. This activates a sample field spreadsheet in which calibrations, controls, and samples can be inserted and run. Click the “connect” icon and choose “Settings on PC”. This will start the gas flows through both units and will activate the NDIR. Wait for the TC furnace to read 900°C before beginning analysis. The gauges on the soil module for the gas lines should read 150psi and 200psi from right to left. Also place any sample boats that will be used in the furnace for several minutes to bake off any remaining residue.

7.3. To run a calibration, minimize the sample run. Click the “New” icon followed by “calibration curve”. This activates the calibration curve wizard.

7.4. Choose SSM-5000A next to system by using the pull down menu, the click **NEXT** twice. Type in the file name specifying that it is a curve in the name (i.e toccurve030510), and then click **NEXT**. Using the pull down menu select ug as the units and click **NEXT**.

7.5. A 6-point curve (for either TC or IC) must be run at least every 3 months to verify the calibration. The calibration may also be updated as necessary as demonstrated by failure of the Laboratory Control Sample (LCS) or Continuing Calibration Verification (CCV). To add the points to the curve select "(1)" then click **ADD**. In the highlighted **Conc:** box type in the concentration of the first point (24000), and then press **OK**. Click on the next point "(2)" and **ADD**. Continue with previous steps until all 6-points have been entered. Click **NEXT** twice and then **FINISH**. Now the curve needs to be run.

7.6. Maximize the sample run and highlight the first row. Select insert then calibration curve from the top menu. Select the calibration curve that was just saved and click **OK**. The calibration curve will be renamed with the date and time (i.e toccal030510, note: name must include the word curve or cal, whichever was not used in the original file name) when the start button is clicked.

7.7. Once all the boxes on the top of the sample run say "Ready", click the start icon (traffic light) on the menu bar. Once the calibration has been started the instrument will prompt two windows.

7.8. The first will state which point will be running, verify the point and press **OK**. The second window will ask for the exact amount of dextrose that has been weighed out. Enter the amount and click OK. For the last point, the blank point, an empty sample boat is used and 500mg is used for the weight.

7.9. Once entered click **OK** and the instrument will prompt a message to move the sample boat to the measure position.

7.10. Open the TC sample port and insert the boat. Close the port tight and set timer for two (2) minutes. These two minutes ensures that the CO2 that entered the instrument has had enough time to go completely through the detector and not give any false positives. When the timer goes off slide the sample boat to the measurement position.

7.11. Once the instrument is done analyzing the sample, about 4-5 minutes, there will be a message prompt to slide the sample boat to the cooling position. After a few minutes it will then prompt again to move the sample boat back in to the port. As soon as the boat has been pulled all the way back the instrument will automatically start the process for the next point. Repeat steps 7.8 to 7.10 for the rest of the calibration.

7.12. When the entire curve is complete, print the sample report and check the curve and its linearity. The correlation coefficient must be greater than or equal to 0.995. The value of the calibration checks must fall within the control limits (80-120% recovery). If not

rerun the sample up to two more times. If the calibration check is still out of the acceptable recovery range, recalibrate the instrument and repeat the procedure. If problem persists, remake the standards and repeat the procedure or perform instrument maintenance. If maintenance is required, record in TOC instrument maintenance notebook.

7.13. Once there is a valid calibration curve, the curve must be saved to the method. Click file, then open. Select method from the pull down menu and select tc method soils. Click the SSM-TC tab and change the calibration to the new one. Click OK. Samples are now ready to run. Select “new” file icon on the menu bar and, then click on sample run. Highlight the first row on the sample run. Click the insert sample icon. The parameter box opens, click the method box and select tc method soils. Click next and enter the sample name. Click next twice then finish.

7.14. Using a calibrated analytical balance weigh out 100-500 mg, wet weight, of the sample into a tarred ceramic boat. Using a disposable pipet add 1:1 phosphoric acid to the sample until there is no more effervescing, usually 2-4 drops. If a sample concentration is greater than 24000 ug/g, a smaller sample amount must be used to achieve a concentration that is within the curve. Samples that require less than 50 mg must be analyzed in quadruplicate and the standard deviations calculated.

NOTE: Please refer to Katahdin Analytical Services SOP CA-108, “Basic Laboratory Technique”, current revision, for more information on subsampling

NOTE: Samples analyzed by method SW846 9060 must be analyzed in quadruplicate.

7.15. Dry the sample in a 103° - 105° C oven for ten minutes. This process removes the inorganic carbon from the sample. For the sample blanks, LCS, and/or CCV, no 1:1 phosphoric acid or drying is needed. For samples that require TC or Percent TC, 1:1 phosphoric acid nor drying is needed.

7.16. To start the analysis, click the start icon. The first sample entered will bring up a file name box. Name the file by analysis and date, click OK. Then the box to enter the weight will open. Enter weight and press OK. Follow steps 7.10 and 7.11 with the exception that each sample is now entered separately throughout the batch.

7.17. Calculate the TC or IC (whichever is being preformed) concentration using the following equation:

$$\frac{\text{Abs C value (instrument reading) in ug}}{\text{Sample Weight (g)}} \times \frac{100}{\%TS} = \text{TC or IC result in ug/g C}$$

7.18. Workgroup samples and get run ID. Enter true values for the LCS and MS and save. Go back to the spreadsheet and enter “LLOYD”, “Percent”, or “TOC” in the comments section for the samples you wish to report. Change QC to match workgroup. Data is then exported by selecting the ASCII export option from the file

menu. Select “save as” and choose parsefiles on LVSlims. Select TOC and type file name. Click save and wait for data to export, then review data in wetrev.\

7.19. A batch sheet is generated (Figure 4). Raw data, calibrations, and batch sheets are reviewed for completeness and accuracy by the Wet Chemistry supervisor or other qualified designee.

7.20. Analysts file printouts of instrument calibrations and sample data in the lab for approximately 3 months for reference. Prior calibrations are archived and all are available for retrieval**.**

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

See below and refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed below and in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases “qualified” data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 A Method Blank is analyzed at the beginning of the run and a CCB is analyzed every ten samples thereafter and at the end of the run. The Method Blank and CCBs are boat blanks. The boat should be analyzed empty. Results must not be greater than the reporting limit (PQL). The PQL for this test is 400mg/kg.

8.2 Analyze an LCS (40-50 mg of Dextrose = 16000-20000ugC for TC) / (28 mg Sodium Hydogen Carbonate = 4000 ug C for IC) with each batch of 20 samples. Acceptance criteria is 80-120% of expected value.

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8.3 Run a CCV (40-50 mg Dextrose = 16000-20000 ug C for TC) / (35.3 mg Hydrogen Carbonate = 4000 ug C for IC) every 10 samples and at the end of each batch. Acceptance criteria is 80-120% of expected value.

8.4 Run a duplicate every 20 samples. Run a matrix spike every 10 samples by weighing out the sample and adding 10 mg of dextrose to it for the TC spike or 35.3 mg of hydrogen carbonate for the IC spike. Run a matrix spike duplicate every 10 samples for SW 9060 samples. The recovery can be determined by calculating the theoretical yield from the sample result based on the weight as compared to the native result and adding 4000 ug C that was added from the spike component. The actual yield divided by the theoretical yield will give the recovery.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL's are determined per type of instrument filed with the Inorganic Department Manager and with the QAO. The MDL standards are prepared following the procedures outlined in this SOP.

Refer to the current revision of Katahdin SOP QA-806, "Method Detection Limit and Instrument Detection Limit and Reporting Limit Studies and Verifications", for procedures on determining the MDL. Refer to the current revision of EPA SW846 9060 and the Lloyd Kahn method for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Determination of Total Organic Carbon in Sediment, Lloyd Kahn, USEPA Region II, 7/88.

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods", SW-846, third Edition, Final Update III, December 1996, Method 9060.

TOC-V series SSM-5000A user's manual.

Installation and Operation of Shimadzu's Solid Sample Module.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.
The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision

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Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision

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TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Method blank	One per prep batch	No analyte detected >PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are <PQL or > 10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
LCS/ICV	One per prep batch of twenty samples (Lloyd Kahn); one per fifteen samples (SW 9060)	80-120% recovery	(1) Investigate source of problem. (2) If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.
CCV	CCV at the beginning of the analysis and one after every 10 samples: same conc. as LCS/ICV	80-120% recovery	(1) Investigate source of problem. (2) If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reanalyze all samples back to last acceptable CCV recovery
Sample Quadruplicate	One every twenty samples. SW 9060 – all samples are analyzed in quadruplicate	≤ 30% RPD	(1) If lab QC in criteria and matrix interference suspected, flag data (2) Else, reanalyze
Matrix spike/Matrix spike duplicate	One MS per ten samples(MSD required every 10 samples for SW 9060)	75-125% recovery	(1) If LCS in criteria and matrix interference suspected, flag data (2) Else, reanalyze
Demonstration of analyst proficiency; accuracy and precision	One time demonstration by each analyst performing the method	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file
MDL and-or LOD/LOQ Verification study	Refer to KAS SOP QA-806, “Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications”, current revision.		

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TABLE 2

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-741-04	METHOD LLOYD KAHN/SW846 9060
Apparatus/Materials		
Reagents	Dextrose solid for TC calibration (Dextrose for LCS) Sodium Carbonate for IC calibration (Sodium Hydrogen Carbonate for LCS)	Potassium Hydrogen Phthalate solution used for calibration.
Sample preservation/ handling		
Procedures	Report one of the duplicate samples using an RPD of 30%.	Report average and range of quadruplicate analysis (SW 9060) Use 3 standard deviation limit (Lloyd Kahn)
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

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FIGURE 1

EXAMPLE OF TOC SOLIDS LOGBOOK PAGE

W662605
R96624

Katahdin Analytical Services, Inc.

Carbon Analysis of Solid Samples - Shimadzu TOC-V_{CPH} / SSM-5000A

Analysis Type and Method (Check One)

____Total Carbon (SW846 9060M)
____Total Organic Carbon (SW846 9060)
✓ Total Organic Carbon (Lloyd Kahn)
____Total Inorganic Carbon (SW846 9060M)
____Other (Specify):

Spiking Information	
LCS Spike Source ID / Compound:	Dextrose SWL1628
CCV Spike Source ID / Compound:	Dextrose SWL413
MS Spike Source ID / Compound:	Dextrose SWL1628

Calibration Information	
Calibration Date:	3/26/09
Calibration Analyst:	CP

Calibrated w/SWL413

	KATAHDIN Sample Number	Sample Wt. (mg)	Sample Type * (Circle One)	Spike Added (mg)	
	CCV		Wet Dry	34.4 W=13760	104%
	Blank		Wet Dry		
	LCS		Wet Dry	35.9 W=14360	105%
	SC1250-7A	495.5	Wet Dry		
	-7Dup	476.0	Wet Dry		
2	-7Dup	498.2	Wet Dry		
1	-7Dup	498.7	Wet Dry		
	-7MS	472.6	Wet Dry	13.8 W=5520	
	-8A	476.8	Wet Dry		
	-9A	465.9	Wet Dry		
1	-10A	492.1	Wet Dry		
2	-11A	455.5	Wet Dry		
	-12A	484.8	Wet Dry		
	CCV		Wet Dry	35.1 W=14040	99%
	CCB		Wet Dry		
1	SC1251-1A	492.8	Wet Dry		
2	-2A	496.0	Wet Dry		
1	-3A	502.0	Wet Dry		
2	-4A	483.0	Wet Dry		
	-5A	476.1	Wet Dry		
	-6A	487.6	Wet Dry		
	-6MS	499.0	Wet Dry	12.7 W=5080	

* "Wet" = field-moist sample (as received). "Dry" = oven-dried sample.

Analyst:	CP	Analysis Date:	3/30/09
Reviewer:	[illegible]	Review Date:	3/31/09

QAWL531 0000018

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FIGURE 2

EXAMPLE OF TOC SOLIDS INSTRUMENT PRINTOUT

	Analysis	Sample Name	Diluti	Result	Comment	Status	Date / Time
1	SSM-TC	CCV	1.00	SSM-TC:14294 ug		Completed	03/30/09 09:00:24 A
2	SSM-TC	BLANK	1.00	SSM-TC:0.000 ug		Completed	03/30/09 09:07:05 A
3	SSM-TC	LCS	1.00	SSM-TC:15095 ug		Completed	03/30/09 09:16:07 A
4	SSM-TC	SC1250-7	1.00	SSM-TC:1919 ug		Completed	03/30/09 09:26:29 A
5	SSM-TC	SC1250-7DUP	1.00	SSM-TC:1961 ug	Heart	Completed	03/30/09 09:36:49 A
6	SSM-TC	SC1250-7DUP	1.00	SSM-TC:1903 ug		Completed	03/30/09 09:46:32 A
7	SSM-TC	SC1250-7DUP	1.00	SSM-TC:2365 ug		Completed	03/30/09 09:56:21 A
8	SSM-TC	SC1250-7MS	1.00	SSM-TC:8509 ug		Completed	03/30/09 10:13:37 A
9	SSM-TC	SC1250-8	1.00	SSM-TC:1959 ug		Completed	03/30/09 10:23:05 A
10	SSM-TC	SC1250-9	1.00	SSM-TC:841.8 ug		Completed	03/30/09 10:34:25 A
11	SSM-TC	SC1250-10	1.00	SSM-TC:2666 ug		Completed	03/30/09 10:45:22 A
12	SSM-TC	SC1250-11	1.00	SSM-TC:3067 ug		Completed	03/30/09 10:57:14 A
13	SSM-TC	SC1250-12	1.00	SSM-TC:4327 ug		Completed	03/30/09 11:07:26 A
14	SSM-TC	CCV	1.00	SSM-TC:13850 ug		Completed	03/30/09 11:16:49 A
15	SSM-TC	CCB	1.00	SSM-TC:0.000 ug		Completed	03/30/09 11:23:41 A
16	SSM-TC	SC1251-1	1.00	SSM-TC:1449 ug		Completed	03/30/09 12:07:43 P
17	SSM-TC	SC1251-2	1.00	SSM-TC:2138 ug		Completed	03/30/09 12:17:09 P
18	SSM-TC	SC1251-3	1.00	SSM-TC:507.5 ug		Completed	03/30/09 12:25:18 P
19	SSM-TC	SC1251-4	1.00	SSM-TC:3697 ug		Completed	03/30/09 12:36:14 P
20	SSM-TC	SC1251-5	1.00	SSM-TC:818.3 ug		Completed	03/30/09 12:46:44 P
21	SSM-TC	SC1251-6	1.00	SSM-TC:3954 ug		Completed	03/30/09 01:03:50 P
22	SSM-TC	SC1251-6MS	1.00	SSM-TC:9405 ug		Completed	03/30/09 01:15:23 P
23	SSM-TC	SC1251-7	1.00	SSM-TC:1793 ug		Completed	03/30/09 01:23:57 P
24	SSM-TC	SC1251-8	1.00	SSM-TC:5474 ug		Completed	03/30/09 01:34:44 P
25	SSM-TC	SC1251-9	1.00	SSM-TC:1089 ug		Completed	03/30/09 01:43:48 P
26	SSM-TC	CCV	1.00	SSM-TC:18080 ug		Completed	03/30/09 01:54:34 P
27	SSM-TC	CCB	1.00	SSM-TC:0.000 ug		Completed	03/30/09 02:05:24 P
28	SSM-TC	SC1279-1	1.00	SSM-TC:1812 ug		Completed	03/30/09 02:15:21 P
29	SSM-TC	SC1279-2	1.00	SSM-TC:415.7 ug		Completed	03/30/09 02:27:26 P
30	SSM-TC	CCV	1.00	SSM-TC:15168 ug		Completed	03/30/09 02:51:32 P
31	SSM-TC	CCB	1.00	SSM-TC:0.000 ug		Completed	03/30/09 03:01:10 P
* 32	SSM-TC	SC1303-4	1.00	SSM-TC:514.5 ug		Completed	03/30/09 02:39:49 P

note time

03/30/09 03:01:25 PM 1/1

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FIGURE 3

EXAMPLE OF TOC SOIL INSTRUMENT RESULTS PRINTOUT

CP 03/30/09 03:01:28 PM LLOYD033009.132

Sample

Sample Name: LCS
Sample ID: <Untitled>
Origin: tc method SOILS.met
Chk. Result

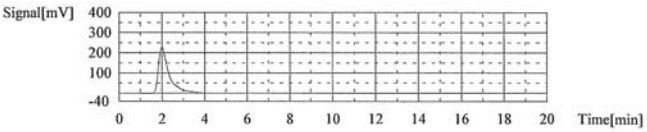
Type	Anal.	Dil.	Density	Result
Unknown	SSM-TC	1.000	1.000mg/uL	SSM-TC:15095 ug

1. Det

Anal.: SSM-TC

No.	Area	CNV	Abs C	Conc.	Weight	Volume	Ex.	Cal. Curve	Date / Time
1	780.7	780.7	15095ug	15095ug	35.90mg	35uL		toccurve032609.2009_03_26_08_19_31.cal	03/30/09 09:16:07 AM

Mean Area 780.7
Mean CNV 780.7
Mean Conc. 15095ug



Sample

Sample Name: SC1250-7
Sample ID: <Untitled>
Origin: tc method SOILS.met
Chk. Result

Type	Anal.	Dil.	Density	Result
Unknown	SSM-TC	1.000	1.000mg/uL	SSM-TC:1919 ug

1. Det

Anal.: SSM-TC

No.	Area	CNV	Abs C	Conc.	Weight	Volume	Ex.	Cal. Curve	Date / Time
1	99.27	99.27	1919ug	1919ug	495.5mg	495uL		toccurve032609.2009_03_26_08_19_31.cal	03/30/09 09:26:29 AM

Mean Area 99.27
Mean CNV 99.27
Mean Conc. 1919ug

Sample

Sample Name: SC1250-7DUP
Sample ID: <Untitled>
Origin: tc method SOILS.met
Chk. Result

Type	Anal.	Dil.	Density	Result
Unknown	SSM-TC	1.000	1.000mg/uL	SSM-TC:1961 ug

2/15

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FIGURE 4

EXAMPLE OF TOC SOLIDS BATCH SHEET

WET CHEMISTRY BATCH REPORT
Mar 30 2009, 03:04 pm
Batch: WG62105

Parameter: TOC In Soil

Date Analyzed: 30-MAR-09

Analyst Initials: CP

Prep Date: N/A

Prep Method: N/A

Prep Chemist: N/A

Sample	Samp Type	Method	Initial Amt.	Final Amt.	Rpt. DF	Result	Rpt Result	TS(%)	PQL	MDL	Adj PQL	RPD	%Rec
SC1250-10	SAMP	LLOYDKAHN	492.1mg	200.00ug	1	7203.58218	7200 ug/gdrywt	75.	400	110	530		
SC1250-11	SAMP	LLOYDKAHN	455.5mg	200.00ug	1	8528.51191	8500 ug/gdrywt	79.	400	110	510		
SC1250-12	SAMP	LLOYDKAHN	484.8mg	200.00ug	1	10774.69944	11000 ug/gdrywt	83.	400	100	480		
SC1250-7	SAMP	LLOYDKAHN	495.5mg	200.00ug	1	4979.75583	5000 ug/gdrywt	78.	400	110	510		
SC1250-8	SAMP	LLOYDKAHN	476.8mg	200.00ug	1	5226.01526	5200 ug/gdrywt	79.	400	110	510		
SC1250-9	SAMP	LLOYDKAHN	465.9mg	200.00ug	1	2216.01214	2200 ug/gdrywt	82.	400	100	490		
SC1251-1	SAMP	LLOYDKAHN	492.8mg	200.00ug	1	3373.92386	3400 ug/gdrywt	87.	400	97	460		
SC1251-2	SAMP	LLOYDKAHN	496mg	200.00ug	1	5041.85542	5000 ug/gdrywt	85.	400	99	470		
SC1251-3	SAMP	LLOYDKAHN	502mg	200.00ug	1	1236.52263	1200 ug/gdrywt	82.	400	100	490		
SC1251-4	SAMP	LLOYDKAHN	483mg	200.00ug	1	9088.17685	9100 ug/gdrywt	84.	400	100	470		
SC1251-5	SAMP	LLOYDKAHN	476.1mg	200.00ug	1	2173.96259	2200 ug/gdrywt	79.	400	110	500		
SC1251-6	SAMP	LLOYDKAHN	487.6mg	200.00ug	1	9419.11655	9400 ug/gdrywt	86.	400	98	460		
SC1251-7	SAMP	LLOYDKAHN	506.8mg	200.00ug	1	3980.78714	4000 ug/gdrywt	89.	400	95	450		
SC1251-8	SAMP	LLOYDKAHN	484mg	200.00ug	1	13394.34538	13000 ug/gdrywt	84.	400	100	470		
SC1251-9	SAMP	LLOYDKAHN	487.2mg	200.00ug	1	2569.51566	2600 ug/gdrywt	87.	400	97	460		
SC1279-1	SAMP	LLOYDKAHN	495.3mg	200.00ug	1	4584.50464	4600 ug/gdrywt	80.	400	110	500		
SC1279-2	SAMP	LLOYDKAHN	488.2mg	200.00ug	1	1120.16745	1100 ug/gdrywt	76.	400	110	530		
SC1303-4	SAMP	LLOYDKAHN	500.2mg	200.00ug	1	1176.05397	1200 ug/gdrywt	87.	400	97	460		
WG62105-1	MBLANK	LLOYDKAHN	500mg	200.00ug	1	0	U400 ug/gdrywt	NA	400	85	400		
WG62105-2	LCS	LLOYDKAHN	35.9mg	200.00ug	1	420473.5376	420000 ug/gdrywt	NA	400	85	400		105
WG62105-3	DUP	LLOYDKAHN	476mg	200.00ug	1	5297.21223	5300 ug/gdrywt	NA	400	110	510	6	
WG62105-4	MS	LLOYDKAHN	472.6mg	200.00ug	1	23150.56203	23000 ug/gdrywt	NA	400	110	510		121
WG62105-5	MS	LLOYDKAHN	499mg	200.00ug	1	21892.50498	22000 ug/gdrywt	NA	400	98	460		105

Comments:

SC1279-2	MS/MSD for Metals only
WG62105-1	SC1250-7
WG62105-2	SC1250-7
WG62105-3	SC1250-7
WG62105-4	SC1250-7
WG62105-5	SC1251-6

Entered by: CP Date: 3/30/09 Accepted by: _____ Date: _____

EMSL Analytical S.O.P.

ASTM DRAFT Sieve Method

Determination of Asbestos in Soil

1.0 Method Description

1.1 Applicable Matrix

This test method covers a procedure to: (a) identify asbestos in soil, (b) provide an estimate of the concentration of asbestos in the sampled soil reported as a percentage, and (c) may size asbestos fibers reported as the number of asbestos structures per gram of sample.

1.2 Scope and application

This analysis method is used for the general testing of soil samples for asbestos. This test method does not describe procedures or techniques required to evaluate the safety or habitability of buildings with asbestos-containing materials or compliance with federal, state, or local regulations or statutes. It is the user's responsibility to make these determinations.

1.3 Summary of Method

The sample is defined as any material that passes through a 19 mm sieve. Material larger than 19 mm will not be included in the analysis . A representative portion of sample soil is collected and dry sieved into coarse (>2-mm), medium (>100-µm to <2-mm), and fine (<100-µm) fractions. Weights for each fraction are taken and recorded. The fine fraction (<100 µm) is analyzed by both polarized light microscopy (PLM) point counting and transmission electron microscopy (TEM). The medium (>100-µm to <2-mm) and coarse (>2-mm) fractions are analyzed by PLM visual estimation. Results are expressed as weight percent. Additional analysis of the fibers from the TEM fine fraction may be performed to derive size data of the fibers observed.

1.4 Detection Limit

This test method is generally applicable for an estimate of the concentration of asbestos down to 0.25% by stereomicroscopy and PLM. TEM results are reported in structures per microgram of soil.

2.0 Interferences

The following minerals have properties (that is, chemical or crystalline structure) that are very similar to asbestos minerals and may interfere with the analysis by causing a false positive to be recorded during the test. Therefore, literature references for these materials shall be maintained in the laboratory for comparison to asbestos minerals so that they are not misidentified as asbestos minerals.

Antigorite,
Palygorskite (Attapulgite),
Halloysite,
Pyroxenes,
Sepiolite,
Vermiculite scrolls,
Fibrous talc, and
Hornblende and other amphiboles other than those listed in 3.1.2.

Collecting any dust particles greater than 1 mm in size in this test method may cause interference and, therefore, must be avoided.

3.0 Definitions

3.1Definitions:
3.1.1 *asbestiform, n*—special type of fibrous habit in which the fibers are separable into thinner fibers and ultimately into fibrils.
3.1.1.1 *Discussion*--This habit accounts for greater flexibility and higher tensile strength than other habits of the same mineral. For more information on asbestiform mineralogy, see Refs **(1)**, **(2),** and **(3)**.[1]
3.1.2 *asbestos, n*—collective term that describes a group of naturally occurring, inorganic, highly fibrous, silicate-dominated minerals that are easily separated into long, thin, flexible fibers when crushed or processed.
3.1.2.1 *Discussion*--Included in the definition are the asbestiform varieties of: serpentine (chrysotile), riebeckite (crocidolite), grunerite (grunerite asbestos), anthophyllite (anthophyllite asbestos), tremolite (tremolite asbestos), and actinolite (actinolite asbestos). The amphibole mineral compositions are defined according to nomenclature of the International Mineralogical Association **(3)**.

Asbestos	**Chemical Abstract Service No.**
Chrysotile	12001-29-5
Crocidolite	12001-28-4
Grunerite asbestos	12172-73-5
Anthophyllite asbestos	77536-67-5
Tremolite asbestos	77536-68-6
Actinolite asbestos	77536-66-4

3.1.3 *fibril, n*—single fiber that cannot be separated into smaller components without losing its fibrous properties or appearance.
3.2 *Definitions of Terms Specific to This Standard Operating Procedure:*
3.2.1 *aspect ratio, n*—ratio of the length of a fibrous particle to its average width.
3.2.2 *bundle, n*—structure composed of three or more fibers in a parallel arrangement with the fibers closer than one fiber diameter to each other.
3.2.3 *cluster, n*—structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group; groupings of fibers shall have more than two points touching.
3.2.4 *debris, n*—materials that are of an amount and size (particles greater than 1 mm in diameter) that can be visually identified as to their source.
3.2.5 *dust, n*—any material composed of particles in a size range of <1 mm and large enough to settle by virtue of their weight from the ambient air (see definition for settleable particulate matter in Test Method D 1739).
3.2.6 *fiber, n*—structure having a minimum length of 0.5 :m, an aspect ratio of 5:1 or greater, and substantially parallel sides **(4)**.
3.2.7 *fibrous, adj*—of a mineral composed of parallel, radiating, or interlaced aggregates of fibers, from which the fibers are sometimes separable, that is, the crystalline aggregate may be referred to as fibrous even if it is not composed of separable fibers, but has that distinct appearance.
3.2.7.1 *Discussion*--The term fibrous is used in a general mineralogical way to describe aggregates of grains that crystallize in a needle-like habit and appear to be composed of fibers. Fibrous has a much more general meaning than asbestos. While it is correct that all asbestos minerals are fibrous, not all minerals having fibrous habits are asbestos.
3.2.8 *indirect preparation, n*—method in which a sample passes through one or more intermediate steps before final filtration.

[1] The boldface numbers in parentheses refer to the list of references at the end of this test method.

3.2.9 *matrix, n*—structure in which one or more fibers, or fiber bundles that are touching, are attached to or partially concealed by a single particle or connected group of nonfibrous particles. The exposed fiber shall meet the fiber definition (*see* 3.2.6).
3.2.10 *soil, n*—loose top layer of the earth's surface consists of rock and mineral particles mixed with decayed organic matter (humus) and is capable of retaining water, providing nutrients for plants, and supporting a wide range of biotic communities.
3.2.10.1 *Discussion*--Soil is formed by a combination of depositional, chemical, and biological processes and plays an important role in the carbon, nitrogen, and hydrologic cycles. Soil types vary widely from one region to another depending on the type of bedrock they overlie and the climate in which they form. In wet and humid regions, for example, soils tend to be thicker than they are in dry regions. See more at Ref **(5)**.
3.2.11 *structures, n*—term that is used to categorize all the types of asbestos particles that are recorded during the analysis (such as fibers, bundles, clusters, and matrices).
3.2.11.1 *Discussion*--Final results of the test are always expressed in asbestos structures per square centimeter.

4.0 Safety

All personnel performing preparation and/or analysis of samples must be familiar with the EMSL Chemical Hygiene Plan.

5.0 Equipment and Supplies

5.1 *Balances*—Balances or scales used in testing fine and coarse aggregate shall have readability and accuracy as follows:
5.1.1 For fine aggregate, readable to 0.1 g and accurate to 0.1 g or 0.1 % of the test load, whichever is greater, at any point within the range of use.
5.1.2 For coarse aggregate, or mixtures of fine and coarse aggregate, readable and accurate to 0.5 g or 0.1 % of the test load, whichever is greater, at any point within the range of use.
5.2 *Sieves*—The sieve cloth shall be mounted on substantial frames constructed in a manner that will prevent loss of material during sieving. The sieve cloth and standard sieve frames shall conform to the requirements of Specification E 11.
5.4 *Oven*—An oven of appropriate size capable of maintaining a uniform temperature of 110 ± 5°C (230 ± 9°F).
5.5 *Transmission Electron Microscope (TEM)*, an 80- to 120-kV TEM, capable of performing electron diffraction, with a fluorescent screen inscribed with calibrated gradations, is required. The TEM shall be equipped with energy dispersive X-ray spectroscopy (EDXA) and it shall have a scanning transmission electron microscopy (STEM) attachment or be capable of producing a spot size of less than 250 nm in diameter in crossover.
5.6 *EDXA*
5.7 *High-Vacuum Carbon Evaporator*, with rotating stage
5.8 *High-Efficiency Particulate Air (HEPA)*, filtered negative flow hood.
5.9 *Exhaust or Fume Hood*
5.10 *Stereo Microscope*
5.11 *Polarized Light Microscope*

6.0 Reagents and Standards

6.1 *Purity of Reagents*—Reagent-grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.[2]

[2] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II of Specification D 1193.
6.3 *Ziploc Bags*
6.4 Plastic *Sample Containers*, with wide mouth screw cap (500 mL) or equivalent sealable container.
6.5 *Waterproof Markers*
6.6 *Forceps (Tweezers)*
6.7 *Ultrasonic Bath*, tabletop model (100 W).
6.8 *Graduated Pipettes (1-, 5-, or 10-mL Sizes)*, glass or plastic.
6.9 *Filter Funnel*, either 25 or 47 mm, glass or disposable. Filter funnel assemblies, either glass or disposable plastic, and using either a 25- or 47-mm diameter filter.
6.10 *Side Arm Filter Flask*, 1000 mL.
6.11 *Mixed Cellulose Ester (MCE) Membrane Filters*, 25- or 47-mm diameter, 0.22- and 5-µm pore size.
6.12 *Polycarbonate (PC) Filters*, 25- or 47-mm diameter, 0.2-µm pore size.
6.13 *Storage Containers*, for the 25- or 47-mm filters (for archiving).
6.14 *Glass Slides*, approximately 76 by 25 mm in size.
6.15 *Scalpel Blades*, No. 10 or equivalent.
6.16 *Cabinet-Type Desiccator*, or low-temperature drying oven.
6.17 *Acetone*, reagent grade.
6.18 *Dimethylformamide (DMF)*
6.19 *Glacial Acetic Acid*
6.20 *Plasma Asher*, low temperature.
6.21 *pH Paper*
6.22 *Air Sampling Pump*, low-volume personal type, capable of achieving a flow rate of 1 to 5 L/min.
6.23 *Rotameter*
6.24 *Air-Sampling Cassettes*, 25 or 37 mm, containing 0.8-µm or smaller pore size MCE or PC filters.
6.25 *Sieves > 2 mm*, 100 µm and collection pan
6.26 *Nonasbestos Mineral*, references as outlined in 6.1.
6.27 *Asbestos Standards*, as outlined in 3.1.2.
6.28 *NIST Calibrated Analytical Balance*
6.29 *Small Vacuum Pump*, that can maintain a pressure of 92 kPa.
6.30 *Petri Dishes*, large glass, approximately 90 mm in diameter.
6.31 *Jaffe Washer*, stainless steel or aluminum mesh screen, 30 to 40 mesh, and approximately 75 by 50 mm in size.
6.32 *Copper TEM Finder Grids*, 200 mesh.
6.33 *Carbon Evaporator Rods*
6.34 *Lens Tissue*
6.35 *Ashless Filter Paper Filters*, 90-mm diameter.
6.36 *Gummed Paper Reinforcement Rings*
6.37 *Wash Bottles*, plastic.
6.38 *Reagent Alcohol*, HPLC grade (Fisher A995 or equivalent).
6.39 *Polarized Light Microscope*
6.40 *Diffraction Grating Replica*
6.41 *Stereo Microscope*
6.42 *Transmission Electron Microscope*
6.43 *Energy Dispersive Spectrometer*
6.44 *Mechanical Sieve Shaker*
6.45 *Diffraction Grating Replica*

7.0 Sample Collection, Preservation, Shipment and Storage

7.1 A representative portion of the soil sample material is collected in a 250-mL plastic container. These containers are available from the laboratory. Soil samples shall be collected in a manner that meets the requirements of this method and the needs of the investigator for whom the soil is being analyzed.

TABLE 1 Sample Area and Depth for Surface Samples

Collected Volume = 250 cm^3					
Length, cm	**Depth, cm**	**Length, in.**	**Depth, in.**	**Collected Media**	
10	2.5	4	1	Loose soil	
15	1.1	6	0.5		
20	0.6	8	0.25		Hardpan
30	0.3	12	0.12		
Collected Volume = 500 cm^3					
Length, cm	**Depth, cm**	**Length, in.**	**Depth, in.**	**Collected Media**	
10	5.0	4	2	Loose soil	
15	2.2	6	1		
20	1.3	8	0.5		
30	0.6	12	0.25		Hardpan
45	0.2	18	0.10		

8.0 Preparation of Soil and Apparatus

8.1 Each soil sample will be dried in an oven at 110 ± 5°C until the weight is stable.
8.2 Under a hood nest the sieves in order of decreasing size of opening from top to bottom on the sieve shaker with the 19mm on top, 2-mm sieve (coarse), 100-µm sieve in the middle (medium), and the collection pan on the bottom (fine).

9.0 Procedure

9.1 Dry the sample to constant mass at a temperature of 110 ± 5°C (230 ± 9°F).
9.1.1Transfer the sample to the sieve rack and place on the top of the 2-mm sieve (coarse). Mist the soil lightly with isopropyl alcohol. Secure the sieve shaker top on the sieve.
9.1.2 Turn on the sieve shaker for 5 min. When finished, remove the sieved soil from each sieved fraction and place into a plastic container or Ziploc bag.
9.1.3 Determine the weight for each fraction.
9.2 *Analysis Summary*
9.2.1 The fine (<100-µm) fraction is analyzed by both PLM point counting and TEM.
9.2.2 The medium (<2-mm and >100-µm) fraction is analyzed by PLM visual estimation.
9.2.3 The coarse (>2-mm) fraction is analyzed by PLM using visual estimation.
9.3 *Analysis –Fine Fraction (<100 µm)*
9.3.1 *Point Count*--Eight separate slide mounts are prepared for the PLM point count at 100× following EPA 600/M4-82-020. All asbestos fibers that are ≥3 µm wide and have an aspect ratio of ≥3:1 are counted.
9.3.2 *TEM Using Semi-quantitative Procedure*
9.3.2.1 Approximately 100 mg of the material is suspended in 5 mL of acetone. The mixture is ultrasonicated for 3 min. It is then centrifuged for 3 min, and the acetone supernatant is decanted to a level of 0.5 mL. The remaining material is re-suspended, and a drop of the suspension is then placed on each of two carbon-coated copper grids (3-mm diameter, 200 mesh).

9.3.2.2 The sample grids are dried and then examined using TEM. At least five openings are examined on each grid at high magnification (15 000×). Record asbestos structures containing fibers that meet a ≥3:1 length:width aspect ratio. Fibers are identified using morphology, selected-area electron diffraction (SAED), and energy-dispersive X-ray spectroscopy. Fibers identified as asbestos are classified as chrysotile, crocidolite, amosite, tremolite-actinolite or anthophyllite. A visual estimate of the type and amount of asbestos fibers is determined. If a fiber count is performed the length and width of fibers are recorded.

9.4 *Analysis- Medium Fraction (<2 mm and >100 μm)*

9.4.1 *Visual Estimation*--Three separate slide mounts are prepared for the PLM visual estimation method at 100× following EPA 600/M4-82-020.

Analysis--Coarse Fraction 2-mm Sieve--The coarse fraction of the sample is analyzed using standard visual estimation techniques. The material is first examined under a stereomicroscope to estimate asbestos content. The asbestos is confirmed by mounting portions of this fraction and analyzing by PLM in accordance with EPA 600/M4-82-020.

10.0 Calculations

10.1 The "total asbestos content" in the vermiculite or soil sample is determined using the following formula:

$$\text{Total Asbestos (\%)} = \frac{[\%_F \text{ PLM PC} * W_F] + [\%_F \text{ TEM} * W_F] + [\%_M \text{ PLM} * W_M] + [\%_C \text{ PLM} * W_C]}{W_F + W_M + W_C} \quad \textbf{(1)}$$

Where:
$\%_F$ = Percentage of asbestos determined by PLM and TEM,
$\%_M$ = Percentage of asbestos determined by PLM,
$\%_C$ = Percentage of asbestos determined by stereomicroscope and PLM,
W_F = Weight of fine fraction of sample,
W_M = Weight of medium fraction of sample, and
W_C = Weight of coarse fraction of sample.

11.0 Reporting

11.1 Report the following information for each soil sample analyzed:
11.1.1 Concentration in percent (%),
11.1.2 Analytical sensitivity,
11.1.3 Types of asbestos present,
11.1.4 Number of asbestos structures counted (if counted),
11.1.5 Effective filtration area or grid area,
11.1.6 Average size of the TEM grid openings that were counted,
11.1.7 Number of grid openings examined,
11.1.8 Sample size used (grams), and
11.1.9 Area of the surface sampled.

12.0 Pollution Prevention / Waste Management

12.1 Pollution Prevention

EMSL Analytical makes all efforts to reduce the volume and toxicity of the waste generated by the laboratory. An effort to manage procurement of hazardous materials has been implemented in order to avoid over ordering. Hazardous waste is classified for proper disposal.

12.2 Waste Management

The waste generated during prep and analysis will be disposed of following safety procedures outlined in the chemical hygiene plan.

13.0 References

(1) Steel, E. and Wylie, A., "Mineralogical Characteristics of Asbestos," in *Geology of Asbestos Deposits*, P.H. Riordon, Ed., SME-AIME, 1981, pp. 93–101.

(2) Zussman, J., "The Mineralogy of Asbestos," in *Asbestos: Properties, Applications and Hazards*, John Wiley and Sons, 1979, pp. 45–67.

(3) Leake, B. E., "Nomenclature of Amphiboles," *American Mineralogist*, Vol 63, 1978, pp. 1023–1052.

(4) "*USEPA Asbestos-Containing Materials in Schools: Final Rule and Notice," Federal Register*, 40 CFR Part 763, Appendix A to Subpart E., Oct. 30, 1987.

(5) American Heritage® Science Dictionary, Houghton Mifflin Co., 2005.

(6) *NIST/NVLAP Program Handbook for Airborne Asbestos Analysis*, NISTIR, August 1989, pp. 89–4137.

(7) *Manual on Asbestos Control: Surveys, Removal and Management*, Second Edition, Andrew Oberta, Ed., ASTM International, West Conshohocken, PA, March 2005.

(8) ''Comparison of the Alternative Asbestos Control Method and the NESHAP Method for Demolition of Asbestos-Containing Buildings," Draft Final Report, Environmental Protection Agency, May 11, 2007.

14.0 Revision History

Revision #	Date	Revision	Initials
1	6/5/09	Brought SOP format into ISO/NELAC compliance	EC

Authorizing Signatures

Author (print)	Author Signature	Date
Reviewer (print)	Reviewer Signature	Date
Corporate Approval (print)	Corporate Approval Signature	Date

SOP Review and Acknowledgement Form

Instructions: Ensure all personnel in laboratory affected by this procedure read and acknowledge. Once completed, the page shall be retained in an Acknowledgments binder along with personnel training.

Laboratory Manager Acknowledgement
My signature below signifies that I have read and understand the entire contents of this document. My signature represents that I agree to fully comply with, implement, and enforce all requirements, procedures, and protocols specified in these procedures set forth in this document and any supporting reference materials or methodologies.

Laboratory Manager Signature _____ Date _____

Laboratory Staff Acknowledgement
My signature below signifies that I have read and understand the entire contents of this document. My signature represents that I agree to fully comply with, implement, and enforce all requirements, procedures, and protocols specified in these procedures set forth in this document and any supporting reference materials or methodologies.

Signature	Date

Target List	EPA Method	Soil Accuracy % Recovery	Aqueous Accuracy % Recovery	Soil Precision RPD (1)	Aqueous Precision RPD (1)
Polychorinated Biphenyls					
Aroclor 1016	8082	53-123	65-112	0-50 (nominal)	0-30 (nominal)
Aroclor 1221	8082	---	---	---	---
Aroclor 1232	8082	---	---	---	---
Aroclor 1242	8082	---	---	---	---
Aroclor 1248	8082	---	---	---	---
Aroclor 1254	8082	---	---	---	---
Aroclor 1260	8082	58-120	62-104	0-50 (nominal)	0-30 (nominal)
Surrogates:					
Tetrachloro-m-xylene	8082	56-115	62-111	---	---
Decachlorobiphenyl	8082	59-124	44-135	---	---
Semivolatile Organic Compounds (full scan)					
1,4-Dioxane	8270C	15-59	10-73	0-50 (nominal)	0-30 (nominal)
n-Nitrosodimethylamine	8270C	43-115	11-95	0-50 (nominal)	0-30 (nominal)
Pyridine	8270C	22-64	10-96	0-50 (nominal)	0-30 (nominal)
2-Picoline	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
n-Nitrosomethylethylamine	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
Methyl Methanesulfonate	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
n-Nitrosodiethylamine	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
Ethyl Methanesulfonate	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
Benzaldehyde	8270C	10-123	10-189	0-50 (nominal)	0-30 (nominal)
Aniline	8270C	25-104	10-59	0-50 (nominal)	0-30 (nominal)
Phenol	8270C	56-108	10-78	0-50 (nominal)	0-30 (nominal)
Bis (2-Chloroethyl) Ether	8270C	50-104	45-95	0-50 (nominal)	0-30 (nominal)
2-Chlorophenol	8270C	57-102	44-91	0-50 (nominal)	0-30 (nominal)
1,3-Dichlorobenzene	8270C	58-91	37-90	0-50 (nominal)	0-30 (nominal)
1,4-Dichlorobenzene	8270C	61-94	38-91	0-50 (nominal)	0-30 (nominal)
1,2-Dichlorobenzene	8270C	65-93	39-94	0-50 (nominal)	0-30 (nominal)
Benzyl Alcohol	8270C	63-109	16-118	0-50 (nominal)	0-30 (nominal)
2,2'-Oxybis(1-chloropropane	8270C	61-98	42-100	0-50 (nominal)	0-30 (nominal)
2-Methylphenol	8270C	60-99	37-87	0-50 (nominal)	0-30 (nominal)
Acetophenone	8270C	59-102	49-102	0-50 (nominal)	0-30 (nominal)
n-Nitrosopyrrolidine	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
o-Toluidine	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
n-Nitroso-di-n-propylamine	8270C	52-93	41-97	0-50 (nominal)	0-30 (nominal)
n-Nitrosomorpholine	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
3&4-Methylphenol	8270C	58-105	28-85	0-50 (nominal)	0-30 (nominal)

Target List	EPA Method	Soil Accuracy % Recovery	Aqueous Accuracy % Recovery	Soil Precision RPD (1)	Aqueous Precision RPD (1)
Hexachloroethane	8270C	59-90	31-90	0-50 (nominal)	0-30 (nominal)
Nitrobenzene	8270C	59-103	48-95	0-50 (nominal)	0-30 (nominal)
n-Nitrosopiperidine	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
Isophorone	8270C	23-110	53-93	0-50 (nominal)	0-30 (nominal)
2-Nitrophenol	8270C	61-100	48-101	0-50 (nominal)	0-30 (nominal)
2,4-Dimethylphenol	8270C	52-99	51-87	0-50 (nominal)	0-30 (nominal)
O,O,O-Triethylphosphorothioate	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
Bis (2-chloroethoxy)methane	8270C	63-99	40-98	0-50 (nominal)	0-30 (nominal)
2,4-Dichlorophenol	8270C	63-102	47-106	0-50 (nominal)	0-30 (nominal)
Benzoic acid	8270C	10-121	10-151	0-50 (nominal)	0-30 (nominal)
1,2,4-Trichlorobenzene	8270C	63-93	38-90	0-50 (nominal)	0-30 (nominal)
Naphthalene	8270C	55-99	48-89	0-50 (nominal)	0-30 (nominal)
2,6-Dichlorophenol	8270C	65-117	45-109	0-50 (nominal)	0-30 (nominal)
Hexachloropropene	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
4-Chloroaniline	8270C	10-89	34-100	0-50 (nominal)	0-30 (nominal)
Hexachlorobutadiene	8270C	59-90	34-86	0-50 (nominal)	0-30 (nominal)
n-Nitroso-di-n-butylamine	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
p-Phenylenediamine	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
Caprolactam	8270C	30-140	10-86	0-50 (nominal)	0-30 (nominal)
4-Chloro-3-methylphenol	8270C	62-109	63-101	0-50 (nominal)	0-30 (nominal)
a,a-Dimethylphenethylamine	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
Safrole	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
2-Methylnaphthalene	8270C	42-127	50-103	0-50 (nominal)	0-30 (nominal)
1-Methylnaphthalene		57-93	27-120	0-50 (nominal)	0-30 (nominal)
Hexachlorocyclopentadiene	8270C	23-107	23-70	0-50 (nominal)	0-30 (nominal)
1,2,4,5-Tetrachlorobenzene	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
2,4,6-Trichlorophenol	8270C	61-105	57-109	0-50 (nominal)	0-30 (nominal)
2,4,5-trichlorophenol	8270C	64-107	53-136	0-50 (nominal)	0-30 (nominal)
Diethyl adipate	8270C	34-133	10-113	0-50 (nominal)	0-30 (nominal)
Isosafrole	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
2-Chloronaphthalene	8270C	68-102	37-76	0-50 (nominal)	0-30 (nominal)
1,1'-Biphenyl	8270C	50-113	51-105	0-50 (nominal)	0-30 (nominal)
1-Chloronaphthalene	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
2-Nitroaniline	8270C	58-120	56-108	0-50 (nominal)	0-30 (nominal)
1,4-Naphthoquinone	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
Dimethyl phthalate	8270C	66-121	10-111	0-50 (nominal)	0-30 (nominal)
1,3-Dinitrobenzene	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)

Target List	EPA Method	Soil Accuracy % Recovery	Aqueous Accuracy % Recovery	Soil Precision RPD (1)	Aqueous Precision RPD (1)
2,6-Dinitrotoluene	8270C	64-109	65-110	0-50 (nominal)	0-30 (nominal)
Acenaphthylene	8270C	59-108	59-97	0-50 (nominal)	0-30 (nominal)
3-Nitroaniline	8270C	10-173	46-97	0-50 (nominal)	0-30 (nominal)
Acenaphthene	8270C	64-106	58-99	0-50 (nominal)	0-30 (nominal)
2,4-Dinitrophenol	8270C	20-81	12-143	0-50 (nominal)	0-30 (nominal)
Pentachlorobenzene	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
Dibenzofuran	8270C	60-108	62-104	0-50 (nominal)	0-30 (nominal)
4-Nitrophenol	8270C	45-139	10-114	0-50 (nominal)	0-30 (nominal)
2,4-Dinitrotoluene	8270C	64-111	66-123	0-50 (nominal)	0-30 (nominal)
1-Naphthylamine	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
2,3,4,6-Tetrachlorophenol	8270C	58-93	49-119	0-50 (nominal)	0-30 (nominal)
2-Naphthylamine	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
Diethylphthalate	8270C	59-113	58-101	0-50 (nominal)	0-30 (nominal)
Fluorene	8270C	57-108	63-107	0-50 (nominal)	0-30 (nominal)
4-Chlorophenyl-phenylether	8270C	67-110	65-100	0-50 (nominal)	0-30 (nominal)
O,O-Diethyl-o-2-pyrazinylphosphoroth	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
5-Nitro-O-toluidine	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
4-Nitroaniline	8270C	40-142	52-106	0-50 (nominal)	0-30 (nominal)
4,6-Dinitro-2-methylphenol	8270C	40-111	52-129	0-50 (nominal)	0-30 (nominal)
n-Nitrosodiphenylamine	8270C	12-145	52-96	0-50 (nominal)	0-30 (nominal)
1,2-Diphenylhydrazine	8270C	53-119	57-98	0-50 (nominal)	0-30 (nominal)
Azobenzene	8270C	58-121	57-98	0-50 (nominal)	0-30 (nominal)
Sulfotepp	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
Diallate	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
Phorate	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
1,3,5-Trinitrobenzene	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
4-Bromophenyl-phenylether	8270C	67-119	56-106	0-50 (nominal)	0-30 (nominal)
Phenacetin	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
Hexachlorobenzene	8270C	65-112	51-112	0-50 (nominal)	0-30 (nominal)
Dimethoate	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
Atrazine	8270C	55-135	83-153	0-50 (nominal)	0-30 (nominal)
Pentachlorophenol	8270C	57-125	41-134	0-50 (nominal)	0-30 (nominal)
Pentachloronitrobenzene	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
4-Aminobiphenyl	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
Proamide	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
Phenanthrene	8270C	61-117	70-107	0-50 (nominal)	0-30 (nominal)
Dinoseb	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)

Target List	EPA Method	Soil Accuracy % Recovery	Aqueous Accuracy % Recovery	Soil Precision RPD (1)	Aqueous Precision RPD (1)
Disulfoton	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
Anthracene	8270C	56-114	67-112	0-50 (nominal)	0-30 (nominal)
Carbazole	8270C	49-168	57-125	0-50 (nominal)	0-30 (nominal)
Methyl parathion	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
Di-n-butylphthalate	8270C	56-129	68-114	0-50 (nominal)	0-30 (nominal)
4-Nitroquinoline-1-oxide	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
Parathion	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
Methapyrilene	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
Isodrin	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
Fluoranthene	8270C	70-115	67-119	0-50 (nominal)	0-30 (nominal)
Benzidine	8270C	10-150	10-100 (nominal)	0-50 (nominal)	0-30 (nominal)
Pyrene	8270C	54-131	58-135	0-50 (nominal)	0-30 (nominal)
Aramite	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
p-Dimethylaminoazobenzene	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
Chlorobenzilate	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
Famphur	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
Kepone	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
3,3'-Dimethylbenzidine	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
Butylbenzylphthalate	8270C	50-131	56-129	0-50 (nominal)	0-30 (nominal)
Bis(2-ethylhexyl)adipate	8270C	39-130	50-142	0-50 (nominal)	0-30 (nominal)
2-Acetylaminofluorene	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
Benzo(a)anthracene	8270C	65-111	66-109	0-50 (nominal)	0-30 (nominal)
3,3'-Dichlorobenzidine	8270C	10-130	36-87	0-50 (nominal)	0-30 (nominal)
Chrysene	8270C	66-107	68-114	0-50 (nominal)	0-30 (nominal)
Bis(2-ethylhexyl)phthalate	8270C	42-142	51-155	0-50 (nominal)	0-30 (nominal)
Di-n-octylphthalate	8270C	10-187	33-184	0-50 (nominal)	0-30 (nominal)
Benzo(b)fluoranthene	8270C	47-121	60-108	0-50 (nominal)	0-30 (nominal)
7,12-Dimethylbenz(a)anthracene	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
Benzo(k)fluoranthene	8270C	37-130	59-125	0-50 (nominal)	0-30 (nominal)
Benzo(a)pyrene	8270C	62-114	63-118	0-50 (nominal)	0-30 (nominal)
Hexachlorophene	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
3-Methylcholanthrene	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
Dibenz(a,j)acridine	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)
Indeno(1,2,3-cd)pyrene	8270C	55-128	55-111	0-50 (nominal)	0-30 (nominal)
Dibenzo(a,h)anthracene	8270C	54-133	61-111	0-50 (nominal)	0-30 (nominal)
Benzo(g,h,i)perylene	8270C	53-126	58-115	0-50 (nominal)	0-30 (nominal)
Surrogates:					

Target List	EPA Method	Soil Accuracy % Recovery	Aqueous Accuracy % Recovery	Soil Precision RPD (1)	Aqueous Precision RPD (1)
2-Fluorophenol	8270C	43-99	10-80	---	---
Phenol-d6	8270C	53-98	10-90	---	---
Nitrobenzene-d5	8270C	47-100	41-91	---	---
2-Fluorobiphenyl	8270C	49-114	43-90	---	---
2,4,6-Tribromophenol	8270C	44-111	37-112	---	---
Terphenyl-d14	8270C	58-140	36-156	---	---
Semivolatile Organic SIM Compounds					
1,4-Dioxane	8270C SIM	30-150 (nominal)	10-93	0-50 (nominal)	0-30 (nominal)
Benzaldehyde	8270C SIM	30-150 (nominal)	10-122	0-50 (nominal)	0-30 (nominal)
Phenol	8270C SIM	30-150 (nominal)	10-77	0-50 (nominal)	0-30 (nominal)
Bis(2-chloroethyl)ether	8270C SIM	30-150 (nominal)	56-87	0-50 (nominal)	0-30 (nominal)
2-Chlorophenol	8270C SIM	30-150 (nominal)	30-112	0-50 (nominal)	0-30 (nominal)
2,2'-Oxybis(1-chloropropane	8270C SIM	30-150 (nominal)	57-90	0-50 (nominal)	0-30 (nominal)
Acetophenone	8270C SIM	30-150 (nominal)	55-95	0-50 (nominal)	0-30 (nominal)
2-Methylphenol	8270C SIM	30-150 (nominal)	29-89	0-50 (nominal)	0-30 (nominal)
n-Nitroso-di-n-propylamine	8270C SIM	30-150 (nominal)	43-86	0-50 (nominal)	0-30 (nominal)
Hexachloroethane	8270C SIM	30-150 (nominal)	48-80	0-50 (nominal)	0-30 (nominal)
3&4-Methylphenol	8270C SIM	30-150 (nominal)	31-78	0-50 (nominal)	0-30 (nominal)
Nitrobenzene	8270C SIM	30-150 (nominal)	43-89	0-50 (nominal)	0-30 (nominal)
Isophorone	8270C SIM	30-150 (nominal)	43-89	0-50 (nominal)	0-30 (nominal)
2-Nitrophenol	8270C SIM	30-150 (nominal)	30-113	0-50 (nominal)	0-30 (nominal)
2,4-Dimethylphenol	8270C SIM	30-150 (nominal)	33-87	0-50 (nominal)	0-30 (nominal)
Bis(2-chloroethoxy)methane	8270C SIM	30-150 (nominal)	53-93	0-50 (nominal)	0-30 (nominal)
2,4-Dichlorophenol	8270C SIM	30-150 (nominal)	31-106	0-50 (nominal)	0-30 (nominal)
Naphthalene	8270C SIM	54-94	46-84	0-50 (nominal)	0-30 (nominal)
4-Chloroaniline	8270C SIM	30-150 (nominal)	38-122	0-50 (nominal)	0-30 (nominal)
Hexachlorobutadiene	8270C SIM	30-150 (nominal)	48-95	0-50 (nominal)	0-30 (nominal)
Caprolactam	8270C SIM	30-150 (nominal)	10-164	0-50 (nominal)	0-30 (nominal)
4-Chloro-3-methylphenol	8270C SIM	30-150 (nominal)	58-107	0-50 (nominal)	0-30 (nominal)
2-Methylnaphthalene	8270C SIM	75-132	51-114	0-50 (nominal)	0-30 (nominal)
1-Methylnaphthalene	8270C SIM	36-114	51-82	0-50 (nominal)	0-30 (nominal)
Hexachlorocyclopentadiene	8270C SIM	30-150 (nominal)	10-96	0-50 (nominal)	0-30 (nominal)
2,4,6-Trichlorophenol	8270C SIM	30-150 (nominal)	19-139	0-50 (nominal)	0-30 (nominal)
2,4,5-Trichlorophenol	8270C SIM	30-150 (nominal)	20-133	0-50 (nominal)	0-30 (nominal)
2-Chloronaphthalene	8270C SIM	30-150 (nominal)	51-93	0-50 (nominal)	0-30 (nominal)
1,1'-Biphenyl	8270C SIM	30-150 (nominal)	56-96	0-50 (nominal)	0-30 (nominal)
2-Nitroaniline	8270C SIM	30-150 (nominal)	54-105	0-50 (nominal)	0-30 (nominal)
Dimethyl phthalate	8270C SIM	30-150 (nominal)	10-107	0-50 (nominal)	0-30 (nominal)
Acenaphthylene	8270C SIM	51-103	55-105	0-50 (nominal)	0-30 (nominal)

Target List	EPA Method	Soil Accuracy % Recovery	Aqueous Accuracy % Recovery	Soil Precision RPD (1)	Aqueous Precision RPD (1)
2,6-Dinitrotoluene	8270C SIM	30-150 (nominal)	47-86	0-50 (nominal)	0-30 (nominal)
3-Nitroaniline	8270C SIM	30-150 (nominal)	50-103	0-50 (nominal)	0-30 (nominal)
Acenaphthene	8270C SIM	56-97	53-90	0-50 (nominal)	0-30 (nominal)
2,4-Dinitrophenol	8270C SIM	30-150 (nominal)	10-95	0-50 (nominal)	0-30 (nominal)
Dibenzofuran	8270C SIM	30-150 (nominal)	56-98	0-50 (nominal)	0-30 (nominal)
4-Nitrophenol	8270C SIM	30-150 (nominal)	10-160	0-50 (nominal)	0-30 (nominal)
2,4-Dinitrotoluene	8270C SIM	30-150 (nominal)	48-95	0-50 (nominal)	0-30 (nominal)
2,3,4,6-Tetrachlorophenol	8270C SIM	30-150 (nominal)	10-101	0-50 (nominal)	0-30 (nominal)
Diethylphthalate	8270C SIM	30-150 (nominal)	38-104	0-50 (nominal)	0-30 (nominal)
Fluorene	8270C SIM	61-103	53-95	0-50 (nominal)	0-30 (nominal)
4-Chlorophenyl-phenylether	8270C SIM	30-150 (nominal)	57-94	0-50 (nominal)	0-30 (nominal)
4-Nitroaniline	8270C SIM	30-150 (nominal)	42-152	0-50 (nominal)	0-30 (nominal)
4,6-Dinitro-2-methylphenol	8270C SIM	30-150 (nominal)	10-77	0-50 (nominal)	0-30 (nominal)
n-Nitrosodiphenylamine	8270C SIM	30-150 (nominal)	48-82	0-50 (nominal)	0-30 (nominal)
4-Bromophenyl-phenylether	8270C SIM	30-150 (nominal)	61-92	0-50 (nominal)	0-30 (nominal)
Hexachlorobenzene	8270C SIM	30-150 (nominal)	62-90	0-50 (nominal)	0-30 (nominal)
Atrazine	8270C SIM	30-150 (nominal)	66-126	0-50 (nominal)	0-30 (nominal)
Pentachlorophenol	8270C SIM	30-150 (nominal)	10-66	0-50 (nominal)	0-30 (nominal)
Phenanthrene	8270C SIM	58-122	73-100	0-50 (nominal)	0-30 (nominal)
Anthracene	8270C SIM	55-113	70-95	0-50 (nominal)	0-30 (nominal)
Carbazole	8270C SIM	30-150 (nominal)	78-126	0-50 (nominal)	0-30 (nominal)
Di-n-butylphthalate	8270C SIM	30-150 (nominal)	46-163	0-50 (nominal)	0-30 (nominal)
Fluoranthene	8270C SIM	68-126	81-109	0-50 (nominal)	0-30 (nominal)
Pyrene	8270C SIM	33-133	71-104	0-50 (nominal)	0-30 (nominal)
Butylbenzylphthalate	8270C SIM	30-150 (nominal)	30-119	0-50 (nominal)	0-30 (nominal)
Benzo(a)anthracene	8270C SIM	50-138	70-110	0-50 (nominal)	0-30 (nominal)
3,3'-Dichlorobenzidine	8270C SIM	30-150 (nominal)	10-118	0-50 (nominal)	0-30 (nominal)
Chrysene	8270C SIM	44-132	70-95	0-50 (nominal)	0-30 (nominal)
Bis(2-ethylhexyl)phthalate	8270C SIM	30-150 (nominal)	97-188	0-50 (nominal)	0-30 (nominal)
Di-n-octylphthalate	8270C SIM	30-150 (nominal)	56-122	0-50 (nominal)	0-30 (nominal)
Benzo(b)fluoranthene	8270C SIM	44-119	67-102	0-50 (nominal)	0-30 (nominal)
Benzo(k)fluoranthene	8270C SIM	40-120	68-103	0-50 (nominal)	0-30 (nominal)
Benzo(a)pyrene	8270C SIM	52-113	63-98	0-50 (nominal)	0-30 (nominal)
Indeno(1,2,3-cd)pyrene	8270C SIM	32-134	61-112	0-50 (nominal)	0-30 (nominal)
Dibenzo(a,h)anthracene	8270C SIM	30-134	66-108	0-50 (nominal)	0-30 (nominal)
Benzo(g,h,i)perylene	8270C SIM	43-121	62-106	0-50 (nominal)	0-30 (nominal)
Surrogates:					

Target List	EPA Method	Soil Accuracy % Recovery	Aqueous Accuracy % Recovery	Soil Precision RPD (1)	Aqueous Precision RPD (1)
2-Methylnaphthalene-D10	8270C SIM	19-94	43-92	---	---
Fluorene-D10	8270C SIM	20-96	29-101	---	---
Pyrene-D10	8270C SIM	31-128	53-166	---	---
2,4-Dibromophenol	8270C SIM	20-116	10-130	---	---
Metals - ICP					
Copper	6010	Manufacturer (6)	80-120	0-20 (nominal)	0-20 (nominal)
Lead	6010	Manufacturer (6)	80-123	0-20 (nominal)	0-20 (nominal)
Zinc	6010	Manufacturer (6)	80-120	0-20 (nominal)	0-20 (nominal)
Inorganic (Misc.)					
TOC, Soil	SW846 9060M	80-120	NA	0-20	NA

LCS and Surrogate limits are updated annually

MDL's are updated annually

1. Represented as Relative Percent Difference
2. Accuracy is determined by Laboratory Control Samples and Matrix Spike/Matrix Spike Duplicates.
3. Accuracy for metals for MS/MSD only applies when the spike is > 4X the native analyte concentration.
4. Practical Quantitation Limits (PQLs) can increase based on percent water content and/or dilution factors.
5. The MDL and PQL for method 6020 include a dilution factor of 5 due to the matrix interference of Hydrochloric acid.
6. LCS limits are based on the manufacturer's limits for the SRM sample. Limits for MS/MSD are nominal (75-125%)
7. MDL's are determined annually therefore the MDLs listed on this table may change.
8. DoD QSM Version 4.1 limits

LABORATORY ACCREDITATION BUREAU

Certificate # L2223

Scope of Accreditation For Katahdin Analytical Services, Inc.

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In recognition of a successful assessment to ISO/IEC 17025:2005 and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (DoD QSM v4.1) based on the National Environmental Laboratory Accreditation Conference Chapter 5 Quality Systems Standard (NELAC Voted Revision June 5, 2003), accreditation is granted to Katahdin Analytical Services to perform the following tests:

Accreditation granted through: **November 4, 2012**

Testing - Environmental

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 608 / 8081B	4,4`-DDD
GC/ECD	EPA 608 / 8081B	4,4`-DDE
GC/ECD	EPA 608 / 8081B	4,4`-DDT
GC/ECD	EPA 608 / 8081B	Aldrin
GC/ECD	EPA 608 / 8081B	alpha-BHC (alpha-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	Alpha-Chlordane
GC/ECD	EPA 608 / 8081B	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	EPA 608 / 8081B	Chlordane (tech.)
GC/ECD	EPA 608 / 8081B	delta-BHC
GC/ECD	EPA 608 / 8081B	Dieldrin
GC/ECD	EPA 608 / 8081B	Endosulfan I
GC/ECD	EPA 608 / 8081B	Endosulfan II
GC/ECD	EPA 608 / 8081B	Endosulfan sulfate
GC/ECD	EPA 608 / 8081B	Endrin
GC/ECD	EPA 608 / 8081B	Endrin aldehyde
GC/ECD	EPA 8081B	Endrin Ketone
GC/ECD	EPA 8081B	gamma-BHC (Lindane gamma-Hexachlorocyclohexane)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8081B	gamma-Chlordane
GC/ECD	EPA 608 / 8081B	Heptachlor
GC/ECD	EPA 608 / 8081B	Heptachlor epoxide
GC/ECD	EPA 8081B	Methoxychlor
GC/ECD	EPA 608 / 8081B	Toxaphene (Chlorinated camphene)
GC/ECD	EPA 608 / 8082A	Aroclor-1016 (PCB-1016)
GC/ECD	EPA 608 / 8082A	Aroclor-1221 (PCB-1221)
GC/ECD	EPA 608 / 8082A	Aroclor-1232 (PCB-1232)
GC/ECD	EPA 608 / 8082A	Aroclor-1242 (PCB-1242)
GC/ECD	EPA 608 / 8082A	Aroclor-1248 (PCB-1248)
GC/ECD	EPA 608 / 8082A	Aroclor-1254 (PCB-1254)
GC/ECD	EPA 608 / 8082A	Aroclor-1260 (PCB-1260)
GC/ECD	EPA 8082A MOD	Aroclor-1262 (PCB-1262)
GC/ECD	EPA 8082A MOD	Aroclor-1268 (PCB-1268)
GC/ECD	EPA 8082A	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ 206)
GC/ECD	EPA 8082A	2,2',3,3',4,4',5,6-Octachlorobiphenyl (BZ 195)
GC/ECD	EPA 8082A	2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ 170)
GC/ECD	EPA 8082A	2,2',3,3',4,4'-Hexachlorobiphenyl (BZ 128)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5, 5'-Heptachlorobiphenyl (BZ 180)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5', 6-Heptachlorobiphenyl (BZ 183)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5'-Hexachlorobiphenyl (BZ 138)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 6, 6'-Heptachlorobiphenyl (BZ 184)
GC/ECD	EPA 8082A	2, 2', 3, 4', 5, 5', 6-Heptachlorobiphenyl (BZ 187)
GC/ECD	EPA 8082A	2, 2', 3, 4, 5'-Pentachlorobiphenyl (BZ 87)
GC/ECD	EPA 8082A	2, 2', 3, 5'-Tetrachlorobiphenyl (BZ 44)
GC/ECD	EPA 8082A	2, 2', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 153)
GC/ECD	EPA 8082A	2, 2', 4, 5, 5'-Pentachlorobiphenyl (BZ 101)
GC/ECD	EPA 8082A	2, 2', 4', 5-Tetrachlorobiphenyl (BZ 49)
GC/ECD	EPA 8082A	2, 2', 5, 5'-Tetrachlorobiphenyl (BZ 52)
GC/ECD	EPA 8082A	2, 2', 5-Trichlorobiphenyl (BZ 18)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5-Hexachlorobiphenyl (BZ 156)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5'-Hexachlorobiphenyl (BZ 157)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4'-Pentachlorobiphenyl (BZ 105)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5, 5'-Heptachlorobiphenyl (BZ 189)
GC/ECD	EPA 8082A	2, 3', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 167)
GC/ECD	EPA 8082A	2, 3', 4, 4', 5-Pentachlorobiphenyl (BZ 118)
GC/ECD	EPA 8082A	2, 3', 4, 4',5-Pentachlorobiphenyl (BZ 123)
GC/ECD	EPA 8082A	2, 3', 4, 4'-Tetrachlorobiphenyl (BZ 66)
GC/ECD	EPA 8082A	2, 3', 4, 4', 5-Pentachlorobiphenyl (BZ 114)
GC/ECD	EPA 8082A	2, 4, 4'-Trichlorobiphenyl (BZ 28)
GC/ECD	EPA 8082A	2, 4'-Dichlorobiphenyl (BZ 8)
GC/ECD	EPA 8082A	3, 3', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 169)
GC/ECD	EPA 8082A	3, 3', 4, 4', 5-Pentachlorobiphenyl (BZ 126)
GC/ECD	EPA 8082A	3, 3', 4, 4'-Tetrachlorobiphenyl (BZ 77)
GC/ECD	EPA 8082A	3, 4, 4', 5-Tetrachlorobiphenyl (BZ 81)
GC/ECD	EPA 8082A	Decachlorobiphenyl (BZ 209)
GC/ECD	EPA 8151A	2, 4, 5-T
GC/ECD	EPA 8151A	2, 4-D
GC/ECD	EPA 8151A	2, 4-DB
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichloroprop
GC/ECD	EPA 8151A	Dinoseb
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCPP
GC/ECD	EPA 8151A	Pentachlorophenol
GC/ECD	EPA 8151A	Silvex (2, 4, 5-TP)
GC/FID	EPA 8015B/C MOD	Diesel range organics (DRO)
GC/FID	EPA 8015B/C MOD	Gasoline range organics (GRO)
GC/FID/PID	MA DEP VPH	Volatile Organic Hydrocarbons
GC/FID	MA DEP EPH	Extractable Petroleum Hydrocarbons
GC/FID	TNRCC Method 1005	Total Petroleum Hydrocarbons
GC/FID	FL-PRO	Petroleum Range Organics
GC/ECD	EPA 8011 / 504	1, 2-Dibromoethane (EDB)
GC/ECD	EPA 8011 / 504	1, 2-Dibromo-3-chloropropane
GC/FID	RSK-175	Methane Ethane Ethene

Non-Potable Water

Technology	Method	Analyte
GC/MS	EPA 8260B,C / 524.2	1, 1, 1, 2-Tetrachloroethane
GC/MS	EPA 624 / 8260B,C / 524.2	1, 1, 1-Trichloroethane
GC/MS	EPA 624 / 8260B,C / 524.2	1, 1, 2, 2-Tetrachloroethane
GC/MS	EPA 8260B,C	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA 624 / 8260B,C / 524.2	1, 1, 2-Trichloroethane
GC/MS	EPA 624 / 8260B,C / 524.2	1, 1-Dichloroethane
GC/MS	EPA 624 / 8260B,C / 524.2	1, 1-Dichloroethene
GC/MS	EPA 8260B,C / 524.2	1, 1-Dichloropropene
GC/MS	EPA 8260B,C / 524.2	1, 2, 3-Trichlorobenzene
GC/MS	EPA 8260B,C / 524.2	1, 2, 3-Trichloropropane
GC/MS	EPA 8260B,C / 524.2	1, 2, 4-Trichlorobenzene
GC/MS	EPA 8260B,C / 524.2	1, 2, 4-Trimethylbenzene
GC/MS	EPA 8260B,C / 524.2	1, 2-Dibromo-3-chloropropane
GC/MS	EPA 8260B,C / 524.2	1, 2-Dibromoethane (EDB)
GC/MS	EPA 624 / 8260B,C / 524.2	1, 2-Dichlorobenzene
GC/MS	EPA 624 / 8260B,C / 524.2	1, 2-Dichloroethane
GC/MS	EPA 624 / 8260B,C / 524.2	1, 2-Dichloropropane
GC/MS	EPA 8260B,C / 524.2	1, 3, 5-Trimethylbenzene
GC/MS	EPA 624 / 8260B,C / 524.2	1, 3-Dichlorobenzene
GC/MS	EPA 8260B,C / 524.2	1, 3-Dichloropropane
GC/MS	EPA 624 / 8260B,C / 524.2	1, 4-Dichlorobenzene
GC/MS	EPA 8260B,C	1, 4-Dioxane
GC/MS	EPA 8260B,C / 524.2	2, 2-Dichloropropane
GC/MS	EPA 8260B,C / 524.2	2-Butanone
GC/MS	EPA 624 / 8260B,C	2-Chloroethyl vinyl ether
GC/MS	EPA 8260B,C / 524.2	2-Chlorotoluene
GC/MS	EPA 8260B,C / 524.2	2-Hexanone
GC/MS	EPA 8260B,C / 524.2	4-Chlorotoluene
GC/MS	EPA 8260B,C / 524.2	4-Methyl-2-pentanone
GC/MS	EPA 8260B,C / 524.2	Acetone
GC/MS	EPA 8260B,C	Acetonitrile
GC/MS	EPA 1624 / 8260B,C	Acrolein
GC/MS	EPA 624 / 8260B,C / 524.2	Acrylonitrile

Non-Potable Water

Technology	Method	Analyte
GC/MS	EPA 8260B,C / 524.2	Allyl chloride
GC/MS	EPA 624 / 8260B,C / 524.2	Benzene
GC/MS	EPA 8260B,C / 524.2	Bromobenzene
GC/MS	EPA 8260B,C / 524.2	Bromochloromethane
GC/MS	EPA 624 / 8260B,C / 524.2	Bromodichloromethane
GC/MS	EPA 624 / 8260B,C / 524.2	Bromoform
GC/MS	EPA 8260B,C / 524.2	Carbon disulfide
GC/MS	EPA 624 / 8260B,C / 524.2	Carbon tetrachloride
GC/MS	EPA 624 / 8260B,C / 524.2	Chlorobenzene
GC/MS	EPA 624 / 8260B,C / 524.2	Chloroethane
GC/MS	EPA 624 / 8260B,C / 524.2	Chloroform
GC/MS	EPA 8260B,C	Chloroprene
GC/MS	EPA 8260B,C / 524.2	cis-1, 2-Dichloroethene
GC/MS	EPA 624 / 8260B,C / 524.2	cis-1, 3-Dichloropropene
GC/MS	EPA 8260B,C	Cyclohexane
GC/MS	EPA 624 / 8260B,C / 524.2	Dibromochloromethane
GC/MS	EPA 8260B,C / 524.2	Dibromomethane
GC/MS	EPA 624 / 8260B,C / 524.2	Dichlorodifluoromethane
GC/MS	EPA 8260B,C / 524.2	Diethyl ether
GC/MS	EPA 8260B,C	Di-isopropylether
GC/MS	EPA 8260B,C / 524.2	Ethyl methacrylate
GC/MS	EPA 624 / 8260B,C / 524.2	Ethylbenzene
GC/MS	EPA 8260B,C	Ethyl-t-butylether
GC/MS	EPA 8260B,C / 524.2	Hexachlorobutadiene
GC/MS	EPA 8260B,C	Iodomethane
GC/MS	EPA 8260B,C	Isobutyl alcohol
GC/MS	EPA 8260B,C / 524.2	Isopropyl benzene
GC/MS	EPA 8260B,C / 524.2	m p-xylenes
GC/MS	EPA 8260B, C	Methyl acetate
GC/MS	EPA 8260B,C / 524.2	Methacrylonitrile
GC/MS	EPA 624 / 8260B,C	Methyl bromide (Bromomethane)
GC/MS	EPA 624 / 8260B,C / 524.2	Methyl chloride (Chloromethane)
GC/MS	EPA 8260B,C / 524.2	Methyl methacrylate

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B,C / 524.2	Methyl tert-butyl ether
GC/MS	EPA 8260B,C	Methylcyclohexane
GC/MS	EPA 624 / 8260B,C / 524.2	Methylene chloride
GC/MS	EPA 8260B,C / 524.2	Naphthalene
GC/MS	EPA 8260B,C / 524.2	n-Butylbenzene
GC/MS	EPA 8260B,C / 524.2	n-Propylbenzene
GC/MS	EPA 8260B,C / 524.2	o-Xylene
GC/MS	EPA 8260B,C / 524.2	p-Isopropyltoluene
GC/MS	EPA 8260B,C / 524.2	Propionitrile
GC/MS	EPA 8260B,C / 524.2	sec-butylbenzene
GC/MS	EPA 8260B,C / 524.2	Styrene
GC/MS	EPA 8260B,C	t-Amylmethylether
GC/MS	EPA 8260B,C / 524.2	tert-Butyl alcohol
GC/MS	EPA 8260B,C	tert-Butylbenzene
GC/MS	EPA 624 / 8260B,C / 524.2	Tetrachloroethene (Perchloroethylene)
GC/MS	EPA 8260B,C / 524.2	Tetrahydrofuran
GC/MS	EPA 624 / 8260B,C / 524.2	Toluene
GC/MS	EPA 624 / 8260B,C / 524.2	trans-1, 2-Dichloroethylene
GC/MS	EPA 624 / 8260B,C / 524.2	trans-1, 3-Dichloropropylene
GC/MS	EPA 8260B,C / 524.2	trans-1, 4-Dichloro-2-butuene
GC/MS	EPA 624 / 8260B,C / 524.2	Trichloroethene (Trichloroethylene)
GC/MS	EPA 624 / 8260B,C / 524.2	Trichlorofluoromethane
GC/MS	EPA 8260B,C	Vinyl acetate
GC/MS	EPA 624 / 8260B,C / 524.2	Vinyl chloride
GC/MS	EPA 624 / 8260B,C	Xylene
GC/MS	EPA 8270C,D	1, 2, 4, 5-Tetrachlorobenzene
GC/MS	EPA 625 / 8270C,D	1, 2, 4-Trichlorobenzene
GC/MS	EPA 625 / 8270C,D	1, 2-Dichlorobenzene
GC/MS	EPA 8270C,D	1, 2-Diphenylhydrazine
GC/MS	EPA 8270C,D	1, 3, 5-Trinitrobenzene
GC/MS	EPA 625 / 8270C,D	1, 3-Dichlorobenzene
GC/MS	EPA 8270C,D	1, 3-Dinitrobenzene
GC/MS	EPA 625 / 8270C,D	1, 4-Dichlorobenzene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C,D	1, 4-Dioxane
GC/MS	EPA 8270C,D	1, 4-Naphthoquinone
GC/MS	EPA 8270C,D	1, 4-Phenylenediamine
GC/MS	EPA 8270C,D	1-Naphthylamine
GC/MS	EPA 8270C,D	2, 3, 4, 6-Tetrachlorophenol
GC/MS	EPA 8270C,D	2, 4, 5-Trochlorophenol
GC/MS	EPA 625 / 8270C,D	2, 4, 6-Trichlorophenol
GC/MS	EPA 625 / 8270C,D	2, 4-Dichlorophenol
GC/MS	EPA 625 / 8270C,D	2, 4-Dimethylphenol
GC/MS	EPA 625 / 8270C,D	2, 4-Dinitrophenol
GC/MS	EPA 625 / 8270C,D	2, 4-Dinitrotoluene (2, 4-DNT)
GC/MS	EPA 8270C,D	2, 6-Dichlorophenol
GC/MS	EPA 625 / 8270C,D	2, 6-Dinitrotoluene (2, 6-DNT)
GC/MS	EPA 8270C,D	2-Acetylaminofluorene
GC/MS	EPA 625 / 8270C,D	2-Chloronaphthalene
GC/MS	EPA 625 / 8270C,D	2-Chlorophenol
GC/MS	EPA 625 / 8270C,D	2-Methyl-4 6-dinitrophenol
GC/MS	EPA 8270C,D	2-Methylnaphthalene
GC/MS	EPA 8270C,D	2-Methylphenol
GC/MS	EPA 8270C,D	2-Naphthylamine
GC/MS	EPA 8270C,D	2-Nitroaniline
GC/MS	EPA 625 / 8270C,D	2-Nitrophenol
GC/MS	EPA 8270C,D	2-Picoline
GC/MS	EPA 625 / 8270C,D	3, 3'-Dichlorobenzidine
GC/MS	EPA 8270C,D	3, 3'-Dimethylbenzidine
GC/MS	EPA 8270C,D	3-Methylcholanthrene
GC/MS	EPA 8270C,D	3-Nitroaniline
GC/MS	EPA 8270C,D	4-Aminobiphenyl
GC/MS	EPA 625 / 8270C,D	4-Bromophenyl phenyl ether
GC/MS	EPA 625 / 8270C,D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C,D	4-Chloroaniline
GC/MS	EPA 625 / 8270C,D	4-Chlorophenyl phenylether
GC/MS	EPA 8270C,D	4-Dimethyl aminoazobenzene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C,D	4-Methylphenol
GC/MS	EPA 8270C,D	4-Nitroaniline
GC/MS	EPA 625 / 8270C,D	4-Nitrophenol
GC/MS	EPA 8270C,D	5-Nitro-o-toluidine
GC/MS	EPA 8270C,D	7,12-Dimethylphenethylamine
GC/MS	EPA 8270C,D	a a-Dimethylphenethylamine
GC/MS	EPA 625 / 8270C,D	Acenaphthene
GC/MS	EPA 625 / 8270C,D	Acenaphthylene
GC/MS	EPA 8270C,D	Acetophenone
GC/MS	EPA 8270C,D	Aniline
GC/MS	EPA 625 / 8270C,D	Anthracene
GC/MS	EPA 8270C,D	Aramite
GC/MS	EPA 8270C,D	Atrazine
GC/MS	EPA 625 / 8270C,D	Benzidine
GC/MS	EPA 625 / 8270C,D	Benzo(a)anthracene
GC/MS	EPA 625 / 8270C,D	Benzo(a)pyrene
GC/MS	EPA 625 / 8270C,D	Benzo(b)fluoranthene
GC/MS	EPA 625 / 8270C,D	Benzo(g h i)perylene
GC/MS	EPA 625 / 8270C,D	Benzo(k)fluoranthene
GC/MS	EPA 8270C,D	Benzoic Acid
GC/MS	EPA 8270C,D	Benzyl alcohol
GC/MS	EPA 8270C,D	Biphenyl
GC/MS	EPA 625 / 8270C,D	bis(2-Chloroethoxy)methane
GC/MS	EPA 625 / 8270C,D	bis(2-Chloroethyl) ether
GC/MS	EPA 625 / 8270C,D	bis(2-Chloroisopropyl) ether (2, 2`-Oxybis(1-chloropropane))
GC/MS	EPA 625 / 8270C,D	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	EPA 625 / 8270C,D	Butyl benzyl phthalate
GC/MS	EPA 8270C,D	Carbazole
GC/MS	EPA 8270C,D	Chlorobenzilate
GC/MS	EPA 625 / 8270C,D	Chrysene
GC/MS	EPA 8270C,D	Diallate
GC/MS	EPA 625 / 8270C,D	Dibenz(a h)anthracene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C,D	Dibenzofuran
GC/MS	EPA 625 / 8270C,D	Diethyl phthalate
GC/MS	EPA 8270C,D	Dimethoate
GC/MS	EPA 625 / 8270C,D	Dimethyl phthalate
GC/MS	EPA 625 / 8270C,D	Di-n-butyl phthalate
GC/MS	EPA 625 / 8270C,D	Di-n-octyl phthalate
GC/MS	EPA 8270C,D	Ethyl methanesulfonate
GC/MS	EPA 8270C,D	Famfur
GC/MS	EPA 625 / 8270C,D	Fluoranthene
GC/MS	EPA 625 / 8270C,D	Fluorene
GC/MS	EPA 625 / 8270C,D	Hexachlorobenzene
GC/MS	EPA 625 / 8270C,D	Hexachlorobutadiene
GC/MS	EPA 625 / 8270C,D	Hexachlorocyclopentadiene
GC/MS	EPA 625 / 8270C,D	Hexachloroethane
GC/MS	EPA 8270C,D	Hexachloropropene
GC/MS	EPA 625 / 8270C,D	Indeno(1, 2, 3-cd)pyrene
GC/MS	EPA 8270C,D	Isodrin
GC/MS	EPA 625 / 8270C,D	Isophorone
GC/MS	EPA 8270C,D	Isosafrole
GC/MS	EPA 8270C,D	Methapyriline
GC/MS	EPA 8270C,D	Methy methanesulfonate
GC/MS	EPA 8270C,D	Methyl parathion
GC/MS	EPA 625 / 8270C,D	Naphthalene
GC/MS	EPA 625 / 8270C,D	Nitrobenzene
GC/MS	EPA 8270C,D	Nitroquinoline-1-oxide
GC/MS	EPA 8270C,D	n-Nitrosodiethylamine
GC/MS	EPA 625 / 8270C,D	n-Nitrosodimethylamine
GC/MS	EPA 8270C,D	n-Nitroso-di-n-butylamine
GC/MS	EPA 625 / 8270C,D	n-Nitrosodi-n-propylamine
GC/MS	EPA 625 / 8270C,D	n-Nitrosodiphenylamine
GC/MS	EPA 8270C,D	n-Nitrosomethylethylamine
GC/MS	EPA 8270C,D	n-Nitrosomorpholine
GC/MS	EPA 8270C,D	n-Nitrosopiperidine

Non-Potable Water

Technology	Method	Analyte
GC/MS	EPA 8270C,D	n-Nitrosopyrrolidine
GC/MS	EPA 8270C,D	o o o-Triethyl phosphorothioate
GC/MS	EPA 8270C,D	o-Toluidine
GC/MS	EPA 8270C,D	Pentachlorobenzene
GC/MS	EPA 8270C,D	Pentachloronitrobenzene
GC/MS	EPA 625 / 8270C,D	Pentachlorophenol
GC/MS	EPA 8270C,D	Phenacetin
GC/MS	EPA 625 / 8270C,D	Phenanthrene
GC/MS	EPA 625 / 8270C,D	Phenol
GC/MS	EPA 8270C,D	Phorate
GC/MS	EPA 8270C,D	Pronamide
GC/MS	EPA 625 / 8270C,D	Pyrene
GC/MS	EPA 8270C,D	Pyrididne
GC/MS	EPA 8270C,D	Safrole
GC/MS	EPA 8270C,D	Thionazin
HPLC/UV	EPA 8330A/8330B	1, 3, 5-Trinitrobenzene
HPLC/UV	EPA 8330A/8330B	1, 3-Dinitrobenzene
HPLC/UV	EPA 8330A/8330B	2, 4, 6-Trinitrotoluene
HPLC/UV	EPA 8330A/8330B	2, 4-Dinitrotoluene
HPLC/UV	EPA 8330A/8330B	2, 6-Dinitrotoluene
HPLC/UV	EPA 8330A/8330B	2-Amino-4, 6 -dinitrotoluene
HPLC/UV	EPA 8330A/8330B	2-Nitrotoluene
HPLC/UV	EPA 8330A/8330B	3-Nitrotoluene
HPLC/UV	EPA 8330A/8330B	4-Amino-2,3-dinitrotoluene
HPLC/UV	EPA 8330A/8330B	4-Nitrotoluene
HPLC/UV	EPA 8330A/8330B	Ethylene glycol dinitrate (EGDN)
HPLC/UV	EPA 8330A/8330B	Hexahydro-1, 3, 5-trinitro-1, 3, 5-triazine (RDX)
HPLC/UV	EPA 8330A/8330B	Nitrobenzene
HPLC/UV	EPA 8330A MOD	Nitroglycerin
HPLC/UV	EPA 8330B	Nitroglycerin
HPLC/UV	EPA 8330A/8330B	Octahydro-1, 3, 5, 7-tetrazocine (HMX)
HPLC/UV	EPA 8330A/8330B	Pentaerythritol Tetranitrate (PETN)
HPLC/UV	EPA 8330A/8330B	Tetryl

Non-Potable Water

Technology	Method	Analyte
CVAA	EPA 245.1 / 7470A	Mercury
CVAF	EPA 1631E	Low Level Mercury
ICP/AES	EPA 200.7 / 6010B,C	Aluminum
ICP/AES	EPA 200.7 / 6010B,C	Antimony
ICP/AES	EPA 200.7 / 6010B,C	Arsenic
ICP/AES	EPA 200.7 / 6010B,C	Barium
ICP/AES	EPA 200.7 / 6010B,C	Beryllium
ICP/AES	EPA 200.7 / 6010B,C	Boron
ICP/AES	EPA 200.7 / 6010B,C	Cadmium
ICP/AES	EPA 200.7 / 6010B,C	Calcium
ICP/AES	EPA 200.7 / 6010B,C	Chromium
ICP/AES	EPA 200.7 / 6010B,C	Cobalt
ICP/AES	EPA 200.7 / 6010B,C	Copper
ICP/AES	EPA 200.7 / 6010B,C	Iron
ICP/AES	EPA 200.7 / 6010B,C	Lead
ICP/AES	EPA 200.7 / 6010B,C	Magnesium
ICP/AES	EPA 200.7 / 6010B,C	Manganese
ICP/AES	EPA 200.7 / 6010B,C	Molybdenum
ICP/AES	EPA 200.7 / 6010B,C	Nickel
ICP/AES	EPA 200.7 / 6010B,C	Potassium
ICP/AES	EPA 200.7 / 6010B,C	Selenium
ICP/AES	EPA 200.7	Silicon
ICP/AES	EPA 200.7 / 6010B,C	Silver
ICP/AES	EPA 200.7 / 6010B,C	Sodium
ICP/AES	EPA 6010B,C	Strontium
ICP/AES	EPA 200.7 / 6010B,C	Thallium
ICP/AES	EPA 200.7 / 6010B,C	Tin
ICP/AES	EPA 200.7 / 6010B,C	Titanium
ICP/AES	EPA 200.7 / 6010B,C	Vanadium
ICP/AES	EPA 200.7 / 6010B,C	Zinc
ICP/MS	EPA 200.8 / 6020A	Aluminum
ICP/MS	EPA 200.8 / 6020A	Antimony
ICP/MS	EPA 200.8 / 6020A	Arsenic

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP/MS	EPA 200.8 / 6020A	Barium
ICP/MS	EPA 200.8 / 6020A	Beryllium
ICP/MS	EPA 200.8 / 6020A	Boron
ICP/MS	EPA 200.8 / 6020A	Cadmium
ICP/MS	EPA 200.8 / 6020A	Calcium
ICP/MS	EPA 200.8 / 6020A	Chromium
ICP/MS	EPA 200.8 / 6020A	Cobalt
ICP/MS	EPA 200.8 / 6020A	Copper
ICP/MS	EPA 200.8 / 6020A	Iron
ICP/MS	EPA 200.8 / 6020A	Lead
ICP/MS	EPA 200.8 / 6020A	Magnesium
ICP/MS	EPA 200.8 / 6020A	Manganese
ICP/MS	EPA 200.8 / 6020A	Molybdenum
ICP/MS	EPA 200.8 / 6020A	Nickel
ICP/MS	EPA 200.8 / 6020A	Potassium
ICP/MS	EPA 200.8 / 6020A	Selenium
ICP/MS	EPA 200.8 / 6020A	Silicon
ICP/MS	EPA 200.8 / 6020A	Silver
ICP/MS	EPA 200.8 / 6020A	Sodium
ICP/MS	EPA 6020A	Strontium
ICP/MS	EPA 200.8 / 6020A	Thallium
ICP/MS	EPA 200.8 / 6020A	Tin
ICP/MS	EPA 200.8 / 6020A	Titanium
ICP/MS	EPA 200.8	Uranium
ICP/MS	EPA 200.8 / 6020A	Vanadium
ICP/MS	EPA 200.8 / 6020A	Zinc
IC	EPA 300.0 / 9056A	Bromide
IC	EPA 300.0 / 9056A	Chloride
IC	EPA 300.0 / 9056A	Nitrate as N
IC	EPA 300.0 / 9056A	Nitrite as N
IC	EPA 300.0 / 9056A	Nitrate + Nitrite
IC	EPA 300.0 / 9056A	Orthophosphate as P
IC	EPA 300.0 / 9056A	Sulfate

Non-Potable Water		
Technology	**Method**	**Analyte**
Titration	EPA 310.2 / SM 2320B	Alkalinity
Caculation	SM 2340C	Hardness
Gravimetric	EPA 1664A	Oil and Grease
Gravimetric	SM 2540B,C,D	Solids
ISE	EPA 120.1 / SM 2510B	Conductivity
ISE	SM 2520B	Practical Salinity
ISE	SM 4500F- C	Fluoride
ISE	SM 4500H+ B	pH
ISE	SM 5210B	TBOD / CBOD
Physical	EPA 1010A	Ignitability
Physical	EPA 9040C	pH
Titration	SM 2340B	Hardness
Titration	SM 4500SO_3 B	Sulfite
Titration	EPA 9034 / SM 4500S^{2-} E	Sulfide
Titration	Chap. 7.3.4	Reactive Sulfide
IR	EPA 9060A / SM 5310B	Total organic carbon
Turbidimetric	EPA 180.1 / SM 2130B	Turbidity
Turbidimetric	EPA 9038 / ASTM 516-02	Sulfate
UV/VIS	EPA 335.4 / EPA 9012B / SM 4500-CN G	Amenable cyanide
UV/VIS	EPA 350.1 / SM 4500NH3 H	Ammonia as N
UV/VIS	SM 3500Fe D	Ferrous Iron
UV/VIS	EPA 351.2	Kjeldahl nitrogen - total
UV/VIS	EPA 353.2 / SM 4500NO3 F	Nitrate + Nitrite
UV/VIS	EPA 353.2 / SM 4500NO3 F	Nitrate as N
UV/VIS	EPA 353.2 / SM 4500NO3 F	Nitrite as N
UV/VIS	EPA 365.1 / SM 4500P E	Orthophosphate as P
UV/VIS	EPA 365.4	Phosphorus total
UV/VIS	EPA 376.3	AVS-SEM
UV/VIS	EPA 410.4	COD
UV/VIS	EPA 420.1 / 9065	Total Phenolics
UV/VIS	SM 4500Cl G	Total Residual Chlorine
UV/VIS	SM 5540C	MBAS

Non-Potable Water		
Technology	**Method**	**Analyte**
UV/VIS	EPA 7196A / SM 3500-Cr D	Chromium VI
UV/VIS	EPA 9012B / 335.4	Total Cyanide
UV/VIS	EPA 9251 / SM 4500Cl E	Chloride
UV/VIS	Chap. 7.3.4	Reactive Cyanide
Preparation	**Method**	**Type**
Cleanup Methods	EPA 3640A	Gel Permeation Clean-up
Cleanup Methods	EPA 3630C	Silica Gel
Cleanup Methods	EPA 3660B	Sulfur Clean-Up
Cleanup Methods	EPA 3665A	Sulfuric Acid Clean-Up
Organic Preparation	EPA 3510C	Separatory Funnel Extraction
Organic Preparation	EPA 3520C	Continuous Liquid-Liquid Extraction
Inorganic Preparation	EPA 3010A	Hotblock
Volatile Organic Preparation	EPA 5030C	Purge and Trap

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8081B	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 (alpha-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	Alpha-Chlordane
GC/ECD	EPA 8081B	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	EPA 608 / 8081B	Chlordane (tech.)
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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8081B	Endrin aldehyde
GC/ECD	EPA 8081B	Endrin Ketone
GC/ECD	EPA 8081B	gamma-BHC (Lindane gamma-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	gamma-Chlordane
GC/ECD	EPA 8081B	Heptachlor
GC/ECD	EPA 8081B	Heptachlor epoxide
GC/ECD	EPA 8081B	Methoxychlor
GC/ECD	EPA 8081B	Toxaphene (Chlorinated camphene)
GC/ECD	EPA 8082A	Aroclor-1016 (PCB-1016)
GC/ECD	EPA 8082A	Aroclor-1221 (PCB-1221)
GC/ECD	EPA 8082A	Aroclor-1232 (PCB-1232)
GC/ECD	EPA 8082A	Aroclor-1242 (PCB-1242)
GC/ECD	EPA 8082A	Aroclor-1248 (PCB-1248)
GC/ECD	EPA 8082A	Aroclor-1254 (PCB-1254)
GC/ECD	EPA 8082A	Aroclor-1260 (PCB-1260)
GC/ECD	EPA 8082A MOD	Aroclor-1262 (PCB-1262)
GC/ECD	EPA 8082A MOD	Aroclor-1268 (PCB-1268)
GC/ECD	EPA 8082A	2, 2', 3, 3', 4, 4', 5, 5', 6-Nonachlorobiphenyl (BZ 206)
GC/ECD	EPA 8082A	2, 2', 3, 3', 4, 4', 5, 6-Octachlorobiphenyl (BZ 195)
GC/ECD	EPA 8082A	2, 2', 3, 3', 4, 4', 5-Heptachlorobiphenyl (BZ 170)
GC/ECD	EPA 8082A	2, 2', 3, 3', 4, 4'-Hexachlorobiphenyl (BZ 128)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5, 5'-Heptachlorobiphenyl (BZ 180)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5', 6-Heptachlorobiphenyl (BZ 183)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5'-Hexachlorobiphenyl (BZ 138)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 6, 6'-Heptachlorobiphenyl (BZ 184)
GC/ECD	EPA 8082A	2, 2', 3, 4', 5, 5', 6-Heptachlorobiphenyl (BZ 187)
GC/ECD	EPA 8082A	2, 2', 3, 4, 5'-Pentachlorobiphenyl (BZ 87)
GC/ECD	EPA 8082A	2, 2', 3, 5'-Tetrachlorobiphenyl (BZ 44)
GC/ECD	EPA 8082A	2, 2', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 153)
GC/ECD	EPA 8082A	2, 2', 4, 5, 5'-Pentachlorobiphenyl (BZ 101)
GC/ECD	EPA 8082A	2, 2', 4', 5-Tetrachlorobiphenyl (BZ 49)
GC/ECD	EPA 8082A	2, 2', 5, 5'-Tetrachlorobiphenyl (BZ 52)
GC/ECD	EPA 8082A	2, 2', 5-Trichlorobiphenyl (BZ 18)

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5-Hexachlorobiphenyl (BZ 156)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5'-Hexachlorobiphenyl (BZ 157)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4'-Pentachlorobiphenyl (BZ 105)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5, 5'-Heptachlorobiphenyl (BZ 189)
GC/ECD	EPA 8082A	2, 3', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 167)
GC/ECD	EPA 8082A	2, 3', 4, 4', 5-Pentachlorobiphenyl (BZ 118)
GC/ECD	EPA 8082A	2, 3', 4, 4',5-Pentachlorobiphenyl (BZ 123)
GC/ECD	EPA 8082A	2, 3', 4, 4'-Tetrachlorobiphenyl (BZ 66)
GC/ECD	EPA 8082A	2, 3', 4, 4', 5-Pentachlorobiphenyl (BZ 114)
GC/ECD	EPA 8082A	2, 4, 4'-Trichlorobiphenyl (BZ 28)
GC/ECD	EPA 8082A	2, 4'-Dichlorobiphenyl (BZ 8)
GC/ECD	EPA 8082A	3, 3', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 169)
GC/ECD	EPA 8082A	3, 3', 4, 4', 5-Pentachlorobiphenyl (BZ 126)
GC/ECD	EPA 8082A	3, 3', 4, 4'-Tetrachlorobiphenyl (BZ 77)
GC/ECD	EPA 8082A	3, 4, 4', 5-Tetrachlorobiphenyl (BZ 81)
GC/ECD	EPA 8082A	Decachlorobiphenyl (BZ 209)
GC/ECD	EPA 8151A	2, 4, 5-T
GC/ECD	EPA 8151A	2, 4-D
GC/ECD	EPA 8151A	2, 4-DB
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichloroprop
GC/ECD	EPA 8151A	Dinoseb
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCPP
GC/ECD	EPA 8151A	Pentachlorophenol
GC/ECD	EPA 8151A	Silvex (2, 4, 5-TP)
GC/FID	EPA 8015C	Diesel range organics (DRO)
GC/FID	EPA 8015C	Gasoline range organics (GRO)
GC/FID/PID	MA DEP VPH	Volatile Organic Hydrocarbons
GC/FID	MA DEP EPH	Extractable Petroleum Hydrocarbons
GC/FID	TNRCC Method 1005	Total Petroleum Hydrocarbons
GC/FID	FL-PRO	Petroleum Range Organics

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8011	1, 2-Dibromoethane (EDB)
GC/ECD	EPA 8011	1, 2-Dibromo-3-chloropropane
GC/MS	EPA 8260B,C	1, 1, 1, 2-Tetrachloroethane
GC/MS	EPA 8260B,C	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA 8260B,C	1, 1, 1-Trichloroethane
GC/MS	EPA 8260B,C	1, 1, 2, 2-Tetrachloroethane
GC/MS	EPA 8260B,C	1, 1, 2-Trichloroethane
GC/MS	EPA 8260B,C	1, 1-Dichloroethane
GC/MS	EPA 8260B,C	1, 1-Dichloroethylene
GC/MS	EPA 8260B,C	1, 1-Dichloropropene
GC/MS	EPA 8260B,C	1, 2, 3-Trichlorobenzene
GC/MS	EPA 8260B,C	1, 2, 3-Trichloropropane
GC/MS	EPA 8260B,C	1, 2, 4-Trichlorobenzene
GC/MS	EPA 8260B,C	1, 2, 4-Trimethylbenzene
GC/MS	EPA 8260B,C	1, 2-Dibromo-3-chloropropane
GC/MS	EPA 8260B,C	1, 2-Dichlorobenzene
GC/MS	EPA 8260B,C	1, 2-Dichloroethane
GC/MS	EPA 8260B,C	1, 2-Dichloropropane
GC/MS	EPA 8260B,C	1, 3, 5-Trimethylbenzene
GC/MS	EPA 8260B,C	1, 3-Dichlorobenzene
GC/MS	EPA 8260B,C	1, 3-Dichloropropane
GC/MS	EPA 8260B,C	1, 4-Dichlorobenzene
GC/MS	EPA 8260B,C	1, 4-Dioxane
GC/MS	EPA 8260B,C	2, 2-Dichloropropane
GC/MS	EPA 8260B,C	2-Butanone
GC/MS	EPA 8260B,C	2-Chloroethyl vinyl ether
GC/MS	EPA 8260B,C	2-Chlorotoluene
GC/MS	EPA 8260B,C	2-Hexanone
GC/MS	EPA 8260B,C	4-Chlorotoluene
GC/MS	EPA 8260B,C	4-Methyl-2-pentanone
GC/MS	EPA 8260B,C	Acetone
GC/MS	EPA 8260B,C	Acetonitrile
GC/MS	EPA 8260B,C	Acrolein

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B,C	Acrylonitrile
GC/MS	EPA 8260B,C	Allyl chloride
GC/MS	EPA 8260B,C	Benzene
GC/MS	EPA 8260B,C	Bromobenzene
GC/MS	EPA 8260B,C	Bromochloromethane
GC/MS	EPA 8260B,C	Bromodichloromethane
GC/MS	EPA 8260B,C	Bromoform
GC/MS	EPA 8260B,C	Carbon disulfide
GC/MS	EPA 8260B,C	Carbon tetrachloride
GC/MS	EPA 8260B,C	Chlorobenzene
GC/MS	EPA 8260B,C	Chloroethane
GC/MS	EPA 8260B,C	Chloroform
GC/MS	EPA 8260B,C	Chloroprene
GC/MS	EPA 8260B,C	cis-1, 2-Dichloroethene
GC/MS	EPA 8260B,C	cis-1, 3-Dichloropropene
GC/MS	EPA 8260B,C	Cyclohexane
GC/MS	EPA 8260B,C	Dibromochloromethane
GC/MS	EPA 8260B,C	Dibromomethane
GC/MS	EPA 8260B,C	Dichlorodifluoromethane
GC/MS	EPA 8260B,C	Diethyl ether
GC/MS	EPA 8260B,C	Di-isopropylether
GC/MS	EPA 8260B,C	1,2-Dibromoethane (EDB)
GC/MS	EPA 8260B,C	Ethyl methacrylate
GC/MS	EPA 8260B,C	Ethylbenzene
GC/MS	EPA 8260B,C	Ethyl-t-butylether
GC/MS	EPA 8260B,C	Hexachlorobutadiene
GC/MS	EPA 8260B,C	Iodomethane
GC/MS	EPA 8260B,C	Isobutyl alcohol
GC/MS	EPA 8260B,C	Isopropyl benzene
GC/MS	EPA 8260B, C	Methyl acetate
GC/MS	EPA 8260B,C	Methacrylonitrile
GC/MS	EPA 8260B,C	Methyl bromide (Bromomethane)
GC/MS	EPA 8260B,C	Methyl chloride (Chloromethane)

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B,C	Methyl methacrylate
GC/MS	EPA 8260B,C	Methyl tert-butyl ether
GC/MS	EPA 8260B,C	Methylcyclohexane
GC/MS	EPA 8260B,C	Methylene chloride
GC/MS	EPA 8260B,C	Naphthalene
GC/MS	EPA 8260B,C	n-Butylbenzene
GC/MS	EPA 8260B,C	n-proplybenzene
GC/MS	EPA 8260B,C	o-Xylene
GC/MS	EPA 8260B,C	p-Isopropyltoluene
GC/MS	EPA 8260B,C	Propionitrile
GC/MS	EPA 8260B,C	sec-butylbenzene
GC/MS	EPA 8260B,C	Styrene
GC/MS	EPA 8260B,C	t-Amylmethylether
GC/MS	EPA 8260B,C	tert-Butyl alcohol
GC/MS	EPA 8260B,C	tert-Butylbenzene
GC/MS	EPA 8260B,C	Tetrachloroethylene (Perchloroethylene)
GC/MS	EPA 8260B,C	Tetrahydrofuran
GC/MS	EPA 8260B,C	Toluene
GC/MS	EPA 8260B,C	trans-1, 2-Dichloroethylene
GC/MS	EPA 8260B,C	trans-1, 3-Dichloropropylene
GC/MS	EPA 8260B,C	Trans-1, 4-Dichloro-2-butuene
GC/MS	EPA 8260B,C	Trichloroethene (Trichloroethylene)
GC/MS	EPA 8260B,C	Trichlorofluoromethane
GC/MS	EPA 8260B,C	Vinyl acetate
GC/MS	EPA 8260B,C	Vinyl chloride
GC/MS	EPA 8260B,C	Xylene
GC/MS	EPA 8270C,D	1-Naphthylamine
GC/MS	EPA 8270C,D	2-Acetylaminofluorene
GC/MS	EPA 8270C,D	2-Chloronaphthalene
GC/MS	EPA 8270C,D	2-Chlorophenol
GC/MS	EPA 8270C,D	2-Methylnaphthalene
GC/MS	EPA 8270C,D	2-Methylphenol
GC/MS	EPA 8270C,D	2-Naphthylamine

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C,D	2-Nitroaniline
GC/MS	EPA 8270C,D	2-Nitrophenol
GC/MS	EPA 8270C,D	2-Picoline
GC/MS	EPA 8270C,D	3-Methylcholanthrene
GC/MS	EPA 8270C,D	3-Nitroaniline
GC/MS	EPA 8270C,D	4-Aminobiphenyl
GC/MS	EPA 8270C,D	4-Bromophenyl phenyl ether
GC/MS	EPA 8270C,D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C,D	4-Chloroaniline
GC/MS	EPA 8270C,D	4-Chlorophenyl phenylether
GC/MS	EPA 8270C,D	4-Dimethyl aminoazobenzene
GC/MS	EPA 8270C,D	4-Methylphenol
GC/MS	EPA 8270C,D	4-Nitroaniline
GC/MS	EPA 8270C,D	4-Nitrophenol
GC/MS	EPA 8270C,D	5-Nitro-o-toluidine
GC/MS	EPA 8270C,D	a a-Dimethylphenethylamine
GC/MS	EPA 8270C,D	Acenaphthene
GC/MS	EPA 8270C,D	Acenaphthylene
GC/MS	EPA 8270C,D	Acetophenone
GC/MS	EPA 8270C,D	Aniline
GC/MS	EPA 8270C,D	Anthracene
GC/MS	EPA 8270C,D	Aramite
GC/MS	EPA 8270C,D	Atrazine
GC/MS	EPA 8270C,D	Benzidine
GC/MS	EPA 8270C,D	Benzo(a)anthracene
GC/MS	EPA 8270C,D	Benzo(a)pyrene
GC/MS	EPA 8270C,D	Benzo(b)fluoranthene
GC/MS	EPA 8270C,D	Benzo(g h i)perylene
GC/MS	EPA 8270C,D	Benzo(k)fluoranthene
GC/MS	EPA 8270C,D	Benzoic Acid
GC/MS	EPA 8270C,D	Benzyl alcohol
GC/MS	EPA 8270C,D	Biphenyl
GC/MS	EPA 8270C,D	bis(2-Chloroethoxy)methane

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C,D	bis(2-Chloroethyl) ether
GC/MS	EPA 8270C,D	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	EPA 8270C,D	Butyl benzyl phthalate
GC/MS	EPA 8270C,D	Carbazole
GC/MS	EPA 8270C,D	Chlorobenzilate
GC/MS	EPA 8270C,D	Chrysene
GC/MS	EPA 8270C,D	Diallate
GC/MS	EPA 8270C,D	Dibenz(a h)anthracene
GC/MS	EPA 8270C,D	Dibenzofuran
GC/MS	EPA 8270C,D	Diethyl phthalate
GC/MS	EPA 8270C,D	Dimethoate
GC/MS	EPA 8270C,D	Dimethyl phthalate
GC/MS	EPA 8270C,D	Di-n-butyl phthalate
GC/MS	EPA 8270C,D	Di-n-octyl phthalate
GC/MS	EPA 8270C,D	Ethyl methanesulfonate
GC/MS	EPA 8270C,D	Famfur
GC/MS	EPA 8270C,D	Fluoranthene
GC/MS	EPA 8270C,D	Fluorene
GC/MS	EPA 8270C,D	Hexachlorobenzene
GC/MS	EPA 8270C,D	Hexachlorobutadiene
GC/MS	EPA 8270C,D	Hexachlorocyclopentadiene
GC/MS	EPA 8270C,D	Hexachloroethane
GC/MS	EPA 8270C,D	Hexachloropropene
GC/MS	EPA 8270C,D	Isodrin
GC/MS	EPA 8270C,D	Isophorone
GC/MS	EPA 8270C,D	Isosafrole
GC/MS	EPA 8270C,D	Methapyriline
GC/MS	EPA 8270C,D	Methyl methanesulfonate
GC/MS	EPA 8270C,D	Methyl parathion
GC/MS	EPA 8270C,D	Naphthalene
GC/MS	EPA 8270C,D	Nitrobenzene
GC/MS	EPA 8270C,D	Nitroquinoline-1-oxide
GC/MS	EPA 8270C,D	n-Nitrosodiethylamine

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C,D	n-Nitrosodimethylamine
GC/MS	EPA 8270C,D	n-Nitroso-di-n-butylamine
GC/MS	EPA 8270C,D	n-Nitrosodi-n-propylamine
GC/MS	EPA 8270C,D	n-Nitrosodiphenylamine
GC/MS	EPA 8270C,D	n-Nitrosomethylethylamine
GC/MS	EPA 8270C,D	n-Nitrosomorpholine
GC/MS	EPA 8270C,D	n-Nitrosopiperidine
GC/MS	EPA 8270C,D	n-Nitrosopyrrolidine
GC/MS	EPA 8270C,D	o o o-Triethyl phosphorothioate
GC/MS	EPA 8270C,D	o-Toluidine
GC/MS	EPA 8270C,D	Pentachlorobenzene
GC/MS	EPA 8270C,D	Pentachloronitrobenzene
GC/MS	EPA 8270C,D	Pentachlorophenol
GC/MS	EPA 8270C,D	Phenacetin
GC/MS	EPA 8270C,D	Phenanthrene
GC/MS	EPA 8270C,D	Phenol
GC/MS	EPA 8270C,D	Phorate
GC/MS	EPA 8270C,D	Pronamide
GC/MS	EPA 8270C,D	Pyrene
GC/MS	EPA 8270C,D	Pyrididne
GC/MS	EPA 8270C,D	Safrole
GC/MS	EPA 8270C,D	Thionazin
GC/MS	EPA 8270C,D	Indeno(1, 2, 3-cd)pyrene
GC/MS	EPA 8270C,D	1, 2, 4-Trichlorobenzene
GC/MS	EPA 8270C,D	1, 3, 5-Trinitrobenzene
GC/MS	EPA 8270C,D	1, 2, 4, 5-Tetrachlorobenzene
GC/MS	EPA 8270C,D	2, 4, 5-Trochlorophenol
GC/MS	EPA 8270C,D	2, 4, 6-Trichlorophenol
GC/MS	EPA 8270C,D	2, 3, 4, 6-Tetrachlorophenol
GC/MS	EPA 8270C,D	1, 2-Dichlorobenzene
GC/MS	EPA 8270C,D	1, 2-Diphenylhydrazine
GC/MS	EPA 8270C,D	1, 3-Dichlorobenzene
GC/MS	EPA 8270C,D	1, 3-Dinitrobenzene

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C,D	1, 4-Dichlorobenzene
GC/MS	EPA 8270C,D	1, 4-Dioxane
GC/MS	EPA 8270C,D	1, 4-Naphthoquinone
GC/MS	EPA 8270C,D	1, 4-Phenylenediamine
GC/MS	EPA 8270C,D	bis(2-Chloroisopropyl) ether (2, 2`-Oxybis(1-chloropropane))
GC/MS	EPA 8270C,D	2, 4-Dichlorophenol
GC/MS	EPA 8270C,D	2, 4-Dimethylphenol
GC/MS	EPA 8270C,D	2, 4-Dinitrophenol
GC/MS	EPA 8270C,D	2, 4-Dinitrotoluene (2 4-DNT)
GC/MS	EPA 8270C,D	2, 6-Dichlorophenol
GC/MS	EPA 8270C,D	2, 6-Dinitrotoluene (2 6-DNT)
GC/MS	EPA 8270C,D	3, 3`-Dichlorobenzidine
GC/MS	EPA 8270C,D	3, 3'-Dimethylbenzidine
GC/MS	EPA 8270C,D	2-Methyl-4, 6-dinitrophenol
GC/MS	EPA 8270C,D	7,12-Dimethylphenethylamine
HPLC/UV	EPA 8330A	1 ,3, 5-Trinitrobenzene
HPLC/UV	EPA 8330A	1, 3-Dinitrobenzene
HPLC/UV	EPA 8330A	2, 4, 6-Trinitrotoluene
HPLC/UV	EPA 8330A	2, 4-Dinitrotoluene
HPLC/UV	EPA 8330A	2, 6-Dinitrotoluene
HPLC/UV	EPA 8330A	2-Amino-4, 6 -dinitrotoluene
HPLC/UV	EPA 8330A	2-Nitrotoluene
HPLC/UV	EPA 8330A	3-Nitrotoluene
HPLC/UV	EPA 8330A	4-Amino-2,3-dinitrotoluene
HPLC/UV	EPA 8330A	4-Nitrotoluene
HPLC/UV	EPA 8330A	Ethylene glycol dinitrate (EGDN)
HPLC/UV	EPA 8330A	Hexahydr-1, 3, 5-trinitro-1, 3, 5-triazine (RDX)
HPLC/UV	EPA 8330A	Nitrobenzene
HPLC/UV	EPA 8330A MOD	Nitroglycerin
HPLC/UV	EPA 8330A	Octahydro-1, 3, 5, 7-tetrazocine (HMX)
HPLC/UV	EPA 8330A	Pentaerythritol Tetranitrate (PETN)
HPLC/UV	EPA 8330A	Tetryl

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
HPLC/UV	8330B (W/O Soil Grinding)	1, 3, 5-Trinitrobenzene
HPLC/UV	8330B (W/O Soil Grinding)	1, 3-Dinitrobenzene
HPLC/UV	8330B (W/O Soil Grinding)	2, 4, 6-Trinitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	2, 4-Dinitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	2, 6-Dinitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	2-Amino-4, 6 -dinitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	2-Nitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	3-Nitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	4-Amino-2,3-dinitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	4-Nitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	Ethylene glycol dinitrate (EGDN)
HPLC/UV	8330B (W/O Soil Grinding)	Hexahydr-1, 3, 5-trinitro-1, 3, 5-triazine (RDX)
HPLC/UV	8330B (W/O Soil Grinding)	Nitrobenzene
HPLC/UV	8330B (W/O Soil Grinding)	Nitroglycerin
HPLC/UV	8330B (W/O Soil Grinding)	Octahydro-1, 3, 5, 7-tetrazocine (HMX)
HPLC/UV	8330B (W/O Soil Grinding)	Pentaerythritol Tetranitrate (PETN)
HPLC/UV	8330B (W/O Soil Grinding)	Tetryl
CVAA	EPA 7471B	Mercury
CVAF	EPA 1631E	Low Level Mercury
ICP/AES	EPA 6010B,C	Aluminum
ICP/AES	EPA 6010B,C	Antimony
ICP/AES	EPA 6010B,C	Arsenic
ICP/AES	EPA 6010B,C	Barium
ICP/AES	EPA 6010B,C	Beryllium
ICP/AES	EPA 6010B,C	Boron
ICP/AES	EPA 6010B,C	Cadmium
ICP/AES	EPA 6010B,C	Calcium
ICP/AES	EPA 6010B,C	Chromium
ICP/AES	EPA 6010B,C	Cobalt
ICP/AES	EPA 6010B,C	Copper
ICP/AES	EPA 6010B,C	Iron
ICP/AES	EPA 6010B,C	Lead
ICP/AES	EPA 6010B,C	Magnesium

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
ICP/AES	EPA 6010B,C	Manganese
ICP/AES	EPA 6010B,C	Molybdenum
ICP/AES	EPA 6010B,C	Nickel
ICP/AES	EPA 6010B,C	Potassium
ICP/AES	EPA 6010B,C	Selenium
ICP/AES	EPA 200.7	Silicon
ICP/AES	EPA 6010B,C	Silver
ICP/AES	EPA 6010B,C	Sodium
ICP/AES	EPA 6010B,C	Strontium
ICP/AES	EPA 6010B,C	Thallium
ICP/AES	EPA 6010B,C	Tin
ICP/AES	EPA 6010B,C	Titanium
ICP/AES	EPA 6010B,C	Vanadium
ICP/AES	EPA 6010B,C	Zinc
ICP/MS	EPA 6020A	Aluminum
ICP/MS	EPA 6020A	Antimony
ICP/MS	EPA 6020A	Arsenic
ICP/MS	EPA 6020A	Barium
ICP/MS	EPA 6020A	Beryllium
ICP/MS	EPA 6020A	Boron
ICP/MS	EPA 6020A	Cadmium
ICP/MS	EPA 6020A	Calcium
ICP/MS	EPA 6020A	Chromium
ICP/MS	EPA 6020A	Cobalt
ICP/MS	EPA 6020A	Copper
ICP/MS	EPA 6020A	Iron
ICP/MS	EPA 6020A	Lead
ICP/MS	EPA 6020A	Magnesium
ICP/MS	EPA 6020A	Manganese
ICP/MS	EPA 6020A	Molybdenum
ICP/MS	EPA 6020A	Nickel
ICP/MS	EPA 6020A	Potassium
ICP/MS	EPA 6020A	Selenium

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
ICP/MS	EPA 6020A	Silver
ICP/MS	EPA 6020A	Sodium
ICP/MS	EPA 6020A	Strontium
ICP/MS	EPA 6020A	Thallium
ICP/MS	EPA 6020A	Tin
ICP/MS	EPA 6020A	Titanium
ICP/MS	EPA 6020A	Vanadium
ICP/MS	EPA 6020A	Zinc
IC	EPA 9056A	Chloride
IC	EPA 9056A	Fluoride
IC	EPA 9056A	Nitrate as N
IC	EPA 9056A	Nitrite as N
IC	EPA 9056A	Sulfate
Gravimetric	EPA 9070A / 9071B	Oil and Grease
Physical	EPA 1010A	Ignitability
Physical	EPA 9045D	pH
Titration	Chap 7.3.4	Reactive Sulfide
IR	Lloyd Kahn	Total organic carbon
Turbidimetric	EPA 9038 / ASTM 516-02	Sulfate
UV/VIS	EPA 350.1 / SM 4500NH3 H	Ammonia as N
UV/VIS	EPA 9251 / SM 4500Cl E	Chloride
UV/VIS	Chap. 7.3.4	Reactive Cyanide
UV/VIS	EPA 376.3	AVS-SEM
UV/VIS	SM 3500Fe D	Ferrous Iron
Cleanup Methods	EPA 3630C	Silica Gel
UV/VIS	EPA 7196	Chromium VI
UV/VIS	EPA 7196A	Chromium VI
UV/VIS	EPA 9012B	Total cyanide
Preparation	**Method**	**Type**
Preparation	EPA 1311	Toxicity Characteristic Leaching Procedure
Preparation	EPA 1312	Synthetic Precipitation Leaching Procedure
Cleanup Methods	EPA 3660B	Sulfur Clean-up
Cleanup Methods	EPA 3620C	Florsil Clean-up

LABORATORY ACCREDITATION BUREAU

Certificate # L2223

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
Preparation	**Method**	**Type**
Cleanup Methods	EPA 3630C	Silica Gel Clean-up
Cleanup Methods	EPA 3640A	GPC Clean-up
Organic Preparation	EPA 3540C	Soxhlet Extraction
Organic Preparation	EPA 3545A	Pressurized Fluid Extraction
Organic Preparation	EPA 3550C	Sonication
Inorganics Preparation	EPA 3050B	Hotblock
Inorganics Preparation	EPA 3060A	Alkaline Digestion
Volatile Organics Preparation	EPA 5035/5035A	Closed System Purge and Trap

Notes:

1) This laboratory offers commercial testing service.

Approved By: _____ Date: January 20, 2011

R. Douglas Leonard
Chief Technical Officer

Issued: 11/04/09 Revised: 01/11/10 Revised: 04/06/10 Revised: 9/9/10 Revised: 10/13/10
Revised: 1/20/11

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: SD-902
Revision History
Cover Page
Page 1

TITLE: **SAMPLE RECEIPT AND INTERNAL CONTROL**

Prepared By: Andrea Colby Date: 6/2002

Approved By:

Group Supervisor: Andrea Colby Date: 6/6/02

Lab Operations Mgr: J. C. Burton Date: 6/5/02

QA Officer: Deborah J. Nadeau Date: 6.6.02

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
04	Changed cover sheet, minor changes to sections 7.1, 7.6, 7.7.4, 7.10 + 7.20. Complete rewrite of sections 7.11 + 7.12 to comply with new KIMS	DN	6.6.02	6.6.02
05	Added verbal date entry to KIMS. Added reference to immediate internal COC book. Added Department Manager reference. Added section 7.7.3. Updated new incoming	DN	05.04	05.04
06	Added procedure + logbook page for checking turbidity of drinking water samples. Changed wet chem shorts board to a book (included example page). Added custody procedures for food/micro. Added VOA soil freezer storage.	DN	01.26.04	01.26.04
07	Added instructions to create lettered labels. Changed sample locations to reflect new building. Removed Figures 8 and 10. Updated Table and Figures w/ current ones. Added wording to sect. 7.7.5 to clarify how pH measurements are taken.	LAD	02/07	02/07
08	Added summary stating sample acceptance policy. Deleted all references to radiation checks (not performed). Add IR gun usage. Reorganized section 7.0 to prioritize time sensitive tasks. Added wireless thermometer monitoring. Updated SRCR. Other minor changes.	DN	~~05/09~~ 08/09 8.4.09	~~05/09~~ 08/09

Added section concerning locking of coolers. Added more detail to 7.18 on unique container IDs. Added more detail on immediate COCs + a section on retention of samples.

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SD-902-08**, titled **Sample Receipt and Internal Control**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SD-902-08**, titled **Sample Receipt and Internal Control**.

Recipient: _____ Date: _____

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

1.0 SCOPE AND APPLICATION

Katahdin Analytical Services, Inc. requires the use of specific receiving, acceptance, identification, storage, and distribution procedures for samples it accepts for analyses. These procedures assure that:

- samples are uniquely identified,
- samples are protected from loss or damage,
- essential sample characteristics are preserved,
- any alteration of samples (e.g., filtration, preservation) is documented,
- the correct samples are analyzed, and
- a record of continuous sample custody and utilization is established.

The purpose of this SOP is to describe the procedures used for the receipt and tracking of samples received by Katahdin Analytical Services, Inc. (Katahdin).

1.1 Definitions

SDG: Sample Delivery Group – A group of samples to be reported as one data package.

1.2 Responsibilities

It is the responsibility of all Katahdin staff who receive samples or handle samples in the course of analysis to follow the procedures set forth in this SOP, to document their understanding of the procedures in their training files (refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability"), and to suggest changes and revisions when appropriate. All technical staff are responsible for monitoring their immediate areas, stopping an activity when a problem is detected or suspected, initiating corrective action when needed, documenting any actions taken, and notifying the appropriate individual (e.g., Department Manager, Operations Manager, QAO). The primary responsibility for implementing real-time corrective actions and maintaining an effective QA self-inspection system resides with Katahdin staff. When problems are identified Katahdin personnel are expected to attempt to resolve situations within the scope of their technical knowledge, and to seek assistance from peers and the Department Manager as necessary.

It is the responsibility of Department Managers to oversee the adherence to Katahdin QC practices and internal documentation of laboratory activities within their area, to take corrective actions where needed and communicate problems to the Operations Manager, QAO or Vice President/President when warranted.

It is the responsibility of the Operations Manager to oversee adherence to Katahdin QA/QC practices by all laboratory groups under his/her authority, to help identify

problems and assure resolution, to facilitate corrective action where needed, and to communicate problems and concerns to the QAO and Vice President/President.

It is the responsibility of the Quality Assurance Officer (QAO) to oversee adherence to this SOP, to conduct periodic audits of each laboratory, to track corrective action reports, resolution, and documentation, and to communicate concerns and report findings to the Vice President/President. The QA Officer shall function independently from laboratory operations and be able to evaluate data objectively and perform assessments without outside influence. The QA Officer has the authority to independently halt production operations (including data reporting) if warranted by quality problems.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Environmental Health & Safety Manual and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes generated during the receipt of samples must be disposed of in accordance with the Katahdin Environmental Health & Safety Manual and SOPs SD-903, "Sample Disposal" and CA-107, "The Management of Hazardous Waste as it Relates to the Disposal of Laboratory Process Waste, Reagents, Solvents and

Standards," current revisions. Expired standards are placed in the Katahdin hazardous waste storage area, and disposed of in accordance with these SOPs.

2.0 SUMMARY OF METHOD

Regulatory, program, and/or method requirements dictate the specifics of sample acceptance. These requirements include, but are not limited to, temperature upon receipt, chemical preservation, container type, sample amount, holding time considerations and complete and accurate documentation of all of these conditions, as well as sample identification. Katahdin's sample acceptance policy is to note any anomalies, discrepancies or non-compliances concerning the receipt of samples. The client is always notified with these issues to direct Katahdin on how and whether to proceed with analysis. All guidance from the client is recorded in the project phone logs and/or on the Sample Receipt Condition Report, which becomes part of the final report. Conditions or analyses performed which do not meet the necessary requirements are narrated or notated as described in the individual analytical SOPs.

3.0 INTERFERENCES

Not applicable.

4.0 APPARATUS AND MATERIALS

4.1 Thermometer – Oakton® Non-Contact Infrared Thermometer, or equivalent, capable of reading 0.1°C and digital probe style capable of reading 0.1°C (used for back-up).

4.2 Capillary tubes – 75 mm Hematocrit Tubes, disposable

4.3 Wide range pH test strips, pH 0 to 14 pH, EMD ColorpHast or equivalent.

4.4 Narrow range pH test strips, pH 0 to 2.5 pH, EMD ColorpHast or equivalent.

4.5 Narrow range pH test strips, pH 11 to 13 pH, EMD ColorpHast or equivalent.

5.0 REAGENTS

Preservatives - refer to Table 1, Sampling and Preservation Requirements, for specifics.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Refer to Table 1, Sampling and Preservation Requirements, for specifics.

7.0 PROCEDURES

PROCEDURES FOR SAMPLE CUSTODIAN

The following procedures include all steps to be completed for satisfactory receipt and acceptance of samples at Katahdin. These steps do not necessarily have to be performed in the exact order as described. Sample deliveries occur constantly throughout the day, so the sample custodian must multi-task and move back and forth between different procedures to accomplish the most critical tasks of checking receipt temperatures and checking for "RUSH" or quick hold time parameters.

7.1 When samples (except for non-environmental food samples) are dropped off, by either a delivery service (i.e. FEDEX or UPS) or by the client, the Chain-of-Custody (COC) should be signed immediately. The client (who is delivering or that has shipped samples with a delivery service) shall sign (at the lab upon delivery or prior to shipment of samples) that they have relinquished custody to the laboratory. The laboratory shall sign and record the date and time that custody is accepted. (Refer to Figures 1-3 for a Katahdin standard COC, a Katahdin Homeowner COC, and a Katahdin Food/Microbiology COC).

7.2 Cut custody seals and open all coolers. Remove the packets containing the client Chains-of-Custody (COCs).

7.3 Using the COCs, enter the date and time of sample receipt and the client name into the next available work order/login number in the sample receipt logbook (Figure 4). Initial each entry (line) to maintain a record of the individual who assigned each group of samples a discreet lab work order/login number. Record the assigned work order numbers in the appropriate space on the client COCs. Complete the log-in entry date and time once samples are logged in as described below.

7.4 Inventory the COCs for any "RUSH" or quick hold time analyses. Notify the appropriate section managers of these analyses. List any samples for analyses that have short hold times in the "Wet Chemistry Shorts and Rushes Logbook" (Figure 5) in the wet chemistry laboratory. Be sure to list the client, number of samples and date and time of the earliest sample. GC or GC/MS personnel must be informed when ENCORES are received so that they may be scheduled for extrusion. Microbiology personnel should also be informed of any microbiology samples that arrive. Parameters that routinely require short analytical hold times are:

Coliforms	Color	pH
Nitrate/Nitrite	Dissolved Oxygen	Turbidity
Ferrous iron	Orthophosphate	Hex. Chromium
MBAS	TBOD	Free CO_2
Sulfite	ENCORE soil samples	Settleable Solids
Odor	Residual Chlorine	CBOD

7.5 Inspect the condition of custody seals, cooler, ice condition and samples received. Note any non-intact conditions on the Sample Receipt Condition Report (SRCR - Figure 6). Notify the Katahdin project manager (PM) of any discrepancies or problems with sample receipt. The PM contacts the client as necessary. If breakage of a potentially hazardous sample is discovered, close and seal the packing container with all the samples inside and move to a hood in the organic extractions area or to the smaller hood in the login area if space permits. One of the three Katahdin Emergency Response Coordinators or the Katahdin Environmental Health & Safety Manager must be notified. Disposition of the broken and other possibly contaminated samples will be determined on a case-by-case basis in accordance with the laboratory's handling procedures for hazardous waste as outlined in the Katahdin Environmental Health & Safety Manual. Generally, when a sample has broken and has mixed with any ice in the cooler, that liquid will be poured off into 2 liter plastic containers and labeled as "do not use". These containers will be disposed of as soon as the disposition of the appropriate samples has been determined through analysis.

7.6 If there is no breakage of a potentially hazardous sample:

Check cooler temperatures using the IR thermometer assigned to the sample receipt area. If a cooler temperature blank is present, aim the IR gun at the temperature blank; otherwise aim the IR gun at any sample in the cooler if no temperature blank is present. Be sure that the IR gun is within 6 inches of the bottle and not aimed at a label on the bottle. Press the trigger on the handle and be sure the red dot is visible on the bottle surface. The IR gun has been set to read in degrees celcius. If checking the temperature of a plastic bottle, set the emissivity at 0.90. If checking the temperature of a glass bottle (either amber or clear), set the emissivity at 0.85. Refer to Figure 7 for manufacturer's instructions on changing the emissivity. Record the temperature on the Sample Receipt Condition Report. Receipt temperatures should be <6 °C, without freezing. Any temperature falling outside of this range must be noted on the SRCR and reported to the appropriate Katahdin project manager.

Note: Samples received for metals analysis only do not have to meet any temperature receipt requirements.

Note: A probe type thermometer is retained as back-up in case there is a problem with the IR thermometer.

7.7 Note the condition of the ice or ice packs. If the ice has melted and the temperature is out of acceptance criteria, note this on the SRCR. For samples that are hand delivered to the laboratory immediately after collection (i.e. sample collection times are <6 hours old), the temperature blank and/or cooler temperature will most likely not meet the acceptance criteria. The samples shall be considered acceptable if there is evidence that the chilling process has begun such as arrival on ice. Note this on the SRCR. If samples (that were just collected) have not arrived on ice, note this on the SRCR, and start the cooling process as soon as possible after arrival at the laboratory.

Note: All clients must be notified when samples are received that do not meet the appropriate temperature requirements. In these cases, certain regulatory requirements may not be met and may invalidate certain data.

7.8 Inventory the samples against the chain of custody (COC). If the COC is incomplete, the sample custodian must inform the appropriate Katahdin project manager (PM). The PM may make changes to correct or complete the COC, but all changes must be initialed and dated. Changes must be noted on the SRCR. Any discrepancies between the samples and the COC must also be noted on the SRCR.

7.9 Using the Sampling and Preservation Requirements Table (Table 1) as a reference, check if samples are in proper containers and received correct pretreatment (e.g., filtration, preservation) for the analyses requested. For aqueous parameters requiring preservation, check pH by inserting a clean capillary tube into the sample and dabbing the tube on wide range pH paper. If the pH is not clearly either less than 2 or greater than 12, the appropriate narrow range pH paper must be used. NOTE: The pH of volatile organic (VOA) samples is checked and recorded by the analyst after completion of analysis and not by sample receipt personnel. The used capillary tube is discarded and a new capillary tube is used for each sample.

Additional preservative is added to samples if the pH is not in the range specified in the Sampling and Preservation Requirements Table. No more than 10% of the original sample volume should be added as preservative. If the client has noted that the sample reacts violently (i.e., foams and bubbles) upon preservation, add no more preservative to the sample. Some clients may wish to be contacted if their samples are found to be improperly preserved. Record all preservation discrepancies on the Sample Receipt Condition Report including the lot number of the preservative added. If additional preservative is added, a sticker with the type of preservative must be placed on the sample container.

Note: Preservatives are obtained from the larger containers in the bottle preparation area.

Note: If samples are received unpreserved for 200.7 or 200.8 analysis, the samples must be preserved to pH <2 with nitric acid. Samples must be held for 16 hours after preservation before sample preparation can begin.

7.10 For samples requiring filtration as pretreatment (i.e. for dissolved metals), the work order/login numbers are recorded in the filtration logbook (see Figure 8). The samples are filtered by the Metals Group.

7.10.1 A 500 mL filter flask and filter funnel are acid rinsed three times in a 10% nitric acid bath, then three times with Laboratory Reagent Grade Water.

7.10.2 A vacuum pump is attached.

7.10.3 A 0.45 micron filter is rinsed three times with 5% nitric acid and three times with Laboratory Reagent Grade Water. The rinsate is discarded.

7.10.4 A sufficient sample aliquot is filtered and preserved with concentrated nitric acid to pH <2.

7.10.5 The bottles are labeled with the work order/login number and other sample information and stored at <6 ° C until the time of digestion.

7.11 Using the Sampling and Preservation Requirements Table (Table 1) as a reference, determine if sufficient volume of sample is present for analysis. Note discrepancies on the SRCR.

7.12 For drinking water samples, enter the appropriate information (work order, date, etc.) into the Measured Turbidity and Preservation of Incoming Samples Logbook. Inform the appropriate analyst of the sample. The turbidity must be measured prior to sample preparation. If the turbidity is <1 NTU, the sample does not have to be digested prior to metals analysis. If the turbidity is >1 NTU, the sample must be digested prior to metals analysis. The sample must be preserved after the turbidity measurement is taken. Record the appropriate information in the logbook (Figure 9).

7.13 Notify the PM immediately if there are any discrepancies or problems with sample receipt. The PM will contact the client for information and resolution as necessary. All decisions to proceed or not to proceed with analysis associated with samples received that do not meet specified acceptance criteria (i.e. cooler temperature, preservation, container, etc.) must be fully documented on the SRCR. Although this form is included with all client reports, additional narration or flagging of data may be necessary.

7.14 Review any additional paperwork that accompanies the sample(s) submitted for analysis along with laboratory-generated information. This includes shipping forms, letters, chain-of-custody forms, sample labels, Incoming Sample Information Sheets (ISIS), quotes, memos, etc. These forms may provide details on specific client

requests. The ISIS will provide information on specifics for log-in. Refer to Figure 10 for an example.

7.15 Resolve any questions or concerns raised by steps 7.1-7.14 by consulting the correspondence files or client services personnel or communicating directly with the client. Note in the notes section of the SRCR any deviations from normal sample handling or analytical procedures (e.g., client requests analysis although hold-time expired).

7.16 When non-environmental food samples are delivered to the laboratory, they are taken immediately to the food/microbiology laboratory and stored in the refrigerators there. A copy of the Chain-of-Custody is left with the analysts. The original paperwork is forwarded to sample log in where the job is logged into the KIMS system.

7.17 The following information is documented via the Katahdin Information Management System (KIMS) and a work order/login COC report (Figure 11) is generated for the samples received:

7.17.1 Log onto KIMS by entering employee ID under "Username", employee specific password under "Password" and KIMS under "Database".

7.17.2 Once logged onto KIMS select "Sample Management" and then "Login".

7.17.3 Select "New" and the next available Login ID number will automatically be entered. Select "OK" and the Sample Definition screen will open.

Note: If a Work Order number has already been opened, select "change" and type in the appropriate number to access the information.

7.17.4 In the Sample Definition Screen, enter the following information.

Client ID -	Enter client sample description.
ReceiveDate -	Enter in date that samples were received in the lab in the format YY-Month-DD.
CollectDate -	Enter in date that samples were collected in the format YY-Month-DDTIME.
TAT -	Enter TAT for hardcopy report.
DueDate -	Due date will automatically be calculated based on calendar days.
VerbalDate -	Manually type in verbal due date.

QuoteRef -	Enter quote number if applicable.
Project -	Enter project number if applicable.
Account -	Enter client specific account number.
Account Name -	Account name will automatically be entered.
Collected By -	Enter name/initials of sampler listed on COC. If unknown, enter "Client".
Locator -	May be used for client ID information when requested by the project manager.
Site -	Enter project site name.
Description -	May be used for long client Ids when requested by the project manager.
Discount -	No entry-not currently used.
Priority -	No entry-not currently used.
Fact. -	No entry-not currently used.
Expected -	No entry-not currently used.
Comments -	Enter MS/MSD, verbal due date and any sample irregularities if applicable.
OrderDate -	Current date is automatically entered.
Matrix -	Enter sample matrix code where

AQ = Aqueous	SLD = Food Solid
SL = Solid, Soil, Sludge	AR = Air
FP = Free Product	SWAB = Swab
WP = Wipe	SAL = Saline
NOAQ = NonAqueous	TIS = Tissue
DW = Drinking Water	

Product Code -	Enter analysis code per test requested on COC.
Type -	Product code type will automatically be entered where S = Stand alone

P = Parent
C = Children

Fact. - No entry-default is 1.

Price - This is left as is by sample log-in. During project management review of the work order, the prices are entered based on quotes or standard prices.

Cost - No entry needed.

Lev - No entry needed.

Type - Container type will automatically be entered.

Bot - Enter number of containers per test for printing of labels.

Login Info - Parameter Data Screen will open. Enter following information

KAS Proj. Manager-	Initials of Katahdin person overseeing the project.
Client PO#-	Client purchase order.
Project-	Project name.
Cooler Temperature-	Temperature blanks or cooler temps.
Delivery Services-	Method of delivery to the lab.
QC Level-	QC Level of report and regulatory agency (ie., IV NFESC).
SDG ID-	Sample Delivery Group ID if applicable.
SDG Status-	Begin, Continue or End.
Analysis Instructions-	PM will enter special instructions regarding project.
Report Instructions-	PM will enter special instructions regarding project.
Regulatory List-	Used for federal programs.
EDD Format-	Specific KAS EDD format.

Select "SAVE" and then "CANCEL".

Addresses - Select "Addresses" and the Address Links screen will open. The billing address is the default address of the

account. Enter the client account code under "Project/Account" and select the report to contact under "Address Type". Select the appropriate boxes for report, report CC and invoice CC. Select "SAVE" and then "CLOSE".

Create Containers - Select "Create Containers". Letters will be assigned to each sample number. Select "OK" until letters have been assigned to each sample number. To manually assign letters, Select "Enter Container IDs" and "OK". Enter sample numbers including letters and select "OK", "Close", "Yes" to save changes, "Cancel" and "Cancel".

7.17.5 To print the login report, select "Reports", "Login" and "Login COC". Enter login number under "Login Number". Select "OK", "Run Report" and then "Print".

7.18 To print labels unique to each bottle, select "Reports", "Login" and "Labels". Enter login number under "Login/Prelogin", select "Background (IDXL)" and select F9 on keyboard under "Select Sample Label". Select "OK" and then "Print". After labels print out select "Cancel".

Note: As stated in "create containers" above, each sample bottle is assigned a unique ID. The job is given a work order number. Each different client sample ID is given a numerical number following the work order number and each sample container with the same client ID is given a container ID using alphabetical letters. This series of work order, sample number and container ID is transcribed throughout the raw data for traceability purposes.

Example: One job containing one client sample with 3 different containers:

SC9001-001(a), SC9001-001(b), SC9001-001(c)

Example: One job containing two client samples with 2 different containers for each:

SC9002-001(a), SC9002-001(b), SC9002-002(a), SC9002-002(b)

7.19 Affix permanent sample number labels to sample containers, assuring that sample IDs on labels correspond to sample bottle IDs. Do not obscure client ID on the bottles.

7.20 Place samples in their designated storage locations and log them in, noting initials, date and time, work order/login and sample numbers, and storage location on the internal laboratory chain of custody form (Figure 12). Place form in the appropriate

binder in the log in area. Non-environmental food samples do not get an internal COC and are taken immediately to the food/microbiology lab for storage.

Storage location of the samples is determined by type of sample and/or type of analysis, as outlined below. Most samples are stored in the walk-in cooler, which is organized by test type and work order/login number.

Specific storage locations are described below.

7.20.1 Aqueous samples for wet chemistry (except hardness, see 7.19.4 below) - left aisle, both sides, as you enter walk-in cooler. TOC vials are to be stored in the trays designated for TOC samples.

7.20.2 Aqueous samples for organic extractions – right aisle, left side, as you enter walk-in cooler.

7.20.3 Non-aqueous samples (all analyses except volatile organics) - to the right and towards the back as you enter walk-in cooler. Non-aqueous samples for volatile organics are stored in "VOA Refrigerator 2" located in the Volatiles Laboratory.

7.20.4 Aqueous samples for metals and/or hardness analyses – right aisle, right side towards the front as you enter walk-in cooler.

7.20.5 Samples (aqueous and solid) for volatile organics analyses (VOA) – All aqueous samples and soil samples in VOA vials (preserved with methanol or sodium bisulfate) are stored in "VOA Refrigerator 1" in the Volatiles Laboratory. VOA soils in jars or ENCORE samplers are stored in "VOA Refrigerator 2" in the Volatiles Laboratory. VOA samples known or suspected to be hazardous (such that cross-contamination of other samples might occur) are placed in a "paint can" and stored in the walk-in.

7.20.6 Soil samples for volatile organics analyses (VOA) that are unpreserved or preserved with Laboratory Reagent Grade Water are stored in "VOA Freezer 1" in the volatiles laboratory.

Sample storage coolers are not locked, but internal chain-of-custody is documented with respect to native samples, extracts and digestates within the laboratory. The laboratory maintains a secure facility with respect to unauthorized personnel, as described in the current revision of Katahdin SOP, AD-004, Laboratory Facility Security and Confidentiality. All sample storage coolers are equipped with locks if specific project or regulatory requirements deem it necessary.

7.21 Sample Receipt gives the Work order/login COC report and confirmation of the job, as logged-in, to the appropriate Katahdin project manager. All chain-of-custody and

other receipt documentation must accompany the job. The project manager reviews the job for accuracy and completeness. Any unresolved issues should be resolved at this time. Any project or program specific forms should be included with the paperwork at this time. These forms may include CLP forms or state-specific forms. The project manager then dispatches the work order/login to the individual department worklists. The dispatched work order/login package is then filed in Data Management where the complete package will eventually be compiled.

7.22 The temperature of all sample storage refrigerators and freezers is recorded daily by assigned individuals. Notebooks containing a record of each refrigerator and freezer temperature history are used for this purpose and are maintained by the assigned individuals. Temperatures above or below the acceptance range are to be brought to the attention of a Department Manager, Operations Manager, or Quality Assurance Officer. Such an occurrence and the actions taken to correct it must be noted in the comments column of the temperature recording notebook next to the temperature measurement. (See Figure 13).

Additionally, temperatures of storage units are monitored continuously by wireless thermometers. A temperature is recorded electronically every 10 minutes. The QAO can generate a specified report as needed, including every reading or maximum/minimum temperatures for a given timeframe. These monitoring devices ensure continual compliance seven days per week. The data can be used to check for problems.

PROCEDURES FOR CHEMISTS

7.23 When removing a sample from its storage location, record on the laboratory internal chain-of-custody (from the appropriate department) the sample number, date and time it was removed, chemist who removed it, and the analysis to be conducted or reason for removal.

7.24 If the samples have not been logged in yet and they need to be pulled in order to analyze short holding time parameters, the analyst taking the sample must use the designated logbook (Immediate Internal COC – Figure 14) to sign the samples out. Many circumstances lead to analysts having to pull samples before they are logged into the KIMS system. It is everyone's responsibility to ensure that all samples can be accounted for at all times. Failure to do so can create confusion and bottle necks for others trying to access the samples. Samples that are pulled before log-in must be returned to the designated bin in the sample receipt area. When the logbook for Immediate Internal COC's is used, the standard internal COC's do not have to be signed at a later date. The Immediate Internal COC Logbook must always be consulted if there is ever a question about whether an internal COC has been completed.

7.25 If a sample is not consumed by an analysis, return the remaining sample to its assigned storage location and enter the date and time returned on the laboratory internal chain-of-custody record.

7.26 If analysis consumes the entire sample, indicate this on the laboratory internal chain-of-custody record.

7.27 After the completion of all analyses, the original "left over" sample containers will remain in sample storage until their final disposal. Samples are held during this period for the purposes of retesting if required by a laboratory corrective action or by a client. Refer to the current revision of Katahdin SOP, SD-903, Sample Disposal, for details on final disposal of samples.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Each thermometer used to monitor sample storage or cooler temperatures must be calibrated annually against a NIST traceable thermometer. The QAO is responsible for ensuring that the thermometer(s) are scheduled for annual calibration and for maintaining the calibration records. All other procedures and documentation listed in this SOP must be followed at all times.

9.0 METHOD PERFORMANCE

Not applicable.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Handbook for Analytical Quality Control in Water and Wastewater Laboratories," U.S. EPA EMSL Office of Research and Development, March 1979.

Code of Federal Regulations 40, Parts 136 and 141.

"Test Methods for Evaluating Solid Waste: Physical/Chemical Methods," SW-846 Chapters 1 & 2, USEPA, Third Edition, including Updates I, II, IIA, and IIB, III June, 1997.

Katahdin Analytical Services, Inc., Environmental Health & Safety Manual, current revision.

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TABLE 1

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – AQUEOUS MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GENERAL CHEMICAL ANALYSES					
Acidity	305.1	100 mL	P,G	1,2	14 days
Alkalinity-Manual Titrimetric	310.1	100 mL	P,G	1,2	14 days
Ammonia-Nitrogen with distill-Auto. Phenate	350.1	1 L	P,G	1,3	28 days
Ammonia-Nitrogen-Automated Phenate	350.1, 350.2	250 mL	P,G	1,3	28 days
Anions (Cl, Br, SO4, NO2, NO3)	300.0	250 mL	P, G	1	48hr/28days
Bicarbonate, Carbonate (see pH & alkalinity)	calc.				
Biochemical Oxygen Demand-Carbonaceous	405.1	1 L	P,G	1	48 hours
Biochemical Oxygen Demand-Total	405.1	1 L	P,G	1	48 hours
Bromide	320.1	500 mL	P,G	1	28 days
Chemical Oxygen Demand-Manual Colorimetric	410.4	100 mL	P,G	1,3	28 days
Chloride-Automated Ferricyanide	325.2	100 mL	P,G	1	28 days
Chlorine, Residual	SM4500-Cl G	100 mL	P,G	1,9	ASAP
Chromium, Hexavalent	SM3500Cr D / SW7196	200 mL	P,G	1,9	24 hours
Color, Apparent	110.2	100 mL	P,G	1,2	48 hours
Cyanide, Amenable-Spectrophotometric	335.1	250 mL	P,G	1,5	14 days
Cyanide, Total-Spectrophotometric	SM4500CN C 335.3, 335.4	250 mL	P,G	1,5	14 days
Dissolved Oxygen(Lab)-Membrane Electrode	360.1	500 mL	G	1	ASAP
Ferrous Iron - Colorimetric	SM3500-Fe D	250mL	P	1	24 hrs
Fluoride with distillation, Potentiometric ISE	SM4500F C/340.2	500 mL	P only	1	28 days
Fluoride, Potentiometric ISE	340.2	200 mL	P only	1	28 days
Free CO_2	SM4500-CO_2 C	250mL	P	1	24 hrs.
Hardness, Total-Manual Titrimetric	130.2,SM2340C	250 mL	P,G	4	6 months
MBAS, Extraction-Colorimetric	SM5540C	1 L	P,G	1	48 hours
Nitrate+Nitrite-Automated Cadmium Reduction	353.2	100 mL	P,G	1,3	28 days
Nitrate-Automated Cadmium Red./Diazotization	353.2	100 mL	P,G	1	48 hours
Nitrite-Automated Diazotization	353.2	100 mL	P,G	1	48 hours
Oil & Grease-Total Recoverable, Gravimetric N-Hexane extractable material N-Hexane extractable material w/ silica gel cleanup	1664	(2) 1 L	glass only	1,11	28 days
pH (Laboratory)	150.1	100 mL	P,G	1,2	24 hours
Phenolics, Total Recoverable-Manual 4AAP	420.1	1000 mL	glass only	1,3	28 days
Phosphate, Ortho- Ascorbic Acid	365.2	100 mL	P,G	1	48 hours
Phosphate,Total	365.4	100 mL	P,G	1,3	28 days
Solids-Filterable Residue (TDS),Gravimetric180	160.1	250 mL	P,G	1	7 days
Solids-Nonfilterable Residue (TSS)	160.2	500 mL	P,G	1	7 days
Solids-Settleable Solids (SS)	160.5	1 L	P,G	1	48 hours

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – AQUEOUS MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GENERAL CHEMICAL ANALYSES					
Solids-Total Solids	160.3	250 mL	P,G	1	7 days
Solids-Total Volatile (TVS)	160.4	250mL	P,G	1	7 days
Solids-Volatile Filterable Residue (VDS)	160.1/160.4	250 mL	P,G	1	7 days
Solids-Volatile Nonfilterable Residue (VSS)	SM 2540 F	500 mL	P,G	1	7 days
Specific Conductance-Wheatstone Bridge	120.1	100 mL	P,G	1,2	28 days
Sulfate-Turbidimetric	375.4	100 mL	P,G	1	28 days
Sulfide-Iodometric	376.1	500 mL	P,G	1,7	7 days
Sulfite-Titrimetric	377.1	500 mL	P,G	1,9	ASAP
Tannin/Lignin-Colorimetric	SM 5550 B	100 mL	P,G	1	7 days
TKN-Auto Block Digest, Spect.	351.2	100 mL	P,G	1,3	28 days
Total Inorganic Carbon	415.1	(2) 40 mL	VOA vial	1	28 days
Total Inorganic Carbon if with TOC	415.1	(2) 40 mL	VOA vial	1	28 days
Total Organic Carbon-Oxidation	415.1	(2) 40 mL	VOA vial	1,3	28 days
Total Organic Halogen	9020	500 mL	Amber Glass	1,3	28 days
Turbidity	180.1	100 mL	P,G	1	48 hours
ELEMENTAL ANALYSES					
Chromium, Hexavalent	7196/6010	500 mL	P,G	1,9	24 hrs
GFAA(Furnace) Elements	SM 3113/ 200 series	500 mL	P,G	4	6 months
ICP Elements	200.7/6010	500 mL	P,G	4	6 months
ICP MS Elements	200.8/6020	500 mL	P,G	4	6 months
Low Level Mercury	1631	500 mL	G	NA	90 days
Mercury	245.1/7470	500 mL	P,G	4	28 days
GC ORGANIC ANALYSES					
BTEX & MTBE	602 & 8021	(2) 40 mL	VOA vial	1,8,9	14 days(~)
EDB, DBCP & 1,2,3-TCP	504.1	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Extractable Petroleum Hydrocarbons	MADEP/EPH	(2) 1000 mL	Amber Glass	12	14days/40days
Formaldehyde	556	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Fuel Oil in Water	8015Modified	(2) 1000 mL	Amber Glass	1,8	7days/40days
Fuel Oil in Water	ME HETL 4.1.25	(2) 1000 mL	Amber Glass	1,8	7days/40days
Gasoline in Water	8015Modified	(2) 40 mL	VOA vial	1,8	14 days
Gasoline in Water	ME HETL 4.2.17	(2) 40 mL	VOA vial	1,8	14 days
Glycols	8015Modified	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Herbicides	8151	(2) 1000 mL	Amber Glass	1	7days/ 40days
Methane, Ethane & ethene	RSK 175	(2) 40 mL	VOA vial	1,8,9	14 days(~)
PCB's (& Congeners)	608 & 8082	(2) 1000 mL	Amber Glass	1	7days/40days

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – AQUEOUS MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GC ORGANIC ANALYSES					
Pesticides	608 & 8081	(2) 1000 mL	Amber Glass	1	7days/40days
Pesticides and PCB's	608 & 8081/8082	(2) 1000 mL	Amber Glass	1	7days/40days
Purgeable Aromatics	602 & 8021	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Purgeable Halocarbons	601 & 8021	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Purgeables, Total	601 & 602	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Purgeables, Total	8021	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Solvents (Direct Injection)	8015M	(2) 40 mL	VOA vial	1	14 days
Volatile Petroleum Hydrocarbons	MADEP/VPH	(2) 40 mL	VOA vial	11	14days
GC/MS ORGANIC ANALYSES					
Acid Extractables-Priority Pollutants	625	(2) 1000 mL	Amber Glass	1	7days/40days
Acid Extractables-TCL	8270	(2) 1000 mL	Amber Glass	1	7days/40days
Base Neutral Extract.-Priority Pollutants	625	(2) 1000 mL	Amber Glass	1	7days/40days
Base Neutral Extractables-TCL	8270	(2) 1000 mL	Amber Glass	1	7days/40days
Drinking Water Volatiles - Low Level	524.2	(3) 40 mL	VOA vial	1,8,9,10	14 days(~)
PCB Homologues	680	(2) 1000 mL	Amber Glass	1	7days/40days
Polyaromatic Hydrocarbons	8270/8270 SIM	(2) 1000 mL	Amber Glass	1	7days/40days
Semivolatile Extractables-Priority Pollutants	625	(2) 1000 mL	Amber Glass	1	7days/40days
Semivolatile Extractables-TCL	8270	(2) 1000 mL	Amber Glass	1	7days/40days
Volatile Organics	8260	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Volatile Organics-Priority Pollutants	624	(2) 40 mL	VOA vial	1,8,9	14 days(~)
HPLC ANALYSES					
HPLC-Explosives	8330, 8332	(2) 1000 mL	Amber Glass	1	7days/40days
MICROBIOLOGICAL ANALYSES					
Coliform, Fecal	SM 9222D, SM 9213D Mod.	100 mL	P,G	1,6	6 hours
Coliform, Total	SM 9222B	100 mL	P,G	1,6	30 hours
Coliform and E-coli, Total	SM9223B/Colitag	100 mL	P,G	1,6	30 hours
E-coli	SM9213D, Colilert/Quantitray	100 mL	P,G	1,6	6 hours
Heterotrophic Plate Count	SM9215B SIMPLATE	100 mL	P,G	1,6	30 hours

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – SOLID MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GENERAL CHEMICAL ANALYSES		**4 oz=100 g**			
% Carbon	9060 mod.	4 oz	Soil Jar	1	28 days
Ammonia-Nitrogen-Automated Phenate	350.1 mod.	4 oz	Soil Jar	1	28 days (^)
Anions	9056	4 oz	Soil Jar	1	48hrs to 28 days from slurry (^)
Cation Exchange Capacity	9081	4 oz	Soil Jar	1	14days/7days (^)
Chloride-Automated Ferricyanide	9251/300.0	4 oz	Soil Jar	1	28days from slurry (^)
Cyanide, Amenable-Spectrophotometric	9012	4 oz	Soil Jar	1	14 days
Cyanide, Total-Spectrophotometric	9012	4 oz	Soil Jar	1	14 days
Fluoride, Potentiometric ISE	300.0 mod./340.2	4 oz	Soil Jar	1	28 days (^)
Lime Equivalency	310.1 mod.	4 oz	Soil Jar	1	28 days (^)
Nitrate+Nitrite-Automated Cadmium Reduction	300.0 mod./353.2	4 oz	Soil Jar	1	28 days (^)
Nitrate-Automated Cadmium Red./Diazotization	300.0 mod./353.2	4 oz	Soil Jar	1	48 hrs from slurry (^)
Nitrite-Automated Diazotization	300.0 mod./353.2	4 oz	Soil Jar	1	48 hrs from slurry (^)
Oil & Grease-Total Recoverable, Gravimetric N-Hexane extractable material N-Hexane extractable material w/ silica gel cleanup	9071	4 oz	Soil Jar	1	28 days (^)
Organic Nitrogen-Auto. Block Digest.,Spectro.	350.1/351.2 mod.	4 oz	Soil Jar	1	28 days (^)
pH (Laboratory)	9045	4 oz	Soil Jar	1	24 hours (^)
Phenolics, Total Recoverable-Manual 4AAP	Mod. 9065	4 oz	Soil Jar	1	28 days (^)
Phosphate, Ortho- Ascorbic Acid	300.0 mod./365.2	4 oz	Soil Jar	1	48 hrs from slurry (^)
Phosphate,Tot.-Auto Ascorbic Acid/Block Dig.	Mod. 365.4	4 oz	Soil Jar	1	28 days (^)
Solids-Ash	SM 2540 F	4 oz	Soil Jar	1	28 days (^)
Solids-Total Solids	CLP-CIP	4 oz	Soil Jar	1	28 days (^)
Solids-Volatile Solids	SM 2540 F	4 oz	Soil Jar	1	28 days (^)
Specific Conductance-Wheatstone Bridge	Mod. 9050	4 oz	Soil Jar	1	28 days (^)
Sulfate-Turbidimetric	9036/9038	4 oz	Soil Jar	1	28 days from slurry (^)
Sulfide-Iodometric	9030	4 oz	Soil Jar	1	7days from slurry (^)
Sulfide-Monier-Williams	40CFR-425	4 oz	Soil Jar	1	28 days (^)
Sulfite-Titrimetric	ASTM D3987/377.1 mod.	4 oz	Soil Jar	1	24 hrs from slurry (^)
TKN-Auto Block Digest,Spectro.	351.2 mod.	4 oz	Soil Jar	1	28 days (^)
Total Organic Halogen	9020/9021	4 oz	Soil Jar	1	28 days (^)
Total Petroleum Hydrocarbons-Extraction, IR	9071	4 oz	Soil Jar	1	28 days (^)
ELEMENTAL ANALYSES					
ICP Elements	6010	4 oz	Soil Jar	1	6 months
ICP MS ELements	6020	4 oz	Soil Jar	1	6 months
GFAA(Furnace) Elements	7000series	4 oz	Soil Jar	1	6 months
Mercury	7471	4 oz	Soil Jar	1	28 days

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – SOLID MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
ELEMENTAL ANALYSES **(cont.)**		**4 oz=100 g**			
Chromium, Hexavalent	3060/7196	4 oz	Soil Jar	1	30dys/24hrs
GC ORGANIC ANALYSES					
BTEX & MTBE	8021	(2) 40 mL	VOA Vial	1	14 days
Explosives - HPLC	8330, 8332	4 oz	Soil Jar	1	14days/40days
Extractable Petroleum Hydrocarbons	MADEP/EPH	4 oz	Soil Jar	1	7days/40days
Fuel Oil	ME HETL 4.1.25	4 oz	Soil Jar	1	14days/40days
Fule Oil	8015 mod.	4 oz	Soil Jar	1	14days/40days
Gasoline	ME HETL 4.2.17	(2) 40 mL	VOA Vial	1	14 days
Gasoline	8015 mod.	(2) 40 mL	VOA Vial	1	14 days
Herbicides	8151	4 oz	Soil Jar	1	14days/40days
PCB's (& Congeners)	8082	4 oz	Soil Jar	1	14days/40days
PCB's in Oil	8082	4 oz	VOA Vial	1	40 days
Pesticides	8081	4 oz	Soil Jar	1	14days/40days
Pesticides and PCB's	8081/8082	4 oz	Soil Jar	1	14days/40days
Purgeable Aromatics	8021	(2) 40 mL	VOA Vial	1	14 days
Purgeable Halocarbons	8021	(2) 40 mL	VOA Vial	1	14 days
Purgeables, Total	8021	(2) 40 mL	VOA Vial	1	14 days
Solvents (Direct Injection)	8015M	(2) 40 mL	VOA Vial	1	14 days
Volatile Petroleum Hydrocarbons	MADEP/VPH	(2)40 mL	VOA vial	13	28days
HPLC ANALYSES					
HPLC-Explosives	8330, 8332	4 oz	Soil Jar	1	7days/40days
GC/MS ANALYSES					
Acid Extractables-Priority Pollutants	8270	4 oz	Soil Jar	1	14 days/40 days
Acid Extractables-TCL	8270	4 oz	Soil Jar	1	14 days/40 days
Base Neutral Extractables-Priority Pollutants	8270	4 oz	Soil Jar	1	14 days/40 days
Base Neutral Extractables-TCL	8270	4 oz	Soil Jar	1	14 days/40 days
Polyaromatic Hydrocarbons	8270/8270SIM	4 oz	Soil Jar	1	14 days/40 days
Semivolatile Extractables-Priority Pollutants	8270	4 oz	Soil Jar	1	14 days/40 days
Semivolatile Extractables-TCL	8270	4 oz	Soil Jar	1	14 days/40 days
Volatile Organics – High Soil (>200 ug/kg)	5035/8260	Please refer to Table 6-2	Encore or similar sampler or VOA Vial or soil jar	14	Extruded w/in 48 hrs. Analyzed w/in 14 days
Volatile Organics – Low Soil (<200 ug/kg)	5035/8260	Please refer to Table 6-2	Encore or similar sampler or VOA Vial	14 or 15	Extruded w/in 48 hrs. Analyzed w/in 14 days
Volatile Organics-Priority Pollutants	8260	(2) 40 mL	VOA Vial	1	14 days
Volatile Organics-TCL	8260	(2) 40 mL	VOA Vial	1	14 days

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – SOLID MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
RCRA - HAZARDOUS WASTE CHARAC.					
Corrosivity-pH	9045	4 oz	Soil Jar	1	24 hours (^)
Ignitability-Flash Point (closed cup)	1010	4 oz	Soil Jar	1	14 days (^)
Reactivity-Reactive Cyanide	7.3.3.2	4 oz	Soil Jar	1	14 days
Reactivity-Reactive Sulfide	7.3.4.1	4 oz	Soil Jar	1	7 days
TCLP					
TCLP Extraction-Volatile Organics	1311	100 g	Soil Jar	1	14 days
TCLP Extraction-Semivolatiles	1311	200 g	Soil Jar	1	14 days
TCLP Extraction-Pesticides & Herbicides	1311	400 g	Soil Jar	1	14 days
TCLP Extraction-Metals	1311	200 g	Soil Jar	1	28 days
TCLP Analysis-Volatile Organics	8260	see above	Soil Jar	1	14 days
TCLP Analysis-Metals	6010/6020	see above	Soil Jar	1	180 days
TCLP Analysis-Mercury	7470	see above	Soil Jar	1	28 days
TCLP Analysis-Semivolatiles	8270	see above	Soil Jar	1	7 days/40 days
TCLP Analysis-Pesticides	8081	see above	Soil Jar	1	7 days/40 days
TCLP Analysis-Herbicides	8151	see above	Soil Jar	1	7 days/40 days

METHODS OF PRESERVATION
1 = Cool at 4 Degrees Celsius
2 = Settled
3 = H_2SO_4 to pH<2
4 = HNO_3 to pH<2
5 = NaOH to pH>12
6 = 1 mL 0.1M $Na_2S_2O_3$ or 1 10 mg pellet
7 = 1 m/L 2NZnAc/L & NaOH
8 = 2 drops 1:1 HCl
9 = No headspace
10 = $Na_2S_2O_3$, if chlorinated
11 = HCl to pH < 2
12 = 5 mL of HCL
13 = 15 mL of methanol
14 = methanol
15 = sodium bisulfate

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 1

EXAMPLE OF STANDARD KATAHDIN CHAIN-OF-CUSTODY FORM

Katahdin ANALYTICAL SERVICES
600 Technology Way
Scarborough, ME 04074
Tel: (207) 874-2400
Fax: (207) 775-4029

CHAIN of CUSTODY

PLEASE BEAR DOWN AND PRINT LEGIBLY IN PEN

Page ____ of ____

Client | Contact | Phone # ( ) | Fax # ( )

Address | City | State | Zip Code

Purchase Order # | Proj. Name / No. | Katahdin Quote #

Bill (if different than above) | Address

Sampler (Print / Sign) | Copies To:

LAB USE ONLY | WORK ORDER #:
KATAHDIN PROJECT NUMBER _____

REMARKS: _____

SHIPPING INFO: ☐ FED EX ☐ UPS ☐ CLIENT

AIRBILL NO: _____

TEMP°C _____ ☐ TEMP BLANK ☐ INTACT ☐ NOT INTACT

ANALYSIS AND CONTAINER TYPE PRESERVATIVES

#	Sample Description	Date / Time coll'd	Matrix	No. of Cntrs.	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N
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COMMENTS

Relinquished By: (Signature)	Date / Time	Received By: (Signature)	Relinquished By: (Signature)	Date / Time	Received By: (Signature)
Relinquished By: (Signature)	Date / Time	Received By: (Signature)	Relinquished By: (Signature)	Date / Time	Received By: (Signature)

THE TERMS AND CONDITIONS ON THE REVERSE SIDE HEREOF SHALL GOVERN SERVICES, EXCEPT WHEN A SIGNED CONTRACTUAL AGREEMENT EXISTS

ORIGINAL

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 2

EXAMPLE OF HOMEOWNER KATAHDIN CHAIN-OF-CUSTODY FORM

Katahdin
ANALYTICAL SERVICES

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

Homeowner Chain of Custody

Client: Contact: Phone: Fax:

Address: City: State: Zip:

Purchase Order #: Project Name/No.: E-mail:

Billing Address (if different):

Sampler (Print/Sign): Copies To:

***Test results are for compliance and will be reported to the state (see statement below). yes no Compliance samples must be received on ice.

Lab Use Only | Work Order #: KAS Project Manager:

Shipping: UPS Fed-Ex Mail Drop-Off

Sample(s) Received on Ice? Yes No Temperature if Iced:

Requested Services

Sample Description (Sample Identification and/or Lot #)	Date Collected	Time Collected	No. of Cntrs.	Standard Homeowner	Arsenic	Total Coliforms	Lead	Safety Test – coliform & N+N	FHA/MSH	Fluoride	Uranium		What's Included in the Standard Test and the FHA/MSH Test.
													Standard Homeowner Total Coliform/e-coli Nitrate, Nitrite Chloride, pH Hardness Copper, Iron Manganese Sodium
													FHA/MSH Standard plus Lead Turbidity Color Odor

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

Per the National Environmental Laboratory Accreditation Program (NELAP) Standards, Katahdin is required to accept samples that have been properly preserved. All sample containers provided to you have been properly preserved, but the proper preservation also requires samples to be received at <6 degrees celcius. The Safe Drinking Water Act regulations only require this for compliance samples (i.e., results that are submitted to the state). By circling no for compliance (above), you acknowledge that the samples described above are not for compliance purposes, and thus may not meet the temperature receipt requirements.

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 3

EXAMPLE OF FOOD/MICROBIOLOGY KATAHDIN CHAIN-OF-CUSTODY FORM

Katahdin
ANALYTICAL SERVICES

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

Chain of Custody

Client: Contact: Phone: Fax:

Address: City: State: Zip:

Purchase Order #: Project Name/No.: E-mail:

Billing Address (if different):

Sampler (Print/Sign): Copies To:

Lab Use Only | Work Order #: KAS Project Manager:

Shipping: UPS Fed-Ex Airbill No.:

Temperature:

Food & Microbiological Services

Sample Description (Sample Identification and/or Lot #)	Date/Time Collected	Matrix	No. of Cntrs.	Plate Count (A/H/S)	Listeria	Yeast and Mold	Salmonella	E-Coli	E-Coli 0157:H7	Staph	Vibrio	Total Coliforms	Campylobacter	Shelf Life	Challenge Study		

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

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 4

EXAMPLE OF KATAHDIN SAMPLE RECEIPT LOGBBOK

KATAHDIN ANALYTICAL SERVICES, INC.

SAMPLE LOG IN

Date Received	Time Received	Date Logged In	Time Logged In	Work Order	Client	Initials
				SA 0094		
				SA 0095		
				SA 0096		
				SA 0097		
				SA 0098		
				SA 0099		
				SA 0100		
				SA 0101		
				SA 0102		
				SA 0103		
				SA 0104		
				SA 0105		
				SA 0106		
				SA 0107		
				SA 0108		
				SA 0109		
				SA 0110		
				SA 0111		
				SA 0112		
				SA 0113		
				SA 0114		
				SA 0115		
				SA 0116		
				SA 0117		
				SA 0118		
				SA 0119		
				SA 0120		
				SA 0121		
				SA 0122		
				SA 0123		
				SA 0124		

Signed By:_____ Date:_____

Reviewed By:_____ Date:_____

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 5

EXAMPLE OF WET CHEMISTRY SHORTS AND RUSHES LOGBOOK

WET CHEMISTRY SHORTS & RUSHES **Receipt Date:**

HOLDING TIME					Immediate			24 Hr		48 Hr								
Work Order / Client	Matrix	Earliest Sampling Date	Earliest Sampling Time	Rush Parameters	pH	DO	Sulfite	Fe +2	Cr+6	Total BOD	Carbon. BOD	Color	Nitrate	Nitrite	OPO4	Set. Solids	Turbidity	Comments (Quick TAT, MS/MSD, etc.)

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 6

EXAMPLE OF SAMPLE RECEIPT CONDITION REPORT FORM

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

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

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

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

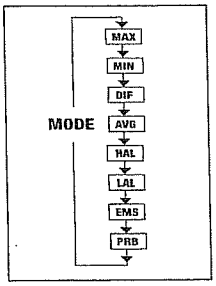
TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

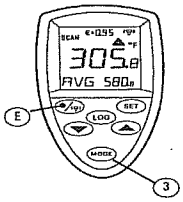
FIGURE 7

IR THERMOMETER MANUFACTURER'S INSTRUCTIONS FOR CHANGING EMISSIVITY

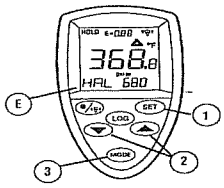
MODE Button Functions
Your infrared thermometer measures Maximum (MAX), Minimum (MIN), Differential (DIF)*, and Average (AVG)** temperatures each time you take a reading. This data is stored and can be recalled with the MODE button (3) until a new measurement is taken. (See "Hold and Recall" for information on how to recall stored data.) When the trigger is pulled again, the unit will begin measuring in the last mode selected. Pressing the MODE button also allows you to access the High Alarm (HAL), Low Alarm (LAL), Emissivity (EMS), Probe temperature (PRB—only available when the probe is connected), and Data logger (LOG). Each time you press MODE, you advance through the mode cycle. The diagram shows the sequence of functions in the Mode cycle.
Note: PRB (probe) is only available in the MODE loop when the contact probe is connected to the unit.

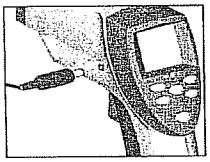
*DIF shows the difference between the maximum and minimum temperatures measured.
**AVG shows the average temperature reading for each time the trigger is pulled or the unit is locked on.

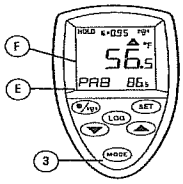




Selecting a Function
To Select the MAX, MIN, DIF, or AVG mode, pull the trigger. While holding the trigger, press the MODE button (3) until the appropriate code appears in the lower left corner of the display (E). Each time you press MODE, you advance through the MODE cycle. The MODE cycle is shown above.







Setting the High Alarm, Low Alarm, and Emissivity
To set values for the High Alarm (HAL), Low Alarm (LAL), and Emissivity, pull the trigger or press the MODE button (3) to activate the display. Press the MODE button until the appropriate code appears in the lower left corner of the display (E). Use the up and down keys (2) to adjust the desired values. To activate the alarms, press SET (1). To deactivate the alarms, press SET again.

Using a Probe (PRB)
Connect the probe to the input on the side of the unit (as shown).
PRB automatically appears in the lower left corner of the display (E, below). The probe temperature is shown in the lower right part of the display. The current infrared temperature continues to show in the center of the display (F). While the probe is connected, you may still cycle through the mode functions by pressing MODE (3).

Note: PRB is only available in the MODE loop when a probe is connected to the unit; the probe temperature will not activate the high alarm or low alarm.

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 8

EXAMPLE OF KATAHDIN SAMPLE FILTRATION LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.
Sample Filtration Logbook

Katahdin Sample No.	Site ID	Filtration Requested By:		Filtered and Preserved By:		
List Individually	(Optional)	Initials	Date	Initials	Date	Time

Reviewed and Approved by: _____ Date: _____

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 9

MEASURED TURBIDITY AND PRESERVATION OF INCOMING SAMPLES LOGBOOK

KATAHDIN ANALYTICAL SERVICES

Measured Turbidity and Preservation of Incoming Samples

KAS Lab Sample ID	Measured Turbidity (NTU)	Turbidity Date	Turbidity Analyst	Preservation Date	Preservation Time	Preservation Analyst
WW6757-9A	0.22	12-19-06		12/14/06	~~1830~~ 1400	DM
↓ -10A	0.18	↓		↓	↓	↓
WW6758-1A	4.80	↓		↓	↓	↓
WW6776-1A	0.68	12-20-06		12/20/06	1000	DM
WW6855-1A	0.92	12-26-06		12/26/06	1600	DM
WW6860-1A	2.40	↓		↓	↓	↓
SA0027-1A	0.52	1-3-07		01/03/07	1445	DM
SA0028-1A	0.72	1-3-07		↓	↓	↓
SA0087-1A	0.26	1-5-07		01/05/07	1645	DM
SA0088-1A	10.4	1-5-07		↓	↓	↓
SA0110-1A	0.47	1-8-07		01/08/07	1430	DM
SA0109-1A	0.32	1-8-07		↓	↓	↓
SA0138-1A	0.12	1-09-07		01/09/07	1700	DM
SA0262-1A	0.14	1-18-07		01/18/07	0930	DM
SA0263-1A	0.33	1-18-07		↓	↓	↓
SA0267-1A	0.30	1-18-07		01/18/07	0950	DM
SA0313-1A	0.58	1-22-07		01/22/07	1030	↓
SA0316-1A	1.70	1-22-07		01/22/07	1345	DM

REVIEWED BY: DATE:

FIGURE 10

EXAMPLE OF LABORATORY INCOMING SAMPLE INFORMATION SHEET (ISIS)

KATAHDIN ANALYTICAL SERVICES, INC. – INCOMING SAMPLE INFORMATION SHEET

CLIENT:	ATTENTION:
ADDRESS:	
TELEPHONE:	FAX:
OTHER CLIENT CONTACTS:	
REPORT CC:	PM/SALES CONTACT:
PO #:	CLIENT ACCOUNT:
KAS QUOTE #:	KIMS QUOTE #:
PROJECT NAME:	KIMS PROJECT REF.:
SDG:	APPLICABLE CERTIFICATION:
RECEIPT DATE:	MEANS OF DELIVERY:

METHOD OR KIMS PRODUCT	MATRIX	# OF SAMPLES	SPECIAL INSTRUCTIONS (I.E LIMITS, PREPS)

SUBCONTRACT PARAMETER	SUBCONTRACT LABORATORY

QC LEVEL:	EDD:
RUSH/VERBAL TAT:	HARDCOPY TAT:
HISTORY:	
OTHER:	

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 11

EXAMPLE OF KATAHDIN WORK ORDER/LOGIN COC REPORT

Katahdin
ANALYTICAL SERVICES

Katahdin Analytical Services
Login Chain of Custody Report (lno1)
Jan. 26, 2007
03:51 PM

Page: 1 of 1

Login Number: SA0395
Account:KATAHD001 Web
Katahdin Analytical Services

Project:

Primary Report Address:
Leslie Dimond
Katahdin Analytical Services
600 Technology Way
P.O. Box 540
Scarborough,ME 04070

Primary Invoice Address:
Accounts Payable
Katahdin Analytical Services
600 Technology Way
P.O. Box 540
Scarborough,ME 04070

Report CC Addresses:
Invoice CC Addresses:

Login Information

ANALYSIS INSTRUCTIONS	:
CHECK NO.	:
CLIENT PO#	:
COOLER TEMPERATURE	: n/a
DELIVERY SERVICES	: In House
EDD FORMAT	:
MAIL DATE	:
PM	: LAD
PROJECT NAME	: QC Holding Blanks
QC LEVEL	: I
REGULATORY LIST	:
REPORT INSTRUCTIONS	:
SDG ID	:
SDG STATUS	:

Laboratory Sample ID	Client Sample Number	Collect Date/Time	Receive Date	PR	Verbal Date	Due Date	Comments
SA0395-1	WHITE FRIDGE	26-JAN-07 15:50	26-JAN-07			08-FEB-07	
Matrix	*Product*	*Hold Date (shortest)*	*Bottle Type*		*Bottle Count*		
Aqueous	S SW8260FULL5ML	09-FEB-07			2		
SA0395-2	BLUE FRIDGE	26-JAN-07 15:50	26-JAN-07			08-FEB-07	
Matrix	*Product*	*Hold Date (shortest)*	*Bottle Type*		*Bottle Count*		
Aqueous	S SW8260FULL5ML	09-FEB-07			2		

Total Samples: 2 ***Total Analyses:*** 2

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 12

EXAMPLE OF LABORATORY INTERNAL CHAIN-OF-CUSTODY FORM

Katahdin Analytical Services, Inc.
Internal Custody Record

EXTRACTIONS (AQ) Work Order #:_____

Sample Numbers Involved	Placed in refrigeration by: Initials
	Date
	Time

Sample Numbers	Analysis	Removed			Returned			Consumed?	
		Initials	Date	Time	Initials	Date	Time	Yes	No

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 13

EXAMPLE OF REFRIGERATOR TEMPERATURE LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.

SAMPLE RECEIPT WALK-IN TEMPERATURE LOG

Corrective Action: Note in the "comments" column and notify the QAO or supervisor; document corrective actions taken and return to control.

Thermometer Location		Sample Receipt Walk-in 1	Comments
Acceptance Criteria		2 to 6 °C	
Date	Initials	Temp (°C)	
02/2/07	NDA	3.7	
02/05/07	DWM	5.8	
02/06/07	DWM	5.8	
02/07/07	DWM	5.8	
02/08/07	DWM	3.8	
02/09/07	DWM	3.5	
02/12/07	DWM	3.5	
02/13/07	DWM	3.5	

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 14

EXAMPLE OF IMMEDIATE INTERNAL COC LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.
INTERNAL CUSTODY RECORD FOR IMMEDIATES

CLIENT	PROJECT	CLIENT ID &/or WORK ORDER #	ANALYSIS	OUT date/time	IN date/time	INIT	Consumed?
Jacobs		WW4813-1A, -2A	ICP	9/13/06 0930 →	0935	DJJ	yes no
Jacobs		WW4883-1A	ICP	9/16/06 0900 →	1000	DJJ	yes no
CES		WW4965	BOD	9/20/06 0900	9/20/06 1000	CP	yes no
CCAR		WW4969	BOD	9/20/06 1000	↓	CP	yes no
GEMF		WW4970	BOD	↓	↓	CP	yes no
Jacobs		WW4962-1A, -2A	ICP	9/20/06 0900 →	1000	DJJ	yes no
Irving		WW4994	BOD	9/21/06 1000	9/21/06 1015	CP	yes no
Highliner		WW4992 ②	BOD	9/21/06 1015		CP	yes no
NATIONAL		WW5000	TS, PERCHLORE PH, SP.COND	9/21/06 1100	9/21/06 1257	↓	yes no
W&C		WW5001	BOD	9/21/06 1300		CP	yes no
Arlens		WW5016	NO3	9/22/06 1300 NO3	09/22/06 1100	KR	yes no
RAISOM		WW5010	↓	↓ ↓	↓	↓	yes no
EcoMaine		WW5029	BOD	9/22/06 1100		CP	yes no

QAQC143

0000095

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: SD-903
Revision History
Cover Page
Page 1

TITLE: **SAMPLE DISPOSAL**

Prepared By: _____ Date: 2/01

Approved By:

Group Supervisor: _____ Date: _____

Operations Manager: Jal C. Burton Date: 2/01

QA Officer: Deborah J. Nadeau Date: 2.01

General Manager: _____ Date: 2/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Format changes, ~~added pollution prevention~~, added updated log-book and greater detail on dis-posal.	DN	2.01	2/01
02	Major rewrite to include more detail on hazardous waste regu-lations + to reflect current practices.	DN	02/05	02/05
03	Rewrite of section 7 to comply with current practices in new facility. Updated Figures 1 to 3.	DN	02.08	02.08
04	Added elementary neutralization to section 7.0. Other minor edits.	DN	05.09	0509

TITLE: SAMPLE DISPOSAL

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SD-903-04**, titled **SAMPLE DISPOSAL**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SD-903-04**, titled **SAMPLE DISPOSAL**.

Recipient: _____ Date: _____

TITLE: SAMPLE DISPOSAL

1.0 SCOPE AND APPLICATION

Katahdin Analytical Services, Inc. requires strict adherence to specific procedures for the disposal of samples. The procedures are designed to categorize waste materials, provide for their safe and timely disposal and to ensure compliance with local and federal regulations pertaining to disposal of chemicals and environmental samples. Any other means of disposal not described in this SOP is prohibited without consent from the Katahdin Environmental Health & Safety Officer and/or the Katahdin Environmental Compliance Officer.

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical personnel for the disposal of samples. These procedures apply to the disposal of all samples received or processed by Katahdin. Refer to the current revision of Katahdin SOP CA-107 regarding the disposal of spent preparation and analysis reagents, standards, sample extracts, distillates, or digestates.

1.1 Definitions

Hazardous Waste – A "Solid Waste" which displays a hazardous characteristic or is specifically listed as hazardous waste.

Solid Waste – Any discarded material that is not excluded from the definition of hazardous waste.

Discarded Material – Material that is abandoned, recycled or inherently waste-like.

Waste (State of Maine) –

- Any useless, unwanted, or discarded substance or material, whether or not such substance or material has any other future use.
- Any substance or material that is spilled, leaked, pumped, poured, emptied or dumped onto the land or into the water or ambient air.
- Materials which are used in a matter constituting disposal, burned for energy recovery, reclaimed, or accumulated speculatively.

Ignitable Hazardous Waste – EPA Waste Code D001

- Liquids with a flash point less than 140°F or 60°C.
- Solids capable of spontaneous combustion under normal temperature and pressure.
- Ignitable compressed gas.
- Oxidizers.

Corrosive Hazardous Waste - Liquids with a pH less than or equal to 2.0 or greater than or equal to 12.5. EPA waste code D002.

Reactive Hazardous Waste – EPA waste code D003.

- A material that reacts violently with water.
- A material that generates toxic gases or fumes.
- Explosives.

Toxic Hazardous Waste – A material that exceeds certain concentration levels based on the toxicity characteristic leaching procedure (TCLP). See Figure 3 for the chemicals and concentration levels covered under this definition.

Listed Wastes – Lists of chemicals that are considered hazardous based on the following criteria

- Virgin chemical or unused product.
- Sole active ingredient.
- Single substance spill debris.

Listed wastes are divided into 5 subcategories

- F-wastes – Describe hazardous waste from non-specific sources usually containing halogenated and non-halogenated solvents.
- K-wastes – Describe hazardous wastes created by specific processes.
- U-wastes – Describe toxic or non-acute hazardous wastes.
- P-wastes – Describe acute hazardous wastes. (Note: Maine considers a material to be a P-listed waste if it contains 10% or more of any P-listed chemical.
- State listed wastes – Maine lists any material with a concentration of greater than 50 ppm Polychlorinated Biphenyls (PCB) as a hazardous waste.

Organics hit – A liquid sample containing greater than 1 mg/L of organic contaminants or a soil sample containing greater than 20 mg/kg of organic contaminants.

1.2 Responsibilities

Only designated analysts/technicians trained in these procedures may dispose of samples or analytical by-products. Each analyst or technician must be familiar with Katahdin Analytical safety procedures. Gloves, safety glasses, lab coats and/or other protective clothing must be worn at all times.

It is the responsibility of the designated Katahdin personnel involved in the disposal of samples to read and understand this SOP, to adhere to the procedures outlined, to properly document their activities in the appropriate lab notebook and file the necessary manifests and reports to outside agencies in the required manner. Refer to

Katahdin SOP QA-805, "Personnel Training & Documentation of Capability," current revision.

It is the responsibility of the Department Managers to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

It is the responsibility of the Katahdin Environmental Health & Safety Officer (EHSO) to manage the proper classification and disposal of samples. Katahdin is responsible for regulatory compliance of Katahdin's waste storage areas (less than 90 day storage). The EHSO ensures compliance of the waste storage areas with applicable state and federal regulations. The EHSO is responsible for providing the appropriate training to all individuals involved in the proper classification and/or disposal of samples. The EHSO is responsible for working with the Laboratory Operations Manager/Environmental Compliance Officer to help identify problems and assure resolution, to facilitate corrective action where needed, and to communicate unresolved problems and concerns to the Laboratory Vice President.

It is the responsibility of the Operations Manager/Environmental Compliance Officer to oversee adherence to Katahdin sample disposal and hazardous waste practices by all laboratory groups under his/her authority, to help identify problems and assure resolution, to facilitate corrective action where needed, and to communicate problems and concerns to the EHSO and/or the Laboratory Vice President.

It is the responsibility of the Laboratory Vice President to provide the necessary resources to meet the regulatory requirements of proper classification and disposal of samples.

2.0 SUMMARY OF METHOD

Not applicable.

3.0 INTERFERENCES

Not applicable.

4.0 APPARATUS AND MATERIALS

Not applicable.

5.0 REAGENTS

Not applicable.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Not applicable.

7.0 PROCEDURES

7.1 Sample purging is the removal of samples from laboratory refrigerated storage. Sample storage areas where samples are removed (purged) from include wet chemistry, organic extractables, metals, volatiles, total organic carbon and soils. Wet chemistry, aqueous metals, organic extractables, total organic carbon, and soils can all be found in the walk-in refrigerator. Aqueous and soil volatiles can be found in the volatiles laboratory refrigerators/freezer.

7.2 Samples are purged from storage, after analysis and reporting, on a routine basis to make room for incoming samples. Samples are to be kept in storage for a duration of 30 days past the report mailed date. Some samples must be kept for 60 or 90 days beyond the report mailed date, depending on specific client requests and contracts.

7.3 The first step in disposing of samples is to generate a disposal list. The disposal list contains sample analysis information stored in the Katahdin Information Management System (KIMS). The analytical data for the samples is compared to the hazardous waste criteria specified in 40CFR Part 261 and to local wastewater discharge criteria. Refer to Figure 4 for 40 CFR Part 261 Characteristic Hazardous Waste Criteria. Based on this comparison, the report displays information on the classification/category for disposal of each sample. The disposal report should be reviewed against the data reports for accuracy. Refer to Figure 2 for an example of a KIMS generated disposal list. The primary disposal categories listed in the report are: non-hazardous, high organics, high metals, flashpoint, high mercury, high PCBs, and high cyanide. Katahdin has established 14 waste stream profiles with a 3rd party waste transporter/waste disposal firm for sample disposal based on these categories. As required, new or special temporary waste profiles are established based on the characteristics of samples.

7.4 Sorting through samples and preparing them for disposal is a crucial quality checkpoint. Samples put into the incorrect waste stream could not only produce adverse environmental effects, but, could also interrupt the 3rd party's waste treatment efficiency, or endanger an individual handling the waste stream. Therefore, when sorting through samples pay close attention to which waste stream each sample falls into.

7.5 Once you are ready to dispose of the samples of interest (the oldest samples that have been purged), these samples must be sorted, logged, and the classification/category (sample knowledge) information recorded.

Sample storage times (as listed in section 7.2) and space should be taken into consideration when purging samples. It is important to make room for future samples, but to make sure that samples are not purged too early. Samples should be pulled from the walk-in or the volatiles refrigerators to make room for new samples. When purging, chose a section that needs extra space the most and remove the oldest samples.

Safety glasses, nitrile gloves, lab coat, and a splash apron must be worn when handing samples during disposal

7.6 Remove the designated purge samples from the shelf one by one and line them up on the countertop in the log-in area. Generally, removing two cartloads at a time is a good amount to purge at one time. For volatile samples in 40mL vials, 5 or 6 vial trays should be purged at a time. Samples should be lined up across the counter with the earliest sample to the left and building up to the right, organizing the samples according to work order and sample number. After the samples are lined up, they should be recorded in the Sample Disposal Logbook (SDL). Refer to Figure 1 for an example SDL page. The location the samples were removed from should also be recorded. Sample storage areas are recorded with the following designations:

VOA (Aq)	Aqueous Volatiles(VOA)
VOA (SL)	Solid Volatiles(VOA)
M	Metals
EXT	Extractables (Organic)
TOC	Total Organic Carbon
WC	Wet Chemistry
S	Soils

7.7 The next step is to use the sample disposal list to determine the earliest release date of the reports and to determine each samples appropriate waste classification/characterization. As stated in section 7.3, the primary disposal categories listed in the report are: non-hazardous, high organics, high metals, flashpoint, high mercury, high PCBs, and high cyanide.

Using the information from the KIMS disposal list, record the appropriate classification for each sample in the SDL. If multiple categories are identified as being present then a single category is selected as controlling. The order of precedence is PCB's, metals and then organics. If another scenario is found, the individual should bring it to the EHSO for a determination of the acceptable waste stream designation or a determination that it should be lab packed separately.

If samples have been sorted that have not been in storage for the 30 days beyond the release date (60 or 90 for certain clients), then these samples need to be placed back in storage and it should be noted in the SDL.

7.8 As stated above, a sample may be categorized into a waste stream based upon the analytes it contains as determined by laboratory testing. In addition, many samples are also categorized as hazardous waste based upon the preservative that they contain. Since many samples contain preservatives, caution must be used when dumping samples. It is also important to ensure that the sample container is empty. This can be accomplished by holding the container upside down and shaking gently until liquid is no longer observed coming out of the container.

7.9 Once waste categories have been determined and entered into the SDL, The following waste categories are disposed of as follows:

7.9.1 Dumping non-hazardous samples (as determined by laboratory testing)

Non-hazardous samples (non-preserved) are poured directly into the sink in the warehouse.

Non-hazardous solid samples are disposed of with the general trash, which is picked up by commercial trash collectors and ultimately disposed of in a waste-to-energy incinerator.

Sample containers from non-hazardous samples are disposed of with the general trash.

7.9.2 Dumping Samples with high Organics (as determined by laboratory testing)

Aqueous samples get dumped into waste stream "K". Containers are disposed of with general trash. Solid samples are placed into waste stream "I" with their containers. The disposal date is recorded in the SDL.

7.9.3 Dumping samples high in metals, including mercury (as determined by the by laboratory testing)

Aqueous samples get disposed of in waste stream "A". Containers are disposed of with general trash. Solid samples are placed in waste stream "L" with their containers. The disposal date is recorded in the SDL.

7.9.4 Dumping Acidic Samples that do not contain any other hazardous waste constituents (as determined by the acidic preservative or by laboratory testing)

Refer to section 7.10 below.

7.9.5 Dumping Basic samples (as determined by the basic preservative or by laboratory testing)

Aqueous samples get disposed of in waste stream "NHi". Containers are disposed of with general trash. The disposal date is recorded in the SDL.

7.9.6 Dumping samples with high PCBs (as determined by laboratory testing)

Aqueous samples are disposed of in waste stream "Q". Containers are disposed of with general trash. Solid samples get disposed of in waste stream "F" with their containers. The disposal date is recorded in the SDL.

7.9.7 Dumping samples with low flashpoints (as determined by laboratory testing)

Aqueous samples are disposed of in waste stream "O". Containers are disposed of with general trash. Solid samples get disposed of in waste stream "I" with their containers. The disposal date is recorded in the SDL.

7.9.8 Dumping samples with high cyanide (as determined by laboratory testing)

Aqueous samples are disposed of in waste stream "NHi". Containers are disposed of with general trash. Solid samples should be set aside for labpack. The disposal date is recorded in the SDL.

7.9.9 Miscellaneous Disposal (as determined by the preservative)

Sodium Bisulfate: Sodium Bisulfate often comes in vials, but may also come in the 2-4oz glass jars. Dump the Sodium Bisulfate out of the container into waste stream "A". There may be remaining soil left in the sample container. The soil's waste stream and dump date will be dictated by the SDL. The disposal date is recorded in the SDL.

Methanol / Free Products: This often comes in vials, but may also come in the 2-4oz glass jars. Dump the methanol out of the container into the mix-flammables accumulation. When this satellite accumulation container gets full it can be dumped into the "O" waste stream. There may be remaining soil left in the sample container. The soil's waste stream and dump date will be dictated by the SDL. Lastly, samples marked "free product" on the Katahdin sample ID label can be dumped into the mixed flammables stream. The disposal date is recorded in the SDL.

7.10 Pursuant to Maine DEP regulations, Katahdin has the necessary agreements, processes and documentation in place to neutralize samples without a license. Refer to the current revision of the Katahdin Environmental Health & Safety Manual for additional information. Generally, the following procedures are followed.

7.10.1 Samples that have been determined to be hazardous due **solely** to the corrosivity characteristic are neutralized using sodium hydroxide pellets. In the warehouse, samples are emptied into a five gallon heavy duty carboy to about 60% capacity. The carboy is kept in a secondary container. Sodium hydroxide pellets are added slowly to the carboy (about 5 grams at a time) and stirred with a long glass stirring rod. The pH is checked with pH paper.

7.10.2 This process is continued until the pH is between 7 and 8. This normally takes about 30-40 grams of sodium hydroxide pellets, but may vary depending on the buffering capacity of the individual samples.

7.10.3 The carboy is emptied into the sink in the warehouse. The tap water is run at the same time as the neutralized material is disposed of. An eyewash station and spill material is located at this sink.

7.10.4 All neutralization activities are documented, including the date and time of neutralization, the name of the person doing the neutralizing, the amount of neutralized liquid discharged, details on the inspection of the drain area and the date and nature of any significant repairs or corrective actions. This documentation is maintained by the EHSO. Refer to Figure 5 for an example logbook page of neutralization documentation.

7.11 Every 3 to 5 weeks a pickup of hazardous waste is scheduled with the 3rd party waste transporter/waste disposal firm. An inventory is faxed to the transporter summarizing the number of drums and waste streams/profiles. As required, a "lab pack" of expired chemicals or orphan samples is organized as necessary. A designated individual, with applicable Hazardous Waste (RCRA) and Department of Transportation (DOT) training, oversees the waste pickup and signs the hazardous manifests and land ban documentation. Within 7 days a copy is forwarded to the Maine Department of Environmental Protection (MEDEP) and the environmental agency in the designation state (if required by that state). Once the report is received at the disposal facility a copy is returned to KATAHDIN and the MEDEP.

7.12 Prior to March 31 of each year, the laboratory prepares the Annual Hazardous Waste Report (i.e., MEDEP modified EPA Form 8700-13A) as required by MEDEP Hazardous Waste Management Rules. The complete report is reviewed by the Katahdin Environmental Compliance Officer and then forwarded to the following address:

Maine Department of Environmental Protection
Bureau of Remediation & Waste Management
State House Station #17
Augusta, ME. 04333
Attn: Annual Hazardous Waste Report

TITLE: SAMPLE DISPOSAL

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

On a daily basis, a designated individual performs quality checks in all hazardous waste storage areas. The daily check documentation is located in login. Any discrepancy is copied to the Operations Manager and the Katahdin Vice President for corrective action. Refer to the current revision of Katahdin SOP CA-107, *The Management of Hazardous Waste as it Relates to the Disposal of Laboratory Process Waste, Reagents, Solvents & Standards*, for more information. Refer to Figure 3 for a copy of the daily check documentation.

9.0 METHOD PERFORMANCE

Not applicable.

10.0 APPLICABLE DOCUMENTS/REFERENCES

USEPA Code of Federal Regulations, 40 CFR Part 261.

Maine Department of Environmental Protection (ME DEP) Hazardous Waste Management Rules

ME DEP modified EPA Form 8700-13A

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Figure 1 Example of Sample Disposal Logbook
Figure 2 Example of KIMS Generated Waste Disposal Report
Figure 3 Example Of Hazardous Waste Area Daily Check Documentation
Figure 4 Characteristic Toxic Hazardous Waste and TCLP concentrations
Figure 5 Example of Elementary Neutralization Logbook

TITLE: SAMPLE DISPOSAL

FIGURE 1

EXAMPLE OF SAMPLE DISPOSAL LOGBOOK (SDL)

KATAHDIN ANALYTICAL SERVICES, INC. –SAMPLE STORAGE/DISPOSAL LOGBOOK

WORK ORDER/ SAMPLE NUMBERS	DEPARTMENT	EARLIEST RELEASE DATE	< CRITERIA	SAMPLE KNOWLEDGE									DATE DISPOSED	INITIALS
			CLEAN	WL	ORG	METS	CN	FP	HG	PCBS				
SAS783-1	WC	10-17-07	✓										1-22-08	GN
SAS786-1		10-17-07	✓											
SAS787-1,2,4		10-17-07	✓											
SAS790-1		10-19-07		✓										
SAS793-1		10-17-07	✓											
SAS795-1-9		10-23-07	✓											
SAS797-1		10-23-07	✓											
SAS798-1,2		10-25-07			✓									
SAS799-1-5		10-31-07	✓											
SAS804-1,2		10-25-07	✓											
SAS809-1,2		10-23-07	1			2				2				
SAS810-1-4	✓	10-23-07	✓										✓	

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TITLE: SAMPLE DISPOSAL

FIGURE 2

EXAMPLE OF KIMS GENERATED WASTE DISPOSAL REPORT

SAMPLE DISPOSAL REPORT

Query by: Login SA6501 to SA7000
Date : 15-JAN-08

Sample	SDG	Status	Mail Date	Parameter	Value		
SA6605-1		NEED	12/02/07				
SA6606-1		NEED	12/02/07				
SA6607-1		NEED	11/15/07				
SA6608-1		NEED	12/06/07				
				ORG	1.17	MG/L	(HIGH)
SA6608-1		NEED	12/06/07				
SA6608-2		NEED	12/06/07				
				AA	13	MG/KG	(HIGH)
SA6609-1		NEED	11/26/07				
SA6609-1		NEED	11/26/07				
SA6610-1		NEED	11/30/07				
SA6611-1	FCS-020	NEED	12/07/07				
SA6611-2	FCS-020	NEED	12/07/07				
SA6611-3	FCS-020	NEED	12/07/07				
SA6611-4	FCS-020	NEED	12/07/07				
SA6611-5	FCS-020	NEED	12/07/07				
SA6611-6	FCS-020	NEED	12/07/07				
SA6611-7	FCS-020	NEED	12/07/07				
SA6611-8	FCS-020	NEED	12/07/07				
SA6612-1	NSA-030	NEED	12/07/07				
SA6612-2	NSA-030	NEED	12/07/07				
SA6612-3	NSA-030	NEED	12/07/07				
SA6612-4	NSA-030	NEED	12/07/07				
				ORG	1.70735	MG/L	(HIGH)
SA6612-5	NSA-030	NEED	12/07/07				
				ORG	1.0481	MG/L	(HIGH)

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TITLE: SAMPLE DISPOSAL

FIGURE 3

EXAMPLE OF HAZARDOUS WASTE STORAGE AREA DAILY CHECK

Daily Checklist for
HAZARDOUS WASTE STORAGE AREA

Week of: 1-28, 20 08

Item/Day:	Monday	Tuesday	Wednesday	Thursday	Friday
1. Are containers closed? (Except when waste is being added)	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
2. Are containers properly labeled with a hazardous waste label?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
3. Do you have access to each container and can you read the label? (36" aisle?)	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
4. Is each container marked with the date storage began?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
5. Are the dates on the containers less than 90 days old?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
6. Is container free of dents, bulges, rust, spills or leaks?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
7. Are all containers on a firm working surface?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
8. Inspection by: Name (No Initials)	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
9. Time of Inspection	16:35	15:00	14:45	14:15	16:25
10. Verification of Inspection (Name/Date)	[illegible] 1-28-08	[illegible] 1-29-08	[illegible] 1-30-08	[illegible] 1-31-08	[illegible] 2-01-08

Deficiency noted:					
Corrective action:					
By (Name/Date):					

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FIGURE 4

CHARACTERISTIC TOXIC HAZARDOUS WASTE AND TCLP CONCENTRATIONS

Chemical Name	CAS Number	Waste Code	TCLP conc. liquid	Equivalent conc. In Soil
Arsenic	7440-38-2	D004	5.0 mg/L	100 mg/kg
Barium	7440-39-3	D005	100 mg/L	2000 mg/kg
Cadmium	7440-43-9	D006	1.0 mg/L	20 mg/kg
Chromium	7440-47-3	D007	5.0 mg/L	100 mg/kg
Lead	7439-92-1	D008	5.0 mg/L	100 mg/kg
Mercury	7439-97-6	D009	0.2 mg/L	4 mg/kg
Selenium	7782-49-2	D010	1.0 mg/L	100 mg/kg
Silver	7440-22-4	D011	5.0 mg/L	20 mg/kg
Endrin	72-20-8	D012	0.02 mg/L	0.4 mg/kg
Lindane	58-89-9	D013	0.4 mg/L	8 mg/kg
Methoxychlor	72-43-5	D014	10 mg/L	200 mg/kg
Toxaphene	8001-35-2	D015	0.5 mg/L	10 mg/kg
2,4-D	94-75-7	D016	10 mg/L	200 mg/kg
2,4,5-TP (Silvex)	93-72-1	D017	1.0 mg/L	20 mg/kg
Benzene	71-43-2	D018	0.5 mg/L	10 mg/kg
Carbon Tetrachloride	56-23-5	D019	0.5 mg/L	10 mg/kg
Chlordane	57-74-9	D020	0.03 mg/L	0.6 mg/kg
Chlorobenzene	108-90-7	D021	100 mg/L	2000 mg/kg
Chloroform	67-66-3	D022	6.0 mg/L	120 mg/kg
o-Cresol	95-48-7	D023	200 mg/L	4000 mg/kg
m-Cresol	108-39-4	D024	200 mg/L	4000 mg/kg
p-Cresol	106-44-5	D025	200 mg/L	4000 mg/kg
Cresol	1319-77-3	D026	200 mg/L	4000 mg/kg
1,4-Dichlorobenzene	106-46-7	D027	7.5 mg/L	150 mg/kg
1,2-Dichloroethane	107-06-2	D028	0.5 mg/L	10 mg/kg
1,1-Dichloroethylene	75-35-4	D029	0.7 mg/L	14 mg/kg
2,4-Dinitrotoluene	121-14-2	D030	0.13 mg/L	2.6 mg/kg
Heptachlor	76-44-8	D031	0.008 mg/L	0.16 mg/kg
Hexachlorobenzene	118-74-1	D032	0.13 mg/L	2.6 mg/kg
Hexachlorobutadiene	87-68-3	D033	0.5 mg/L	10 mg/kg
Hexachloroethane	67-72-1	D034	3.0 mg/L	60 mg/kg
Methyl Ethyl Ketone	78-93-3	D035	200 mg/L	4000 mg/kg
Nitrobenzene	98-95-3	D036	2.0 mg/L	40 mg/kg
Pentachlorophenol	87-86-5	D037	100 mg/L	2000 mg/kg
Pyridine	110-86-1	D038	5.0 mg/L	100 mg/kg
Tetrachloroethylene	127-18-4	D039	0.7 mg/L	14 mg/kg
Trichloroethylene	79-01-6	D040	0.5 mg/L	10 mg/kg

FIGURE 4, cont'd

CHARACTERISTIC TOXIC HAZARDOUS WASTE AND TCLP CONCENTRATIONS

Chemical Name	CAS Number	Waste Code	TCLP conc. liquid	Equivalent conc. In Soil
2,4,5-Trichlorophenol	95-95-4	D041	400 mg/L	8000 mg/kg
2,4,6-Trichlorophenol	88-06-2	D042	2.0 mg/L	40 mg/kg
Vinyl Chloride	75-01-4	D043	0.2 mg/L	4.0 mg/kg

TITLE: SAMPLE DISPOSAL

FIGURE 5

EXAMPLE OF ELEMENTARY NEUTRALIZATION LOGBOOK

Katahdin Analytical Services, Inc. – Elementary Neutralization Logbook

Date: 3-4-09		**Time:** 12:00	**Analyst:** GN
# of gallons neutralized	Final pH	Condition of drain and sink area before and after neutralization.	Significant Repairs or Corrective Actions
5	5	good	
6	7	good	
6	5	good	
6	6	good	
2	8	good	

25
240

Date: 3-10-09		**Time:** 13:45	**Analyst:** GN
# of gallons neutralized	Final pH	Condition of drain and sink area before and after neutralization.	Significant Repairs or Corrective Actions
6	7	good	
6	6	good	
5	6	good	
6	8	good	
6	5	good	
6	8	good	
5	5	good	
6	7	good	
3	5	good	

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Title: Particle Size Analysis (ASTM D 2217 and D422-63)

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1.0 Scope and Application

This SOP describes the laboratory procedure for the determination of particle size distribution in soils.

2.0 Summary of Method

A portion of sample is soaked in a dispersing agent then partitioned into separate portions, material retained on a #10 sieve and material passing the #10 sieve. The material retained on the #10 sieve is dried to constant weight then passed through a large size sieve stack; the material retained on each sieve is measured and recorded. Material passing the #10 sieve is subject to hydrometer analysis then passed through a small size sieve stack, the material retained on each sieve is measured and recorded. All measurements, large and small sieves and hydrometer readings and the hygroscopic moisture are used to establish the particle size distribution of the sample.

This SOP is based on the following reference methods:

- ASTM Standard D 2217 – 85 (Rapproved 1998) "Standard Practice for Wet Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

- ASTM Standard D 422-63 (Rapproved 2007) "Standard Test Method for Particle-Size Analysis of Soils", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

NOTE: ASTM D2217 was method was withdrawn without replacement by ASTM in 2007. A withdrawn standard is an ASTM standard that has been discontinued by the ASTM Sponsoring Committee responsible for the standard.

If the laboratory has modified the procedure from the reference method(s) a list of modifications will be provided in Section 16.0.

3.0 Definitions

Not Applicable

4.0 Interferences

Not Applicable

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

None

5.2 Primary Materials Used

Not Applicable

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment used meets the specification of this SOP.

- Top-Loading Balance, capable of weight measurement to 0.01 g
- Mechanical Stirring Device and Dispersion Cup
- Thermometer: Accurate to 0.5°C
- Mortar and Rubber Tipped Pestle
- Sedimentation Cylinder(s) 1000 mL
- Hydrometer: ASTM 151H in specification E 100.
- Sieves, of the following size(s): Gilson Company, Inc. or equivalent
  3.0” (75.00 mm)
  2.0” (50.00 mm)
  1.5” (37.50 mm)
  1.0” (25.00 mm)
  3/4" (19.00 mm)
  3/8” (9.50 mm)
  # 4 (4.75 mm)
  #10 (2.00 mm)
  #20 (850.0 um)
  #40 (425 um)
  #60 (250.0 um)
  #80 (180.0 um)
  #100 (150.0 um)
  #200 (75.0 um)
- Drying Oven with temperature range of 60-110°C
- Stainless Steel Spatulas & Spoons
- Metal & Bristle Brushes
- Ro-Tap Sieve Shaker, W. S. Tyler or equivalent.
- Timing Device with second hand and capable of counting up to 25 hours

7.0 Reagents and Standards

- Reverse Osmosis (RO) water: In-House System
- Sodium Hexametaphosphate: ELE International or equivalent.

Sodium Hexametaphosphate Solution: Add 120 g of sodium hexametaphosphate and 2940 g of reagent water to a 1-gallon bottle. Add a stir rod to the container and place on a stir plate. Mix the solution until it is homogeneous. Assign an expiration date of 30 days from the date made

unless the parent reagent expires sooner in which case use the earliest expiration date. Store the prepared solution at ambient temperature.

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection so these procedures are not included in this SOP. Sampling requirements may be found in the published reference method.

Listed below are minimum sample size, preservation and holding time requirements:

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time	Reference
Solid	Glass Jar w/ Teflon Lid	500 g	None	None	ASTM D422-63

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

Not Applicable

10.0 Procedure

10.1 Equipment Calibration

Check the calibration of the balance on each day of use prior to use using at least 2 Class S weights that bracket the range of use. Record in the logbook designated for this purpose.

Check the temperature of the drying oven(s) each day of use, prior to use. Record in the logbook designated for this purpose.

NOTE: The QA Manager or her designee checks the calibration of liquid in glass thermometers annually against a NIST-traceable thermometer following the procedures given in laboratory SOP BR-QA-004. Electronic / digital thermometers that are battery-operated are checked quarterly using the same procedure.

Calibrate the hydrometers every two years following the procedure given in BR-GT-008.

Calibrate the sieves 6 months following the procedure given in BR-GT-008.

Calibrate the Ro-Tap sieve shaker every 12 months following the procedure given in BR-GT-008.

10.2 Hygroscopic Moisture Determination

Label an aluminum pan with the Lab ID for each sample. Tare the balance, weigh each pan and record the weight measurement in the spreadsheet.

Mix the sample with a stainless steel spatula. Measure at least 10-15 g of each sample into the labeled aluminum pan and record the weight of sample in the spreadsheet.

Place the pan + sample in an oven maintained at a temperature of 110ºC and dry the sample for at least 16 hours. Reweigh each pan and record the weight measurement in the spreadsheet.

Percent solids are calculated using the equation given in Section 11.0.

10.3 Sample Preparation

Use the calculated percent solids and the sample characteristic for each sample to determine the amount needed for analysis using Table 2. For example, if the calculated percent solids for a sample are 50% and the sample characteristic is sand, use 200 g for analysis. If there is an insufficient amount of sample available, initiate a nonconformance memo (NCM) and contact the PM for further instruction.

Place a 1000 mL plastic beaker on the balance and tare the balance. Weight the amount of sample for analysis and record the weight in the bench sheet.

Add 125 mL of sodium hexametaphosphate solution to each beaker. Stir to mix and soak the sample in this solution for 16 hours

10.4 Sample Partition

Rinse the sample slurry into a dispersion cup using reagent water. Fill the dispersion cup ½ full with reagent water and place the cup on the blender to mix for one minute.

NOTE: Some samples may not be amendable to using the blender examples include but not limited to large gravel, sands, or organic material. If the sample is not amenable, initiate a NCM to notify the PM of the anomaly and proceed to the next step without blending the sample.

Place a #10 sieve on a 1000 mL graduated cylinder. Pour the sample through the sieve. Rinse the dispersion cup with reagent water and pour the rinse through the sieve. Repeat until transfer is complete. Bring the volume in the graduated cylinder to 1000 mL with reagent water. Cover the cylinder with a rubber stopper and equilibrate the sample to ambient temperature in preparation for hydrometer analysis.

Label a medium size aluminum dish with the sample's LAB ID then transfer the sample material that was retained on the #10 sieve to the dish. Place the aluminum dish in the drying oven set at 110 ± 5° C and dry the sample material for at least 16 hours or until constant weight. Set aside for sieve analysis.

10.5 Hydrometer Analysis

Prepare a hydrometer rinse bath by adding 1000 mL of reagent water to a 1000 mL graduated cylinder

Record the hydrometer ID and start time on the worksheet. Set the timer for the elapsed time and perform each task as listed in Table 1: Hydrometer Reading Table.

To shake the cylinder, rotate the flask up and down for one minute approximating at least 60 turns. One turn down and one turn up equals two turns.

To take a hydrometer reading, gently insert the hydrometer into the graduated cylinder and wait ~ 20 seconds. Read the hydrometer from the top of the meniscus to the nearest 0.0005. Enter the reading on the worksheet. After each reading, clean the hydrometer by twisting and dropping the hydrometer into the hydrometer rinse bath.

Insert a temperature probe into the cylinder to the same depth used for the hydrometer reading. Read the temperature to the nearest 0.5°C and enter the temperature measurement on the worksheet. Rinse the temperature probe in the hydrometer rinse bath.

Repeat the above process taking hydrometer readings every 2, 5, 15, 30, 60, 240 and 1440 minutes as per Table 1 then proceed to small sieve analysis.

10.6 Sieve Analysis

Inspect the sample material in the aluminum pan and record a description of the non-soil material (e.g.- sticks, grass, wood, plastic), hardness of material and shape of material in the worksheet.

Hardness qualifiers include hard, soft or brittle. Shape qualifiers include well rounded, rounded, subrounded, subangular, and angular.

<u>Large Sieves</u>

Weigh the 3/4”, 3/8”, #4 and #10 sieves and enter the weight measurements in the worksheet as the tare weight.

Stack the sieves then transfer the sample material from the aluminum dish to the sieve stack. If the sample material is less than 30 g, manually shake the sieve stack for 2 minutes. If the sample material is greater than 30 g, place the sieve stack into the Ro-tap machine and shake the sieve stack for 10 minutes.

Weigh each sieve and record these measurements in the worksheet.

<u>Small Sieves</u>

Transfer the sample from the graduated cylinder to a #200 wet wash sieve. Wash the sample through the #200 sieve until the water runs clear then transfer the material retained on the sieve to a 250 mL glass beaker labeled with the sample’s LAB ID.

Place the beaker in the drying oven and dry at a temperature of 110ºC for at least 16 hours. After 16 hours, remove the beaker from the oven and allow it to cool.

Gently mix the dried contents of the beaker with a rubber-tipped pestle to break any soil aggregates that may have formed during the drying stage.

Tare the balance and weigh the sieve stack sized between #20 and #200 and record the tare weights.

Transfer the sample to the sieve stack and ensure complete transfer. Use hair or wire brushes to clean the beaker. Place the sieve stack on the Rotap machine and shake for ten minutes.

Weigh each sieve and record these measurements in the worksheet.

11.0 Calculations / Data Reduction

11.1 Calculations

Sample Used (SU): Dry Preparation

$$SU = (pan + dry\ sample - pan) - (pan + non - soil\ material - pan) \otimes HMCF$$

Where:
HMCF = Hygroscopic moisture correction factor.

Sieve Analysis (Percent Finer = PF)

Large Sieves:

3 inch: PF = 100-100 (Sieve and Sample (3 inch) - Sieve (3 inch))/SU*

2 inch: PF = PF (3 inch) - 100(Sieve and Sample (2 inch) - Sieve (2 inch))/SU and so on through the #10 Sieve.*

Small Sieves:

#20: PF = PF(#10) - 100(mass passing #10/sample mass (Hyd))*(sieve and sample (#20) - sieve(#20))/sample used*

#40: PF = PF (#20) - 100(mass passing #10/sample mass (Hyd))*(sieve and sample (#40) - sieve (#40))/sample used and so on up through #10 sieve.*

Hydrometer Analysis

Particle size, Micron

1000*sqrt [930*viscosity/980*(SG-1))*(effective depth/time)]

Viscosity at sample temperature, poises
Effective Depth, cm = 16.29-264.5*(actual Hydrometer reading - 1) above equation for effective depth based on equation found with table 2 in method, in which 16.29 = 0.5*(14.0-67.0/27.8)+10.5 and 264.5 = (10.5-2.3)/0.031
Time, minutes = Time of hydrometer reading from beginning of sedimentation
Sqrt - square root
SG - Specific Gravity of soil
Viscosity - is the resistance of a liquid to flow
Percent Finer (PF):

PF = Constant*(actual hydrometer reading - hydrometer correction factor - 1)

Where:
Constant = (100,000/W)*SG/(SG-1)
W = (Total sample used *sample used for hydrometer analysis*HMCF)/Amount of total sample

passing #10 sieve
Hydrometer Correction = slope*sample temperature + Intercept
Slope = ((low temp. reading -1)-(high temp. reading -1)/(low temp. - high temp.))
Intercept = (low temp. reading -1) - (low temp. * slope)

11.2 Data Reduction

11.2.1 Primary Data Review

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Upload the batch information into LIMS and complete the batch editor and worksheet. Initiate NCMs for any anomalies observed during the preparation process. Set the status of the batch to 1st level review.

11.2.2 Secondary Data Review

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements and verify those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Check the batch editor and worksheet to verify the batch is complete and any outages are documented with an NCM along with the results of any corrective actions taken. Set the status of the batch to second level review.

11.2.3 Lab Complete

Review the batch, run QC checker as appropriate and set the status to lab complete.

11.2.4 Data Reporting

Sample results are reported from the laboratory's LIMS system using the formatter specified by the Project Manager.

11.2.5 Data Archival

Data are stored in the laboratory's LIMS system.

12.0 Method Performance

Not Applicable

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide

by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001. The following waste streams are produced when this method is carried out.

- Solid Waste-Satellite Container: Solid Waste 5 Gallon Plastic Bucket (inside fume hood)
- Liquid Waste- 55 gallon poly drum

15.0 References / Cross-References

- ASTM Standard D 2217 – 85 (Reapproved 1998) "Standard Practice for Wet Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

- ASTM Standard D 422-63 (Rapproved 2007) "Standard Test Method for Particle-Size Analysis of Soils", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

16.0 Method Modifications

D2217: The laboratory performs sample portioning after soaking the solution in the dispersing agent because the dispersion agent helps break up aggregates associated with clay and sediments.

D422: The laboratory does not always use the recommended amount of sample for analysis because sufficient sample volume is not always received.

17.0 Attachments

- Table 1: Hydrometer Reading Table (For up to 12 Sedimentation Cylinders)
- Table 2: Percent Solids Table for Weight Determination for D422.

18.0 Revision History

BR-GT-006, Revision 6:

- Title Page: Updated approval signatures
- All Sections: Removed references to dry preparation by ASTM D421; Added procedure for wet preparation.
- Attachments: Inserted Percent Solids Table

Table 1: Hydrometer Reading Table (For up to 12 Sedimentation Cylinders)

Elapsed Time (hr:min)	Task	Cyl. No.	Actual Time (min)	Elapsed Time (hr:min)	Task	Cyl. No.	Actual Time (min)
0:00	Shake	1		1:01	Read	10	5
0:01	**Place**	**1**		**1:02**	Shake	11	
0:01	**Shake**	**2**		**1:03**	Place	11	
0:02	Place	2		1:04	Read	9	15
0:03	Read	1	2	1:05	Read	11	2
0:04	Read	2	2	1:06	Read	7	31
0:06	Read	1	5	1:07	Read	3	58
0:07	Read	2	5	1:08	Read	11	5
0:08	Shake	3		**1:09**	Shake	12	
0:09	**Place**	**3**		**1:10**	Place	12	
0:09	**Shake**	**4**		1:11	Read	10	15
0:10	Place	4		1:12	Read	12	2
0:11	Read	3	2	1:13	Read	4	63
0:12	Read	4	2	1:14	Read	8	32
0:14	Read	3	5	1:15	Read	12	5
0:15	Read	4	5	1:18	Read	11	15
0:16	Read	1	15	1:19	Read	9	30
0:17	Read	2	15	1:21	Read	5	60
0:20	Shake	5		1:25	Read	12	15
0:21	Place	5		1:26	Read	10	30
0:23	Read	5	2	1:27	Read	6	59
0:24	Read	3	15	1:33	Read	11	30
0:25	Read	4	15	1:34	Read	7	59
0:26	Read	5	5	1:41	Read	12	31
0:27	Shake	6		1:42	Read	8	60
0:28	Place	6		1:52	Read	9	63
0:30	Read	6	2	1:53	Read	10	57
0:31	Read	1	30	2:06	Read	11	63
0:32	Read	2	30	2:07	Read	12	57
0:33	Read	6	5	4:17	Read	1	256
0:34	Shake	7		4:18	Read	2	256
0:35	Place	7		4:19	Read	3	250
0:36	Read	5	15	4:20	Read	4	250
0:37	Read	7	2	4:21	Read	5	240
0:38	Read	3	29	4:22	Read	6	234
0:39	Read	4	29	5:00	Read	7	265
0:40	Read	7	5	5:01	Read	8	259
0:41	Shake	8		5:02	Read	9	253
0:42	Place	8		5:03	Read	10	247
0:43	Read	6	15	5:04	Read	11	241
0:44	Read	8	2	5:05	Read	12	235
0:47	Read	8	5	24:01	Read	1	1440
0:48	Shake	9		24:02	Read	2	1440
0:49	Place	9		24:03	Read	3	1434
0:50	Read	7	15	24:04	Read	4	1434
0:51	Read	9	2	24:05	Read	5	1424
0:52	Read	5	31	24:06	Read	6	1418
0:54	Read	9	5	24:07	Read	7	1412
0:55	Shake	10		24:08	Read	8	1406

0:56	Place	10		24:09	Read	9	1400
0:57	Read	8	15	24:10	Read	10	1394
0:58	Read	10	2	24:11	Read	11	1388
0:59	Read	6	31	24:12	Read	12	1382
1:00	**Read**	**1**	**59**				
1:00	**Read**	**2**	**58**				

Source: Laboratory Prepared Reference Document

Table 2: Percent Solids Table for Weight Determination for D422.

Percent Solid Table

Quantities of sample (in grams) to be utilized in Wet method version of ASTM D854 and D422

% Sol	Spec Grav	Hydrometer Slt/Cl	Slt/Snd	Snd	Snd/Gr
	25	**50**	**75**	**100**	**200**
1	2500	5000	7500	10000	20000
2	1250	2500	3750	5000	10000
3	833	1667	2500	3333	6667
4	625	1250	1875	2500	5000
5	500	1000	1500	2000	4000
6	417	833	1250	1667	3333
7	357	714	1071	1429	2857
8	313	625	938	1250	2500
9	278	556	833	1111	2222
10	250	500	750	1000	2000
11	227	455	682	909	1818
12	208	417	625	833	1667
13	192	385	577	769	1538
14	179	357	536	714	1429
15	167	333	500	667	1333
16	156	313	469	625	1250
17	147	294	441	588	1176
18	139	278	417	556	1111
19	132	263	395	526	1053
20	125	250	375	500	1000
21	119	238	357	476	952
22	114	227	341	455	909
23	109	217	326	435	870
24	104	208	313	417	833
25	100	200	300	400	800
26	96	192	288	385	769
27	93	185	278	370	741
28	89	179	268	357	714
29	86	172	259	345	690
30	83	167	250	333	667
31	81	161	242	323	645
32	78	156	234	313	625
33	76	152	227	303	606
34	74	147	221	294	588
35	71	143	214	286	571
36	69	139	208	278	556
37	68	135	203	270	541
38	66	132	197	263	526
39	64	128	192	256	513
40	63	125	188	250	500
41	61	122	183	244	488
42	60	119	179	238	476
43	58	116	174	233	465
44	57	114	170	227	455
45	56	111	167	222	444
46	54	109	163	217	435
47	53	106	160	213	426
48	52	104	156	208	417
49	51	102	153	204	408
50	50	100	150	200	400

% Sol	Spec Grav	Hydrometer Slt/Cl	Slt/Snd	Snd	Snd/Gr
	25	**50**	**75**	**100**	**200**
51	49	98	147	196	392
52	48	96	144	192	385
53	47	94	142	189	377
54	46	93	139	185	370
55	45	91	136	182	364
56	45	89	134	179	357
57	44	88	132	175	351
58	43	86	129	172	345
59	42	85	127	169	339
60	42	83	125	167	333
61	41	82	123	164	328
62	40	81	121	161	323
63	40	79	119	159	317
64	39	78	117	156	313
65	38	77	115	154	308
66	38	76	114	152	303
67	37	75	112	149	299
68	37	74	110	147	294
69	36	72	109	145	290
70	36	71	107	143	286
71	35	70	106	141	282
72	35	69	104	139	278
73	34	68	103	137	274
74	34	68	101	135	270
75	33	67	100	133	267
76	33	66	99	132	263
77	32	65	97	130	260
78	32	64	96	128	256
79	32	63	95	127	253
80	31	63	94	125	250
81	31	62	93	123	247
82	30	61	91	122	244
83	30	60	90	120	241
84	30	60	89	119	238
85	29	59	88	118	235
86	29	58	87	116	233
87	29	57	86	115	230
88	28	57	85	114	227
89	28	56	84	112	225
90	28	56	83	111	222
91	27	55	82	110	220
92	27	54	82	109	217
93	27	54	81	108	215
94	27	53	80	106	213
95	26	53	79	105	211
96	26	52	78	104	208
97	26	52	77	103	206
98	26	51	77	102	204
99	25	51	76	101	202
100	25	50	75	100	200

E-2 ARI

Analytical Resources, Incorporated
Analytical Chemists and Consultants

Standard Operating Procedure

Organic Extractions
Sample Chain of Custody
Archive and Disposal

SOP 1019 S

Revision 3

Revision Date: 10/20/04
Effective Date: 10/20/04

Prepared By:

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Standard Operating Procedure
Organic Extraction Sample Chain of Custody,
Archival, and Disposal

1.0 Scope and Application

1.1 This document details the procedure for maintaining sample chain of custody in ARI's Organic Extraction Laboratory, and describes the procedures for archival and disposal of those samples.

2.0 Definitions

2.1 Archive times – Period of time ARI holds samples in archive prior to disposal. Archive times begin the day sample data is delivered to ARI's client. Archive times are: 60 days for waters, 90 days for soils/solids, and 1 year for frozen sediment and soil. Longer or shorter holding times may be specified by ARI's client.

2.2 Flags – Samples that requiring special handling (chain of custody, archival, or disposal) are flagged by sample receiving by using color coded stickers. Red labels are used to designate samples known or presumed to be hazardous and yellow labels designate samples to be shared by more than one analytical group within ARI.

2.3 Regulated Soils - All soils samples originating outside the United States and designated areas within the US are regulated by the United States Department of Agriculture (USDA) and must be handled separately from other samples. Procedures for handling USDA regulated soil are available in ARI SOP 0056S. Regulated soils are designated with an autoclave flag on the Organic Extraction Summary Sheet.

3.0 Equipment

3.1 Autoclave at 60lbs pressure and 123°C.
3.2 Glass crusher.

4.0 Documentation

4.1 Organic Extractions Walk-in Cooler #5 Log Book (Form 0008F)
4.2 EPA Sample Walk-in Log (0032F)
4.3 Organic Extraction Summary sheet
4.4 Outside Walk-in Cooler Log (Extraction Summary Sheet)
4.5 GC/MS Extractions Sample Extract Log (3032F)
4.6 GC Extractions Sample Extract Log (3033F)
4.7 GC/MS Extract Refrigerator Log (7004F)
4.8 GC Extract Refrigerator Log (4057F)
4.9 TPH Refrigerator Log (4112F)
4.10 GC/MS ABN Extract Archive Log (7006F)
4.11 GC Sample Archive Log (4086F)
4.12 TPH Extract Archival/Disposal Log (4113F)
4.13 Sample Sign Out Log (3059F)

5.0 In-house Modifications to Referenced Method

5.1 N/A

6.0 Procedure

6.1 Chain of Custody from Sample Receiving to Organic Extractions Walk-in Cooler #5 Log Book (0008F). See SOPs 1011S (Internal Chain of Custody – Semivolatiles Section 6.0-6.1.3) and 1009S (Internal Chain of Custody – GC section 6.2-6.3.2.2).

6.2 Extraction Laboratory Chain of Custody:

6.2.1 Locate samples needed for preparation or analysis in Organic Extractions Walk-in Cooler #5 Log Book (0008F).

6.2.2 Find numbered bin inside walk-in cooler and remove needed samples.

6.2.3 Record in Logbook which samples were taken, initial and date removal. Sample of Custody is now with Extraction Laboratory Technician.

6.2.4 Extract and process samples.

6.2.5 If samples require initial screening, multiple extractions, or are shared with other labs, return samples to same bin, re-initial and re-date return, and note which method of preparation was performed. Custody is returned to Cooler #5. This may occur more than once, again re-initial and re-date return, and note which method of preparation was performed.

6.2.6 Share samples will be monitored by archive sample custodian bi-weekly, or when all labs have completed sample preparation or analysis. Share samples will then be archived. Share samples are flagged with a yellow dot sticker.

6.2.7 If all methods of sample preparation have been finished, archive samples. Custody is relinquished to sample custodian. (See section 6.3)

6.2.8 For Custody of extracts. See SOP #'s 1011S (Internal Chain of Custody – Semivolatiles section 6.4-6.4.2) & 1009S (Internal Chain of Custody – GC section 6.2.2.-6.3.2.2.).

6.3 Sample Archive (Sample Custodian)

6.3.1 Make a copy of the Organic Extraction Summary sheet. This will be used to record sample archive location in archive book.

6.3.2 Place the residual samples in the Outside Walk-in cooler. Put samples in bin designated by Sample Custodian. All bins have a specific numbers and letter prefixes. Soil and solid bins have an "S" and water sample bins will have a "W". Freezer samples (primarily sediments) are placed in a bin in the Inside walk-in and are archived by the Sample Custodian following the same protocol. Freezer bins are labeled with an "F".

6.3.3 Enter on Organic Extraction Summary sheet bin number, initial and date entry. Next to each sample ID letter, note how many jars/bottles are being archived (e.g. +1). File sheet in sample archive logbook in alpha-numeric order. Chain of custody is resumed to the Outside Walk-in cooler until discard. Samples to be frozen must be placed in zip lock freezer bags to prevent injury and cross-contamination if sample jars break. Enter archive in LIMS by filling Archive Worklist and distributing.

6.3.4 Empty sample jars/bottles after preparation can be logged as "consumed". Enter number of consumed samples on LIMS sample sheet. File sheet in sample archive logbook in numerical order. Remove label and throw away jar/bottle. Record consumed samples on LIMS by filling Consumed Samples Worklist and distributing.

6.3.5 To remove samples from archive for re-extract or re-analysis, locate Organic Extraction Summary sheet in sample archive logbook which contains location of samples and bin number. Pull samples needed from walk-in. Record which samples are removed in the Sample Sign Out Logbook. Initial and date removal. When sample preparation is finished return samples to same archive bin and re-initial and re-date return in Sample Sign Out Logbook.

6.3.6 To monitor bins for discard, process an Archive Report for bins in LIMS. Find latest discard date on report and highlight. This will be the latest day that bin can be discarded to meet archive hold times for all samples in that bin. Keep sheet in designated bin.

6.4 Sample Discard

6.4.1 When archive hold times expire, samples are ready for discard. Remove bin from walk-in and cross check samples in bin to Archive Report. All samples in bin must have a discard date earlier or the day of to be discarded.

6.4.2 Non-hazardous waters can be poured down the archive room drain with running water. Non-hazardous soils and sediments can remain in jars. Remove labels from all sample containers and discard into dumpster.

6.4.3 Record discard on Organic Extraction Summary sheet, initial, and date disposal.

6.4.4 Do not discard samples flagged with red dot sticker. If no analysis report is processed, analysis has found samples not hazardous and except for oils, samples can be disposed of normally. If a hazard report is processed by LIMS, analysis has found samples to be above limit acceptable for normal discard. Put report and hazardous samples in hazardous closet to be treated and disposed of by Hazardous Waste Manager. **Put all oils, hazardous or not, in hazardous closet to be disposed of by Hazardous Waste Manager.**

6.4.5 Samples flagged for autoclave must be processed before discard.

6.4.5.1 Remove lids from jars and stack jars on tray in one or two layers.

6.4.5.2 Remove steam from autoclave by turning dial to O symbol. Fill autoclave with water up to the grove by turning dial to "Fill Water".

6.4.5.3 Tape jars with autoclave tape and load tray in cylinder. Close autoclave door and turn lever down to lock. Turn power on and set timer for one hour. Autoclave will run to 60lbs of pressure at 123°C.

6.4.5.4 Use of glass crusher to discard sample jars is optional.

7.0 Review

7.1 Sample custodian reviews all logbooks and generates new binders when full.

7.2 Organic Extraction Supervisor or Lead reviews all logbooks weekly.

8.0 Quality Control

8.1 N/A

9.0 Corrective Actions

9.1 Walk-in refrigerators and freezers not holding temperatures are not in compliance with QA recommendations and need temperature adjustments or service.

10.0 Miscellaneous Notes and Precautions

10.1 SafeKnit or Armidillo gloves must be worn when removing labels from sample jars to prevent injury.

10.2 Protective lab gear must be worn at all times; this includes lab. Coat, gloves, and goggles.

10.3 Extra caution should be taken when using glass crusher.

11.0 References

N/A

12.0 Appendices

N/A

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333 INSIDE WALK-IN LOG

Date Logged in	Log in Initials	Client Name	ARI Job No.	Sample ID	Matrix		Bin No.	Other Location	Removed By Date/ID	Returned By/Date/ID	For Which Laboratory(s)?
					Soil	H2O					

0008F

Rev.5
04/98

ORGANIC EXTRACTIONS SUMMARY 05/16/97

Inquiry Number: 1

Contact: Kissel, Christine
Client: Landau Associates, Inc.

Logged by: Mary Lou
Analysis Requested: 05/16/97

ARI Job NO: 5557
PC: Bryan
RTS: 05/16/97
Data Due: 05/27/97

Project No: 32028
Todd Shipyards
Harbor Island, WA

Special Instructions: None Required

Analytical Protocol: In-house
Deliverables: Level (II)

FM GK BOA Date 5/19/97

4 Samples

	LIMS ID	ARI ID	Client ID	Sampled	Matrix	8015 HLID
1.	97-7709 Okay	5557E	FW-2	05/16/97 11:45	Product	X
2.	97-7710 Okay	5557F	FW-3	05/16/97 11:30	Product	X
3.	97-7721 Okay	5557G	FW-4	05/16/97 12:00	Product	X
4.	97-7722 Okay	5557H	FW-5	05/16/97 10:30	Product	X

** HOLD SAMPLE

ORGANIC EXTRACTIONS SUMMARY 09/14/98

ARI Job NO: Y554
PC: Jeff
VISR: 09/14/98
Data Due: 10/17/98

Inquiry Number: 169

Logged by: Amy
Analysis Requested: 09/14/98

Contact: Carpenter, Kathryn
Client: Woodward Clyde

Project No:
Tongue Point
Tongue Point

Special Instructions: NO SAMPLE SHARING

Analytical Protocol: PSDDA
Deliverables: WWC/IRPIMS edds

PM OK _____ Date _____

5 Samples

**	LIMS ID	ARI ID	Client ID		Sampled	Matrix	PSDDA SVOA	PSDDA Pest/PCB	WIPHO Other		
F 1.	98-18836 Okay	Y554A	TS01	75.22	09/11/98 9:20	Tissue	X homogenize and split for dioxin	X	X	1 1	Special florisil setup on ASE
F 2.	98-18837 Okay	Y554B	TS02	55.04	09/11/98 8:54	Tissue	X homogenize and split for dioxin	X	X	1 1	Special florisil setup on ASE
F 3.	98-18838 Okay	Y554C	TS03 MS/MSD	81.05	09/11/98 11:20	Tissue	X homogenize and split for dioxin	X	X	1 1	Special florisil setup on ASE
F 4.	98-18839 Okay	Y554D	TS04	77.38	09/11/98 11:00	Tissue	X homogenize and split for dioxin	X	X	1 1	Special florisil setup on ASE
F 5.	98-18840 Okay	Y554E	TS05		09/11/98 10:30	Tissue	X homogenize and split for dioxin	X	X	1 1	Special florisil setup on ASE

F2-569 9/16/98 9.F.

Frozen 9/16/98 MT

98-19210 F

** H=HOLD SAMPLE; F=FREEZE SAMPLE; A=AUTOCLAVE BEFORE DISPOSAL

ORGANIC EXTRACTIONS SUMMARY 11/24/98

ARI Job NO: Z311
PC: Jennifer
VTSR: 11/24/98
Data Due: 12/05/98

Inquiry Number: NONE

Logged by: Carrie
Analysis Requested: 11/24/98

Project No: A98143

Contact: Neland, Dale
Client: Boeing Defense and Space Group

Special Instructions: No

Analytical Protocol: In-house
Deliverables:

PM OK JMB Date 11/24/98

1 Sample

**	LIMS ID	ARI ID	Client ID	Sampled	Matrix	Total Solids	WIPHD: Other
1.	98-24310	Z311A	46358	11/23/98 13:45	Soil	X	X
	Okay						

Archived 5-53 11/24/98

Discarded 3/18/99 J.F.

** H=HOLD SAMPLE; F=FREEZE SAMPLE; A=AUTOCLAVE BEFORE DISPOSAL

ORGANIC EXTRACTIONS SUMMARY 03/19/99

Inquiry Number: DONE

Contact: Herrenkohl, Mark

Client: Science Applications Intl.

Special Instructions: Yes

Logged by: Zach

Analysis Requested: 03/19/99

Analytical Protocol: PDBDX- 1, 4 and C

Deliverables: Full

ARI Job No: AB95

CC: Jennifer

VISR: 03/19/99

Data Due: 04/02/99

Project No: 01 0440-04 1214-400

EW Chem Rob

PM OK: MK Date: 3/19/99

1 Sample

**	LIMS ID	ARI ID	Client ID	Sampled	Matrix	8270 SVOA	8081 Pest/PCB
1.	99 3177	AB95A	980343	03/18/99 12:05	Water	X	X
	Okay				Organics Preserved With HCl		

Consumed

Eg 3/23/99

** H-HOLD SAMPLE; F-FREEZE SAMPLE; A-AUTOCLAVE BEFORE DISPOSAL

ORGANIC EXTRACTIONS SUMMARY 03/19/99

ARI Job No: AB98

Inquiry Number: NONE

PC: Mark

Logged by: Carrie

VTSR: 03/19/99

Analysis Requested: 03/19/99

Data Due: 03/30/99

Contact: Stremcha, Dan

Client: ThermoRetec

Project No: 1 3423

TPC GM Sampling

Special Instructions: 4

Analytical Protocol: In house

Deliverables:

PM OK [illegible] Date 3/19/99

3 Samples

**	LIMS ID	ARI ID	Client ID	Sampled	Matrix	8040 PCP
1.	99-3394	AB98A	25-B	03/18/99 9:42	Water	X
	Okay					
2.	99-3395	AB98B	14-A	03/18/99 11:10	Water	X Product in water sample
	Okay					
3.	99-3397	AB98D	GM-8	03/18/99 10:35	Water	X
	Okay					

** H=HOLD SAMPLE; F=FREEZE SAMPLE; A=AUTOCLAVE BEFORE DISPOSAL

[illegible] Services Summary Report

Inquiry Number: [illegible]

Contact: Kane, Bill

Client: EcoCompliance Corporation

Logged by: Amy

Analysis Requested: 01/18/99

ARI Job No: [illegible]33

PC: Jen

VISR: 01/18/99

Data Due: 01/19/99

Project No: 98-12001

Special Instructions:

for 01/19/99

6 Samples

Analytical Protocol: In-house

Deliverables:

PM OK Date

**	LIMS ID	ARI ID	Client ID	Sampled	Matrix	Total Solids	8015 MCID
1.	99-577	77358	1	01/18/99	Soil	X	X
	Okay		1				
2.	99-578	77359	2	01/18/99	Soil	X	X
	Okay		1				
3.	99-579	77360	3	01/18/99	Soil	X	X
	Okay		1				
4.	99-580	77361	4	01/18/99	Soil	X	X
	Okay		1				
5.	99-581	77362	5	01/18/99	Soil	X	X
	Okay		1				
6.	99-582	77363	6	01/18/99	Soil	X	X
	Okay		1				

** H=HOLD SAMPLE; F=FREEZE SAMPLE; A=AUTOCLAVE BEFORE DISPOSAL

ORGANIC SCHEDULING SUMMARY 01/12/99

Inquiry Number: NONE

Contact: Hilmont, Robert

Client: Fedrec Environmental

Special Instructions: Yes

J Samples

Logged by: Amy

Analysis Requested: 01/12/99

Analytical Protocol: PSDUA

Deliverables:

AKI Job NO: Z679

PC: Jeff

VTSR: 01/12/99

Data Due: 01/26/99

Project No: 403-001

SMS

PM OK [illegible] Date 1/14/99

	CLIENT ID	AKI ID	CLIENT ID	Sampled	Matrix	Total Solids	Ext pH	PSDUA SVOA	PSDUA Pest/PCB	FREEZER F1-588 1/18/99
F 1.	99-588 OKAY	Z679A	403001001	01/12/99 12:15	Sediment	X	X	X	X	+1
F 2.	99-589 OKAY	Z679B	403001002	01/12/99 12:45	Sediment	X	X	X	X	+1
F 3.	99-590 OKAY	Z679C	403001003	01/12/99 13:15	Sediment	X	X	X	X	+1

** A=HOLD SAMPLE; F=FREEZE SAMPLE; A=AUTOCLAVE BEFORE DISPOSAL

Archive Report - Frozen Samples

Samples in Archive Section F1-597

LIMS ID	ARI ID	Number of Bottles	Discard Date	Autoclave
99-6144	AG08M	1	08/20/99	
99-6145	AG09A	1	07/30/99	
99-6146	AG09B	1	07/30/99	
99-6147	AG09C	1	07/30/99	
99-6148	AG09D	1	07/30/99	
99-6149	AG09E	1	07/30/99	
99-6151	AG09G	1	07/30/99	
99-6152	AG09H	1	07/30/99	
99-6153	AG09I	1	07/30/99	
99-6154	AG09J	1	07/30/99	
99-6450	AG70A	1	07/30/99	
99-6451	AG70B	1	07/30/99	
99-6570	AG85A	2	07/25/99	
99-6571	AG85B	1	07/25/99	
99-6572	AG85C	2	07/25/99	
99-6605	AG94A	2	07/28/99	
99-6606	AG94B	2	07/28/99	
99-6607	AG94C	2	07/28/99	
99-6608	AG94D	2	07/28/99	
99-6609	AG94E	2	07/28/99	
99-6610	AG94F	2	07/28/99	
99-6611	AG94G	2	07/28/99	
99-6612	AG94H	2	07/28/99	
99-6613	AG94I	2	07/28/99	
99-6614	AG94J	2	07/28/99	
99-6615	AG94K	1	07/28/99	
99-6616	AG94L	2	07/28/99	
99-6617	AG94M	2	07/28/99	
99-6618	AG94N	2	07/28/99	
99-6619	AG94O	2	07/28/99	
99-6620	AG94P	2	07/28/99	
99-6621	AG94Q	2	07/28/99	
99-6622	AG94R	2	07/28/99	
99-6623	AG94S	3	07/28/99	
99-6624	AG94T	3	07/28/99	
99-6625	AG94U	3	07/28/99	
99-6626	AG94V	3	07/28/99	
99-6627	AG94W	3	07/28/99	
99-6628	AG94X	3	07/28/99	
99-6629	AG94Y	0	07/28/99	
99-3587	AC27E	2	06/05/99	

FACSIMILE TRANSMITTAL

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Addressee: Mark Herrenkohl Fax No: 425-487-1491

Company: Science Applications Intl. Phone No: 425-485-5800

From: Jan Felkins
for Jennifer Baier, Project Manager Date: 12/04/98

Project Reference: EHOU 1998 Remedial Programs
Eagle Harbor

The following samples that are stored in our freezer/cooler are due to be discarded. Please contact ARI if the status has changed regarding these samples, otherwise they will be discarded on 12/24/98.

SAMPLE ID	MATRIX	ARI LIMS#	ARI ID
980309002	Sediment	98-4506	V654A
980309003	Sediment	98-4507	V654B
980309005	Sediment	98-4508	V654C

Thank you for your time.

Jan Felkins
ANALYTICAL RESOURCES, INC.

FI-548 12/14/98
throw them all out!

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GCMS SAMPLE EXTRACT LOG

Date	ARI Sample ID	Final Volume	Initials

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GC ECD/FID SAMPLE EXTRACT LOG

Date	ARI Sample ID	Final Volume	Initials

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GC/MS EXTRACT REFRIGERATOR LOG

ARI Job Number	Analysis	Number of Extracts	(Extractions) Date In	Initials	(ABNs) Date Out	Initials

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GC EXTRACT REFRIGERATOR LOG

ARI Job Number	Analysis	Number of Extracts	Logged In By	Date	Received By	Date

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TPH EXTRACT REFRIGERATOR LOG

ARI Job Number	Analysis	Number of Extracts	Date In/Initials	Date Out/Initials

4112F

Rev. 1
3/11/97

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GC/MS ABN EXTRACT ARCHIVE LOG

Archive Box No.: _____ Analysis Dates: _____

Instrument ID: _____ Date Archived/Analyst: _____

Date Discarded/Analyst: _____

ARI Job No.	Comments (If necessary)

Page_____

ANALYTICAL
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GC SAMPLE EXTRACT ARCHIVE LOG

Instrument	Run Dates	Initially Logged in		Final Archive (✓)	Disposal	
		Initials	Date		Initials	Date

TPH EXTRACT ARCHIVE/DISPOSAL LOG

Instrument/ Rack Number	Analysis Dates	Archive Box Number	Archive Date/Initials	Discard Date/Initials	Comments

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Sample Signout

JOB NO.	LIST	BIN NO.	INITIALS/DATE	RETURNED	REBINNED

7/13/99

Archive 333 Building Report

Samples in Archive Bin W-197

LIMS ID	ARI ID	Number of Bottles	Discard Date	Autoclave
99-3257	AB73A	1	05/30/99	
99-5031	AE43A	1	07/03/99	
99-5032	AE43B	1	07/03/99	
99-5196	AE66A	1	06/29/99	
99-5197	AE66B	1	06/29/99	
99-5198	AE67A	1	06/29/99	
99-5199	AE67B	1	06/29/99	
99-5200	AE67C	1	06/29/99	
99-5262	AE80H	1	07/13/99	
99-5291	AE82D	1	07/02/99	
99-5274	AE80T	1	07/13/99	

Discarded 7/12/00 G.F.

AD44-

#544

Archive Report - Frozen Samples

Samples in Archive Section F2-578

LIMS ID	ARI ID	Number of Bottles	Discard Date	Autoclave
98-21661	Y931K	1	01/01/99	
98-21662	Y931L	1	01/01/99	
98-21663	Y931M	1	01/01/99	
98-21664	Y931N	1	01/01/99	
98-21665	Y931O	1	01/01/99	
98-21666	Y931P	1	01/01/99	
98-21667	Y931Q	1	01/01/99	
98-21668	Y931R	1	01/01/99	
98-21669	Y931S	1	01/01/99	
98-21670	Y931T	1	01/01/99	
98-22015	Y976A	0	01/20/99	
98-22016	Y976B	0	01/20/99	
98-22017	Y976C	0	01/20/99	
98-22018	Y976D	0	01/20/99	
98-22019	Y976E	0	01/20/99	
98-22020	Y976F	0	01/20/99	
98-22021	Y976G	0	01/20/99	
98-22022	Y976H	0	01/20/99	
98-22023	Y976I	0	01/20/99	
98-22024	Y976J	0	01/20/99	
98-22025	Y976K	0	01/20/99	
98-22026	Y976L	0	[illegible]	

9/7/99

Archive 333 Building Report

Samples in Archive Bin S-109

LIMS ID	ARI ID	Number of Bottles	Discard Date	Autoclave
99-7251	AH91A	1	09/05/99	
99-7252	AH91B	1	09/05/99	
99-7253	AH91C	1	09/05/99	
99-7254	AH91D	1	09/05/99	
99-7255	AH91E	1	09/05/99	
99-7274	AH95A	1	09/05/99	
99-7275	AH95B	1	09/05/99	
99-7276	AH95C	1	09/05/99	
99-7277	AH95D	1	09/05/99	
99-7278	AH95E	1	09/05/99	
99-7279	AH95F	1	09/05/99	
99-7280	AH95G	1	09/05/99	
99-7281	AH95H	1	09/05/99	
99-7404	AI21A	1	09/07/99	
99-7405	AI21B	1	09/07/99	
99-7406	AI21C	1	09/07/99	
99-7558	AI46A	1	09/07/99	
99-7559	AI46B	1	09/07/99	
99-7560	AI46C	1	09/07/99	
99-7562	AI46E	1	09/07/99	
99-7561	AI46D	1	09/07/99	

Analytical Resources, Incorporated
Analytical Chemists and Consultants

Standard Operating Procedure

Total Solids

SOP 1023S
Revision 000

Revision Date: 12/16/09
Effective Date: 12/21/09

Prepared By:

Dave Mitchell

Approvals:

David R Mitchell
Laboratory / Section Manager

David R Mitchell
Quality Assurance

4611 South 134th Place, Suite 100 • Tukwila WA 98168 • **206-695-6200** • **206-695-6201** fax

Analytical Resources, Incorporated
Analytical Chemists and Consultants

Annual Review

SOP Number: 1023S

Title: Total Solids for Metals & Organics Samples

The ARI employee named below certifies that this SOP is accurate, complete and requires no revisions

Name	Reviewer's Signature	Date

Standard Operating Procedure: Total Solids

1. Scope and Application

1.1. Analyte concentrations in solid samples are reported on a dry weight basis unless project documentation indicates otherwise. A "Total Solids" (TS) percentage must be determined to correlate analyte concentration with dry weight.

1.1.1. TS are determined by drying a portion of sample at elevated temperature for 12 to 24 hours and calculating percent total solids as outlined below.

1.1.2. TS must be determined immediately before, or immediately after weighting samples for the preparation process.

1.1.3. TS must be determined separately for each sample container and may be used for all analyzes from that container.

1.1.4. When TS must be determined in less than 12 hours, a microwave procedure may be used. The procedure is posted on the microwave oven in the Organics Extraction Laboratory. The microwave procedure is only for "rush" or emergency situations. Do not use the microwave procedure for routine samples.

1.2. The procedure in this document is used to determine Total Solids (TS) for reporting of data from solid samples (soil, sediment, etc.) on a "dry weight" basis. This SOP is used by the Organics Extraction, Volatile Organics, Metals and GeoTech Laboratories. When TS is specifically requested the TS is determine in ARI's Conventionals Laboratory using SOP 639S "Solids".

2. Summary of the Procedure

2.1. A weighed portion of sample is oven dried and the percent difference between the wet and dry sample calculated.

3. Definitions

3.1. Total Solids. (TS). The total weight of all solid materials (dissolved and particulate) contained per unit weight or volume of sample. For soils or sediments, it is the weight remaining after drying a known weight of sample, usually expressed as a percent.

4. Interferences

4.1. Heterogeneous samples: Samples must be appropriately mixed prior to withdrawing the aliquot for analysis. Anything, including non-representative artifacts, that will interfere with the ability to take a representative sub-sample must be documented on the analysts "Green sheet"

and discussed with the Project Manager prior to beginning the analysis. Do not exclude any materials from the sample without discussion with the Project Manager.

4.2. Sample residues for weighing must be at room temperature to avoid convective interference in the balance chamber.

4.3. Constant dry weight is assumed to be reached after the sample has dried for a period of 12 to 24 hours. In the event that the sample has not been dried for the minimum 12 hours, or upon specific request from the client, attainment of constant dry weight must be documented.

5. Safety

5.1. Environmental Samples may contain hazardous materials; treat them as potential health hazards.

5.2. The drying oven must be contained in a hood or vented to prevent potentially toxic fumes from a heavily contaminated sample from entering the laboratory.

5.3. Always wear safety glasses and hand protection when working with hot samples.

6. Equipment and Supplies

6.1. ALUMINUM WEIGHING DISHES.

6.2. DRYING OVENS Gravity convected, adjustable to maintain 104 ± 2 ℃. Gravity convection is preferred for the drying of samples since mechanically convected ovens can create drafts that could displace dried materials from their containers. Mechanically convected ovens should only be used for drying of equipment and materials used for the analysis.

6.3. Analytical balance. The balance used must be capable of measurement to 3 places. Balance verification is accomplished using ASTM E 617-97 traceable weights. The calibration weight must return a value within 1 mg or 10% of its known weight, whichever is less.

6.4. Weights for balance verification. Troemner Calibration weights, ASTM E 617-97 traceable (equivalent to old NBS Class S and S-1 standards). Each weight set is certified every 5 years by an outside metrologist. A certificate documenting the “true” weight of each calibration weight accompanies each set.

7. Reagents and Standards

7.1. Not Applicable

8. Sample Collection, Preservation, Shipment and Storage

8.1. Sub-sampling is a critical component of solids analysis. In order to obtain accurate and reproducible results, be certain that the sample is well mixed and that the particulates are uniformly distributed throughout the sample prior to withdrawing the aliquot.

9. Quality Control

9.1. Balance Verification. Each analytical balance is verified against ASTM E 617-97 traceable (NTIS equivalent to older NBS Class S and S-1 standards) weights at the beginning each weighing cycle.

9.2. Record which analytical balance and oven are used for the TS determination on the bench sheet

9.3. Verify that the oven used is at 104 ±2°C.

9.4. Record the Oven ID on the TS bench sheet.

10. Calibration and Standardization

10.1. The accuracy of each analytical balance is verified daily against ASTM E 617-97 traceable (NTIS equivalent to old NBS Class S and S-1 standards) weights covering the range of determination.

10.2. The balance verification is documented in a balance specific logbook.

11. Procedure

11.1. Analyte concentrations in solid samples are reported on a dry weight basis unless project documentation indicates otherwise. A “Total Solids” (TS) percentage must be determined to correlate analyte concentration with dry weight.

11.1.1. TS are determined by drying a portion of sample at 104 ± 2 ℃ for 12 to 24 hours and calculating percent total solids as outlined below.

11.1.2. TS must be determined immediately before, or immediately after weighting samples for the preparation process.

11.1.3. TS must be determined separately for each sample container and may be used for all analyzes from that container.

11.1.4. When TS must be determined in less than 12 hours, a microwave procedure may be used. The procedure is posted on the microwave oven in the Organics Extraction Laboratory. The microwave procedure is only for “rush” or emergency situations. Do not use the microwave procedure for routine samples.

11.1.5. TS determined using this procedure are used to calculate analytes values reported on a dry weight basis. Total Solids as a reported analyte is performed in ARI’s Conventionals Laboratory following SOP 639S “Solids”.

11.2. Obtain a copy of Total Solids Bench Sheet (Form 5050F) and enter the header information, analyst name, date, oven ID, balance ID and ARI sample identification(s).

11.3. Verify that the balance calibration has been documented for the day using bracketing weights. If not, verify and document balance accuracy before proceeding using the procedure described in ARI SOP 1003S.

11.4. Zero the balance.

11.5. Obtain an empty aluminum weighing pan write the sample number on the pan with permanent marker.

11.6. Weigh the empty pan and record its weight to the nearest 0.01 gram in the "Tare Weight" column of the TS bench sheet. Record all digits displayed by that balance. Do not round the numbers.

11.7. Make sure the sample is homogenous by thorough mixing.

11.7.1. Decant any water layer from samples to be extracted for semivolatile organics (SVOA) extractions prior to homogenization.

11.7.2. Samples for volatile organics analysis (VOA) must not be homogenized in this manner. Ideally a second container which may be homogenized will be collected for VOA TS determination. When the second container is not available the TS determined in another lab section may be used. In this case, the source of the TS data must be clearly identified on the TS bench sheet.

11.8. Weigh 10 ± 5 grams of wet sample into the weighing pan. Record the exact weight to the nearest 0.01 gram in the "Tare + Sample Wet" column of the log form. Using less sample is permissible only when less than 5 g of sample are available (Note Section 11.11.2 below)

11.8.1. The minimum sample size for GeoTech samples is defined in accordance with Table 1 of ASTM D2216 or following requirements of the ASTM method applied to the sample.

11.9. Verify and record oven temperature on the TS bench sheet.

11.10. Place the weighing pans in the oven, record the time, and dry at an oven temperature of 102-106℃ overnight (12-24 hours) but no longer tha n 24 hours. If dried for less than 12 hours, it must be documented that constant weight was attained.

11.10.1. To demonstrate that constant weight has been attained, data must be recorded for a minimum of two repetitive weigh/dry/weigh cycles with a minimum of 1-hour drying time in each cycle.

11.10.2. Constant weight would be defined as a loss in weight of no greater than 0.01g between the initial and final weightings of the last cycle.

11.11. Remove pans from the oven and allow them to equilibrate to ambient temperature for no more than one hour.

11.12. Record analyst name, oven temperature, date, and the time of removal on the TS bench sheet.

11.13. Verify the balance accuracy and record the results in the balance specific logbook.

11.14. When cool, weigh and record the exact weight to the nearest 0.01 grams in the "Tare + Sample Dry" column of the log form.

11.14.1. When dry samples cannot be weighed within 60 minutes of removal from the oven, they must be placed in a desiccator to prevent adsorption of moisture from the atmosphere.

11.14.2. When wet samples less than 0.5 g are used, the dry sample must be cooled in a desiccator.

11.15. Data Entry Procedures:

11.15.1. Organics Extraction Laboratory: Enter in LIMS using worklist

11.15.2. Metals Preparation Lab: Data is entered into dBase

11.15.3. VOA laboratory: Enter in LIMS using worklist

11.15.4. GeoTech laboratory: Enter in LIMS using worklist

11.16. Place the Total Solids Benchsheet in the appropriate project folder.

12. Review

12.1. Supervisor reviews Service Request, and assigns analysis and communicates any special instructions to the analyst as appropriate.

12.2. Analyst reviews Service Request and any special instructions, obtains the appropriate bench sheet and proceeds with the analysis. A handwritten bench sheet is generated and the data are then entered into the computer for data reduction.

12.3. Creation of Worklists. The final computer generated result is placed into the method folder in chronological sequence and a copy is placed into the job folder. The data undergo supervisor review and the analysts then enters the data into LIMS to create the worklist.

12.4. Distribution of worklists. The supervisor reviews the LIMS input, distributes acceptable data and returns the analysis package to analyst for placement in the job folder.

12.5. The completed analysis package is then reviewed by a QA Reviewer and the final report printed.

12.6. The final report is then again reviewed for accuracy and completeness, signed by the QA reviewer and delivered to the Project Manager for final disposition to the client.

13. Data Analysis and Calculations

13.1. TS is a gravimetric determination in which the dry weight of solid residue is expressed relative to the volume or mass of sample used to obtain that residue. Specific calculations are as follows:

13.1.1. Percent solids for reporting chemistry data is calculated by the LIMS as follows:

$$\% \ Solids = \left[\frac{(Tare + Sample\ Dry) - Tare}{(Tare + Sample\ Wet) - Tare} \right] * 100$$

13.1.2. Moisture Content for reporting GeoTech data is calculated as follows:

$$MoistureContent = \left[\frac{(Tare + Sample\ Wet) - Tare + Sample\ Dry}{(Tare + Sample\ Dry) - Tare} \right] * 100$$

14. Method Performance

14.1. Approximately 10 grams of sample are typically taken for analysis. While the balance reports to 0.001 g, a sensitivity of 0.01 g is routinely used to report data. This yields a method sensitivity of 0.1% for TS.

15. Pollution Prevention

15.1. Not Applicable

16. Data Assessment and Acceptance Criteria for Quality Control Measures

16.1. Samples dried less than 12 hours

16.1.1. Demonstrate constant weight as described in Sections 11.13.1 and 11.13.2.

16.2. When samples have dried more than 24 hours

16.2.1. Repeat the TS determination on an additional sample aliquot.

16.2.2. When sample is not available or a repeat determination is not practical, document the excessive weighing time on the TS bench sheet in the comments section.

16.3. When the balance is out of control

16.3.1. Use the corrective action procedures in ARI SOP 1003S to bring balance back in control.

16.3.2. When balance in out of calibration or non functional use a similar balance from another lab section.

16.4. Oven temperature s out of control

16.4.1. Adjust oven temperature and allow the temperature to equilibrate to required range.

17. Corrective Actions for Out of Control Events

17.1. Not Applicable

18. Contingencies for Handling Out-of-Control or Unacceptable Data

18.1. Not Applicable

19. Waste Management

19.1. Samples may contain RCRA hazardous constituents and these are screened in ARI's LIMS system. Samples which LIMS identified as containing one or more hazardous constituents will be segregated into identified waste profiles and disposed of through our RCRA TSD (Treatment, Storage Disposal facility). Samples not having identified hazardous constituents will disposed as routine garbage.

20. Method References

20.1. USEPA Contract laboratory Program, Statement of Work for Inorganic Analysis. ILM05.3 March 2004. Exhibit D.

Analytical Resources, Incorporated
Analytical Chemists and Consultants

Standard Operating Procedure

Extraction of Soil/Sediment Samples Using Sonication or Microwave (MARS) (EPA Methods 3550B and 3546)

SOP 3304S
Revision 001
Revision Date: 7/29/10
Effective Date: 7/29/10

Prepared By:

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Approvals:

Laboratory / Section Manager

Quality Assurance

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Annual Review

SOP Number:	3311S
Title:	Extraction of Soil/Sediment Samples Using Sonication or Microwave (MARS) (EPA Methods 3550B and 3546)

The ARI employee named below certifies that this SOP is accurate, complete and requires no revisions

Name	Reviewer's Signature	Date

Standard Operating Procedure 3304S - Extraction of Soil/Sediment Samples Using Sonication or Microwave (MARS) (EPA Methods 3550B and 3546)

1. Scope and Application

1.1. This document describes the procedures used at ARI to perform solvent extraction of soil, sediment, sludge, and solid waste environmental samples using sonication or microwave radiation (MARS) for semi-volatile organics analyses. Procedures in this document are based on EPA Methods 3550B and 3546. The procedures described are used for GC and GC-MS analyses listed in Appendix 20.1. Sample weights, extraction solvents and extract final volumes vary depending on client or project specific requirements.

1.2. This document provides an outline of procedures and/or tasks for each analysis listed in Appendix 20.1. The specific requirements for each analysis are provided in a Bench Sheet. A unique Bench Sheet is available for each analysis. Bench Sheets define the extraction protocol and are used to document the extraction procedure. Appendix 20.3 includes all current Bench Sheets applicable to this SOP.

1.3. An outline or brief summary of the tasks required to complete the analyses in Appendix 20.1 are provided in this document. Step by step instructions are documented in the task specific "Task Instructions" (TI 008-TI 028) provided in Appendix 20.2. Training of Organic Extractions Analysts is based on the Analytical Bench Sheets and Task Instructions. Analysts proficiency is documented for each Task Instruction.

2. Summary of the Procedure

2.1. Modified extraction levels may be required as the GC Supervisor or Organic Extractions Supervisor determines. Such decisions will be based on the project detection limit requirements. Appropriate surrogate and spike levels should be added based on anticipated Final Effective Volume, (FEV).

2.2. A specified weight of soil/sediment sample is weighed into an appropriate sized glass beaker and the pH is adjusted as necessary.

2.3. Samples for **sonication** are spiked with analysis specific surrogate standards and covered with a specified organic solvent and an appropriate amount of anhydrous sodium sulfate is mixed in to dry the samples. The samples are extracted three times with specified solvent and collected in an appropriate size glass Erlenmeyer flask.

2.4. The extracts are concentrated to a designated final volume using a Kuderna-Danish (KD) concentrator apparatus on a water bath and/or a turbo-vap system.

2.5. Extracts may be subjected to method specific solvent exchange, cleanup or separation procedures before they are relinquished to the GC laboratory for analysis.

2.6. Samples for **microwave** are mixed with anhydrous sodium sulfate to dry the samples and transferred to the extraction vessel. 20-40 mL of the appropriate extraction solvent is added. The appropriate spiking solutions are added and the vessel is sealed to a precise torque with the MARS capping apparatus.

2.7. The vessel is transferred to the MARS carousel and heated to 105° C for 20 minutes.

2.8. After cooling, the solvent is transferred to a turbo vap tube. The soil/sediment in the vessel is rinsed two times with a suitable solvent and added to the turbo vap tube and concentrated by turbo vap to a suitable volume for analysis or further clean-up steps.

2.9. Extracts may be subjected to method specific solvent exchange, cleanup or separation procedures before they are relinquished to the GC laboratory for analysis.

2.10. Project specified Quality Assurance samples are prepared and extracted using identical procedures.

3. Definitions

3.1. Ultrasonic Extraction (Sonication) uses ultrasonic energy from the disrupter. Very active mixing of the sample and solvent occurs when the ultrasonic pulse is activated. Three extractions are performed with the appropriate solvent and collected for each sample.

3.2. Microwave Extraction (Microwave assisted reaction system or MARS) – An extraction apparatus utilizing microwave radiation to produce elevated temperature and pressure in a closed vessel system to effect rapid extraction of organic compounds into a suitable solvent. The system utilizes temperature feedback from the vessels to maintain a narrow temperature window during the extraction process.

3.3. Laboratory Control Sample (LCS) – A sample matrix, free from the analytes of interest, spiked with verified amounts of analyes or a material containing known amounts of analytes. It is generally used to establish intra-laboratory or analyst-specific precision or to assess the performance of all or a portion of the measurement system.

3.4. Matrix Spike (MS)- A sample prepared by adding a known mass of target analyte to a specified amount of sample matrix for which an independent estimate of target analyte concentration is available. Matrix spikes are used, to determine the effect of the sample matrix on a method's recovery efficiency.

3.5. Matrix Spike Duplicate (MSD)- A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

3.6. Method Blank (MB) – A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in

which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

3.7. Surrogate – A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

3.8. Sulfuric Acid/Silica gel for TPH-D: clean-up can be used to remove naturally occurring non-petroleum organics (e.g. leaf litter, bark, etc.), which may contribute biogenic interferences and assist in the reduction or elimination of these interferences. Acid Clean-up procedure is strictly designated for TPH-D analyses. This method of clean up is not suitable for AK-102, AK-103 or HCID analyses.

3.9. Silica-gel - A regenerative adsorbent with weakly acidic properties which can be used in column chromatography for the separation of analytes from interfering compounds.

3.10. Sulfuric Acid Clean up for PCB: A rigorous clean up process used in the removal of most organic chemicals prior to analysis of PCB's. It does not destroy chlorinated benzene or chlorinated naphthalene.

3.11. Tetra Butyl Ammonium Sulfite (TBAS)- reagent used to remove elemental sulfur from solvent extracts. Preparation of the reagent is described in ARI SOP 334S Sulfur Removal.

3.12. Puget Sound Estuary Program (PSEP): Requires up to a 100 g wet weight extraction.

3.13. Puget Sound Dredge Disposal Act (PSDDA): Requires an extraction that is equivalent to 25 g dry weight to 0.5 mL Final Effective Volume (FEV).

4. Interferences

4.1. Standard solutions, solvents or supports materials must not contain extraneous compounds or other chemical interferences. All standards are verified by GC/ECD, GC-FID or GC/MS prior to use. All solvent lots and support materials are checked for purity prior to use.

4.2. Glassware used in the procedure is cleaned and kiln fired as described in ARI SOP 301S. Glassware is rinsed with clean solvent just prior to use.

4.3. Extracting solvent may contain impurities that could interfere with analyses. Use solvents from a supplier's lot that have are verified free of such contaminates.

4.4. Laboratory supplies and equipment are potential sources of interfering contamination

4.4.1 Items such as gloves, bench paper, rubber stoppers should not come into contact with samples or extracts.

4.5. Airborne dust and other debris may contaminate samples. Samples and extracts must be covered at all times.

5. Safety

5.1. The toxicity or carcinogenicity of each reagent used in this SOP are not precisely defined. Treat each compound as a potential health hazard. Reduce exposure to all chemicals to the lowest possible level by whatever means available.

5.2. Always wear appropriate PPE (personal protective equipment) when working in the Organics Extraction Laboratory. Gloves, safety glasses, ear protection, lab coats, respirators, face shields, etc. are provided for your protection.

5.3. DO NOT attempt to cleanup solvent spills in the laboratory. Immediately evacuate the area and contact a member of the Emergency Response Team (ERT) for assistance.

5.4. Material Safety Data Sheets (MSDS) that outline hazards, exposure limits, treatments and regulatory guidelines are available for all chemicals used in this procedure and should be consulted when such information is required. The MSDS file is located in the central project management area. MSDS are also available online, at http//hazard.com/MSDS/.

5.5. Environmental Samples may contain hazardous material; treat them as potential health hazards.

5.6. Dispose of all unwanted, broken glassware into a broken glassware disposal box. Inspect every piece of glassware prior to use. Do not use glassware that is chipped, cracked, etched, or scratched. Set aside glassware with minor damage for repair.

5.7. Use nitrile gloves when working with organic solvents; latex gloves are not appropriate for working with methylene chloride or other solvents.

6. Equipment and Supplies

6.1. Equipment

6.1.1 Personal protective equipment (PPE)

6.1.1.1. Gloves

6.1.1.2. Laboratory coat

6.1.1.3. Goggles

6.1.2 Centrifuge Beckmann GP or equivalent

6.1.3 UltraSonic Sonicator – Branson 450 with ¾" titanium horn

6.1.4 MARS System (Microwave), CEM Corporation

6.1.4.1. Teflon Vessels

6.1.5 Top loading balance-accurate to 0.02g

6.1.6 Drying oven (VWR BIOE) set at 105℃

6.1.7 Portable Paint Shaker-Blair Turbo II or equivalent

6.1.8 RapidTrace Workstation-(SPE)

6.1.9 Water Bath, Precision Scientific or equivalent

6.1.10 Turbo Vap LV, Zymark, (set at 40º C)

6.1.11 Vortex Mixer-Fisher Scientific or equivalent

6.1.12 Gel Permeation Chromatography (GPC). See ARI SOP 306S, "GPC Clean-up".

6.1.13 Hamilton gastight syringes: 25µL, 50µL, 100µL, 250µL, 1.0mL, 5.0mL, and 10.0mL

6.2. Glassware

6.2.1 Separatory funnel (SF), glass, 250mL, with PTFE stopper and stopcock plug (Herb)

6.2.2 Erlenmeyer flask, Glass, 500mL or 250mL

6.2.3 Funnel, glass, 80mm or 100mm

6.2.4 Pasteur Pipette, borosilicate glass, 5 ¾ inch, disposable

6.2.5 Drying Column, glass, 25mm X 250mm

6.2.6 Kuderna-Danish (KD) concentrating apparatus, glass, 500mL, and three ball Snyder Column

6.2.7 Turbo Tube, glass, 60mL

6.2.8 VOA vial, glass, 40mL, with PTFE lined screw cap

6.2.9 Scintillation vial, glass, 20mL, with foil lined screw cap

6.2.10 Centrifuge tube, borosilicate glass, 15mL, with PTFE lined screw cap

6.2.11 Disposable culture tube, borosilicate glass, 16 X 100mm

6.2.12 Medium Micro Column, glass, 16 X 297mm

6.2.13 Culture tube, glass, 16mm X 100mm

6.2.14 Beaker, glass, 600mL, 400mL, 250mL, 150mL, 100mL or 50mL

6.2.15 Graduated cylinder, glass, 50mL

6.2.16 Vial, auto sampler, clear glass, 1.5mL with PTFE lined crimp cap

6.2.17 Vial, amber glass, 1.5mL with PTFE lined screw cap

6.3. Supplies

6.3.1 Wide Sharpie Permanent Pen

6.3.2 Boiling Chips-PTFE (Chemware)

6.3.3 Green, Yellow, Pink, Red, Blue, White, and Grey label tapes

6.3.4 Stainless steel spatula

6.3.5 45mm aluminum weighing dish

6.3.6 Kim wipe

6.3.7 Rubber bulb, amber latex, 1mL

6.3.8 Narrow range pH paper

6.3.9 Filter, 0.20µm, (13mm PTFE), Whatman Puridisc

6.3.10 Filter, 0.45µm, (13-25mm PTFE), Whatman Puridisc

6.3.11 Filter, 0.45µm, GDX HPF (25mm), Whatman

7. Reagents and Standards for soil/sediment extractions: Reagents by Parameter, Matrix, Task Instruction, and Preparation of the solutions is located in Appendix 20.2, (TI-020)

7.1. Acetone, EMD (AX0120-8) or equivalent

7.2. Hexanes, Fisher Optima (H303-4) or equivalent

7.2.1 50:50 Hexane/Methylene Chloride solution

7.2.2 70:30 Hexane/Methylene Chloride solution

7.2.3 80:20 Hexane/Acetone solution

7.2.4 Methylene Chloride, Fisher (D 154-4) or equivalent

7.2.4.1. 70:30 Methylene Chloride/Hexane solution

7.2.4.2. 50:50 Methylene Chloride/Acetone solution

7.2.4.3. 50:50 Methylene Chloride/Ether solution

7.2.4.4. 70:30 Methylene Chloride/Ether solution

7.3. Ethyl Ether, Fisher Pesticide Grade (E199-4) or equivalent

7.4. Pentane, Fisher HPLC grade, (P399-4) or equivalent

7.4.1 90:10 Pentane/Hexane solution

7.4.2 60:40 Pentane/Methylene Chloride solution

7.5. Concentrated hydrochloric acid, Fisher (A144-212) or equivalent

7.5.1 1:1 hydrochloric acid solution

7.6. Concentrated sulfuric acid, Fisher (A300-500) or equivalent

7.6.1 1:1 sulfuric acid solution

7.7. Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours

7.7.1 Acidified (Herb) sodium sulfate

7.7.2 Pre-deactivated sodium sulfate: Acidified for (BAN)

7.8. Neutral glass wool: prepared by baking in kiln at 400º C for 4 hours

7.8.1 Pre-de-activated glass wool: Acidified for (BAN)

7.9. Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

7.10. 37% Potassium hydroxide (KOH) solution

7.10.1 Potassium hydroxide pellets (85+%), Sigma Aldrich (221473-1KG)

7.11. 0.10% Tropolone in Methylene Chloride solution

7.11.1 Tropolone, 98%, Aldrich (T89702-25g) or equivalent

7.12. 1.5 M Hexyl magnesium bromide in diethyl ether reagent

7.12.1 1-Bromohexane, 99%, AlfaAesar, 2500g

7.12.2 Magnesium, chips, 99.98%, Aldrich

7.12.3 Ethyl Ether, Burdick & Jackson High Purity (107-4) or equivalent

7.13. Tetrabutylammonium sulfite (TBAS) reagent

7.13.1 Tetrabutylammonium hydrogensulfate, ACS Grade, Sigma Aldrich (155837-1KG)

7.13.2 Sodium sulfite, Anhydrous, Fisher, (S430-500)

7.14. Ethyl Acetate, Fisher, (E191-4)

7.15. Silica Gel (SPE) darts, 3mL/500mg, Supelco Discovery

7.16. Silica Gel, 60-200 mesh, Fisher, (S704-10) or equivalent

7.17. 0% Silica Gel

7.17.1 Activity 0% Alumina

7.18. Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions. Preparation of the solutions is documented in the Reagent Preparation Logbook (Form 10076F)

8. Sample Collection, Preservation, Shipment and Storage

8.1. Required holding times of soil and sediment samples is 14 days from time of collection and samples are extracted with-in this two week holding period unless previously frozen. Freezing will stop the holding time until thawed. When holding times are or may be compromised inform your supervisor immediately.

8.2. Samples are placed in bins in Refrigerator 05 and recorded in the Refrigerator 05 Logbook by Sample Receiving staff. When retrieving samples from Refrigerator 05 you must record your action in the Logbook.

8.3. Following extraction, soils are archived in walk-in Refrigerator 35 and stored between 2˚C - 6˚C until the disposal date. Upon client's request, soils will be frozen and stored in Freezer 51 at temperature of -20 to-10˚C (minimum temperature) and held for 1 year.

8.4. All sediment samples are automatically frozen and are stored in Freezer 51 at -20 to -10˚C and held for 1 year.

8.5. Some samples are shared with the metal and or conventional laboratories. These samples are placed in a share bin in Refrigerator 36. SOP 1019S includes procedures for handling shared samples. **Excess standing water can NOT be discarded if samples are shared between laboratories.**

9. Quality Control

9.1. Quality Control requirements are specified on the analysis specific bench sheet

9.2. Generally, all samples are spiked with surrogate standard(s), and each extraction batch of 20 or fewer samples include a Method Blank (MB) and a Lab Control Sample (LCS).

9.3. When requested by ARI's client's or required by published analytical protocol other QA samples such as Matrix Spikes, Replicates, Standard Reference Materials are prepared and extracted.

9.4. To verify that surrogate and matrix spikes is accurate; all spike additions are witnessed and documented by a second laboratory technician.

9.5. QC samples including Method Blanks and Laboratory Control Samples (LCS) must be subjected to the clean-up procedures along with any samples that require that technique.

9.6. Prior to beginning the extraction process, determine what procedures are required by reviewing the bench sheet. Follow all information listed for extraction volumes, surrogate and spike additions, pH requirements and special instructions listed at the bottom of all bench sheets. Bench sheet Forms are listed in Appendix 20.1 and examples are contained in Appendix 20.3.

9.7. Proceed slowly and carefully while extracting samples. Note all problems, concerns, errors or deviations from the standard procedure on an Analyst Notes Form (Form 3056F). What seem to be minor errors or deviations may have a significant impact on the final results. Analyst must report all deviations from standard procedures to the laboratory supervisor as soon as possible. Failure to do so may result in a disciplinary process.

9.8. Label glassware and vials with permanent markers.

9.9. Verify sample identification is transferred correctly when transferring or vialing samples and extracts.

9.10. The LIMS Data entry person will review all bench sheets before distribution.

9.11. The laboratory supervisor reviews all logbooks for completeness and accuracy monthly.

9.12. The QA section periodically reviews the standard preparation process, including standard bottles, logbooks and standard certificates and traceability to standardized sources.

10. Calibration and Standardization

10.1. Not Applicable

11. Procedure

11.1. The procedure includes a series of tasks performed sequentially. The tasks are listed in Table 01. A different analyst may be assigned to any task. Detailed procedures for each task are contained in specific "Task Instructions" (TI). All tasks are listed in Table 01. Details of the first nine task instructions are included in this section (TI 021 thru TI 029). "Task Instructions" (TI 008 thru TI 020) are detailed in Appendix 20.2.

Table 01-Task for Extraction of Soil/Sediment samples			
	Preparation Step	**Optional Tasks**	**Task Instruction**
1	Prepare for extraction		TI 021
2	Weigh sample	Sonication/MARS/Vortex	TI 022
3	Spike sample	Sonication	TI 023
4	Dry sample	Sonication	TI 024
5	Extract sample	Sonication	TI 025
6	Dry sample	MARS (microwave)	TI 026
7	Spike sample	MARS (microwave)	TI 027
8	Extract sample	MARS (microwave)	TI 028
9	Medium Level-Waste Dilution Extraction	Vortex	TI 029
10	Concentrate extract	Kuderna-Danish	TI 008
		Turbo-Vap	TI 009
11	Extract Cleanup/Hydrolysis/Derivitization	Sulfuric Acid/Silica Gel for TPH-D	TI 011
		Sulfuric Acid for Pesticide or PCB	TI 012
		Sulfur removal	TI 013
		SPE-Silica Gel	TI 014
		Alumina	TI 015
		EPH Fractionation	TI 016
		Hydrolysis/Derivitization for TBT	TI 017
		Hydrolysis/Derivitization for Herbicides & PCP	TI 018
12	Vialing of final extract		TI 019

11.2. **(TI-021)** Preparing for extraction

11.2.1 Review the appropriate bench sheet Form listed in Appendix 20.1 (current bench sheets by parameter are provided in Appendix 20.4) to determine sample weight to be extracted, surrogate and spike additions, pH requirements, final effective volume, reporting limits requested and any other special requirements or instructions.

11.2.2 Obtain an Analyst Notes Form (3056F) and enter required data on the forms. Add header and sample identifications to the forms as appropriate. (Current example form is provided in Appendix 20.4).

11.2.3 Remove samples from Extractions Incoming Samples Refrigerator 5 and allow them to warm to room temperature.

11.2.4 Verify the client Identification's with the ARI labels. If the identifications do not match, inform the Organic Extractions Supervisor immediately.

11.2.5 Percent total solids are analyzed when the samples are screened. See Sample Screening (ARI SOP# 359S). Percent total solids will determine how much wet weight is needed to meet detection limits.

11.2.6 **Note: MARS (microwave) only:** The limit factors on sample amount is the % total solids are less than 60% and the wet weight does not exceed 20 grams. The combined sample/sodium sulfate mixture must be below the ¾ mark in the microwave vessel. Less than 60 % solids or higher than 20 grams wet weight must be extracted by sonication.

11.2.7 Label each appropriate size beaker or scintillation vial (see bench sheet) with the appropriate color label tape (see Table 02 below) for the analysis containing the following information: ARI job number, sample ID letter, matrix ID, and type of extraction eq: (Pesticide or PCB). Method blank and LCS labels must also include the date of extraction.

11.2.8 **Note: All glassware or microwave vessels associated with TBT must be pre-rinsed with 0.10% Tropolone in Methylene Chloride. Note: TBT extracts must be completed with in 48 hours!**

11.2.9 Table 02

Table 02: Color coded Label Tape by Matrix and Analysis

Analysis	Matrix	Label Tape Color
Pest	Soil/Sediment	Green
PCB	Soil/Sediment/Solid/Oil/Wipe	Green
SIM OP Pest	Soil/Sediment	Green
PCP	Soil/Sediment	Purple
Herb	Soil/Sediment	Pink
TBT	Soil/Sediment	Red
EPH	Soil/Sediment	Yellow (Aliphatic), White (Aromatic)
TPHD	Soil/Sediment	Grey
HCID	Soil/Sediment	No Label
AK102/103	Soil/Sediment	Grey

8270 PNA	Soil/Sediment	Green
SIM PNA	Soil/Sediment	Green
BAN or SIM BAN	Soil/Sediment	Green

11.3. **(TI-022)** Weigh sample for Sonication/MARS/Vortex

11.3.1 See Table 03 below for pH requirements for sodium sulfate, sample, and glass wool for specific analysis and extraction technique.

Table 03: pH requirements per analysis

Analysis	Sodium Sulfate	Sample pH/Solution	Glass Wool
Pest	Neutral	Neutral	Neutral
PCB	Neutral	Neutral	Neutral
SIM OP Pest	Neutral	Neutral	Neutral
PCP	Neutral	Acidic/ Conc. Sulfuric Acid	No Glass wool
Herb	Acidified (Herb) Sodium Sulfate for MB and LCS Acidified (Herb) Sodium Sulfate for drying soil/sediment	Acidic/ Conc. HCL	No Glass wool
TBT	Neutral	Neutral	Neutral
EPH	Neutral	Neutral	Neutral
TPH	Neutral	Neutral	Neutral
HCID	Neutral	Neutral	Neutral
AK102/103	Neutral	Neutral	Neutral
8270 PNA	Neutral	Neutral	Neutral
SIM PNA	Neutral	Neutral	Neutral
BAN SIM BAN	Pre-deactivated sodium sulfate: (TI-020) MB and LCS only ---------- Neutral for drying soil/sediment	Neutral	Sonication funnels: Pre-deactivated glass wool: (TI-020) ---------- KD Drying Column: Pre-deactivated glass wool: (TI-020) ---------- MARS funnels: Neutral glass wool

11.3.2 Mix samples to ensure that a homogeneous and representative aliquot is being used. If samples are not being shared with other labs then discard any standing water and

extraneous materials (large rocks, sticks, etc.) and note on the Analyst Notes Form (3056F). **Note: Do not decant standing water from samples that are shared with other labs.**

11.3.3 Using a top loading balance, prepare two appropriate sized beakers or scintillation vials with anhydrous sodium sulfate (pH type-see Table 3) listed on the bench sheet, for the method blank and one for the LCS. Weigh appropriate amount of sample listed on the bench sheet into a pre-labeled beaker or scintillation vial.

11.3.4 Proceed to section 11.4 to 11.6.8 for spiking, drying soil/sediment with sodium sulfate, and extracting by sonication or medium level extraction details.

11.3.5 Proceed to section 11.7 to 11.9.10 for MARS drying soil/sediment with sodium sulfate, spiking and microwave extracting details.

11.4. **(TI-023)** Spiking Samples for Sonication

11.4.1 Add the appropriate volume of surrogate solution specified on the bench sheet to all samples including MB, LCS, LSD, MS, and MSD. To verify that the surrogate spiking is accurate, the surrogate addition will be witnessed and documented by another laboratory technician. If the sample is an LCS, LCS Duplicate, or MS, MSD, proceed to section 11.4.3.

11.4.2 <u>As samples are being surrogated</u>, add enough of the appropriate solvent (see bench sheet) so the solvent layer is about a ½ inch above each sample for sonication. Correct volumes of solvent for medium levels are listed on the bench sheet.

11.4.3 Add the appropriate volume of spike solution specified on the bench sheet to the LCS, LCS Duplicate and any MS, MSD sample. To verify that the matrix spiking is accurate, the spike addition will be witnessed and documented by another laboratory technician.

11.5. **(TI-024) and (TI-029)** Drying soil/sediment samples for sonication or vortex for medium level.

11.5.1 Add a sufficient amount of anhydrous sodium sulfate to dry the samples. (See table 3 for sodium sulfate pH requirements). <u>Start with 5-25 grams max at first depending on the weight of the sample</u>. Mix well and let sit for a few minutes. When samples sit for a period of time, clumping may occur. Break up any clumping prior to extracting. Add more sulfate if samples are gummy or sticking to the glass beaker during sonication or vortex.

11.5.2 The samples are now ready for extraction by sonication. Proceed to 11.6.

11.5.3 Medium Level/Waste Dilution samples contained in scintillation vials are vortexed for 2 minutes. Proceed to section 11.6.8 for concentration procedures.

11.6. **(TI-025)** Extracting samples by sonication

11.6.1 All sonicators must be functioning properly and monitored by the lab technician to make sure of proper disruption. Form 3090F details the Sonicator Function Test for each Sonicator and is documented weekly.

11.6.2 Place the sonic probe into the sample beaker with the tip just below the surface of the solvent but above the soil/sediment layer. Set the timer for 3 minutes. Power output should be 4-10 depending on matrix. Adjust mode to pulse and duty cycle to 70%.

11.6.3 Once the timer shuts off, remove the beaker and pour the extraction solvent through the funnel into the corresponding labeled Erlenmeyer flask or K-D.

11.6.4 Repeat steps 11.6.2 and 11.6.3 two more times with the appropriate solvent.

11.6.5 After the last sonication, the sample is transferred to the 70mm funnel. The beaker is then rinsed with the appropriate solvent. Pour the rinsate through the funnel and then rinse the funnel and extracted sample with small amounts of the appropriate solvent.

11.6.6 Remove the funnel and discard the soil/sediment into buckets marked for halogenated waste.

11.6.7 Submit the sample extract for concentration task as specified on the bench sheet.

11.6.8 Concentrate sample as specified on the bench sheet. Options include:

11.6.8.1. **Concentration and solvent exchange by Kuderna-Danish using TI-008**

11.6.8.2. **Concentration and solvent exchange by Turbo Vap using TI-009**

11.6.9 Concentrated extracts may require clean-up, hydrolysis, derivitization or separation as specified on the bench sheet. Extract treatment options/requirements are: (See bench sheet details).

11.6.9.1. **TPH-D Sulfuric Acid/Silica Gel Clean-up using TI-011S**

11.6.9.2. **Pest or PCB Sulfuric Acid Clean-up using TI-012**

11.6.9.3. **Pest or PCB Sulfur removal using TI-013**

11.6.9.4. **Silica Gel (SPE) Clean-up for Pest, PCB, PNA, and TPH-D using TI-014**

11.6.9.5. **Alumina Clean-up for TBT using TI-015**

11.6.9.6. **EPH Fractionation using TI-016**

11.6.9.7. **Derivitization and Hydrolysis for TBT using TI-017**

11.6.9.8. **Hydrolysis & Derivitization procedures for Herbicide and PCP using TI-018S**

11.6.9.9. **GPC Clean-up for BAN, SIM BAN (See SOP 306S)**

11.7. **(TI-026)** Drying soil/sediment samples for MARS (microwave)

11.7.1 See previous table 3 for pH requirements for sodium sulfate, sample, and glass wool for specific analysis and extraction technique. See appropriate bench sheet listed in Appendix 20.1

11.7.2 Add a sufficient amount of anhydrous sodium sulfate to dry the samples. Start with 10 grams max at first depending on the weight of the sample. Mix well and let sit for a few minutes. When samples sit for a period of time, clumping may occur. Break up any

clumping prior to extracting. Add more sulfate if samples are gummy or sticking to the glass beaker.

11.7.3 Using a glass powder funnel each sample is transferred to a microwave vessel with the appropriate sample label. Add approximately 20mL – 40ml of the appropriate solvent (see table 4) to the vessel. Enough extraction solvent must be added to completely cover the sample contents with an additional 1" of solvent above the solids.

11.7.4 Use the following Table 4 for MARS correct extraction solvent systems:

Table 04-MARS Extraction/Rinsing Solvent Requirements		
Analysis	**Extraction Solvent**	**Rinse Solvent**
TPH	DCM	DCM
PEST	50:50 Hexane/DCM	Hexane
PCB	50:50 Hexane/DCM	Hexane
BAN	DCM	DCM
TBT	0.10% Tropolone/ DCM	Hexane
PNA	DCM	DCM

11.7.5 The labeled beakers, funnels, and spatulas are to be set aside for use later in the preparation.

11.8. **(TI-027)** Spiking Samples for MARS (microwave)

11.8.1 Add the appropriate volume of surrogate solution specified on the bench sheet to all samples including MB, LCS, LSD, MS, and MSD. To verify that the surrogate spiking is accurate, the surrogate addition will be witnessed and documented by another laboratory technician. If the sample is an LCS, LCS Duplicate, or MS, MSD, proceed to section 11.8.2

11.8.2 Add the appropriate volume of spike solution specified on the bench sheet to the LCS, LCS Duplicate and any MS, MSD sample. To verify that the matrix spiking is accurate, the spike addition will be witnessed and documented by another laboratory technician.

11.9. **(TI-028)** Extracting samples by MARS (microwave)

11.9.1 The MARS stoppers and caps are loosely put on the vessels and the caps are torqued to a specific value by the MARS capping station.

11.9.2 The MARS sample vessels are shaken to ensure the sample mixture is free flowing and not clumped at the bottom of the vessel. This also eliminates air pockets within the sample matrix.

11.9.3 Place the labeled Teflon™ vessels into the MARS carousel as evenly spaced as possible in order for the system to accurately measure the average vessel temperature during the extraction process.

11.9.4 Choose the correct temperature profile based on the number of samples in the carousel. Generally use: 560 watt for 5-9 samples, 800 watt for 10-20 samples, 1200 watt for 20-30 samples, and 1600 watt for 30-40 samples. Although the microwave system will dynamically adjust the microwave power, these starting values facilitate a smooth temperature profile.

11.9.5 After the carousel is seated and the door is closed push the start button on the MARS system. The samples will extract at 105° C for twenty minutes.

11.9.6 After the microwave is finished, remove the carousel. Allow the samples to cool to the touch with no visible boiling can be seen in the vessel.

11.9.7 After the vessels have cooled the sample is shaken to ensure the sample mixture is free flowing and not clumped at the bottom of the vessel. The caps are removed with the MARS capping station.

11.9.8 To collect the extract, the original powder funnel is used for each corresponding extract and plugged with glass wool that has approximately ½ inch of sodium sulfate on top. Place the funnel on top of the corresponding pre-labeled turbo tubes. The solvent is poured through the funnel and the extracts are collected into labeled turbo vap tubes.

11.9.9 Rinse the remaining sample contents in the MARS vessel and add approximately 10 ml of the wash solvent. The MARS vessel is capped and shaken well by hand to ensure a through rising of the contents. The solvent is poured through the powder funnel and collected into the previous turbo tube fraction. Repeat this step one more time with an additional 10 ml of fresh rinse solvent.

11.9.10 The extracts are now ready for concentration.

11.9.10.1. **Concentration and solvent exchange by Turbo Vap using TI-009**

11.9.11 Concentrated extracts may require clean-up, hydrolysis, derivitization or separation as specified on the bench sheet. Extract treatment options/requirements are: (See bench sheet details).

11.9.11.1. **TPH-D Sulfuric Acid/Silica Gel Clean-up using TI-011S**

11.9.11.2. **Pest or PCB Sulfuric Acid Clean-up using TI-012**

11.9.11.3. **Pest or PCB Sulfur removal using TI-013**

11.9.11.4. **Silica Gel (SPE) Clean-up for Pest, PCB, PNA, and TPH-D using TI-014**

11.9.11.5. **Alumina Clean-up for TBT using TI-015**

11.9.11.6. **EPH Fractionation using TI-016**

11.9.11.7. **Derivitization and Hydrolysis for TBT using TI-017**

11.9.11.8. **Hydrolysis & Derivitization procedures for Herbicide and PCP using TI-018S**

11.9.11.9. GPC Clean-up for BAN, SIM BAN (See SOP 306S)

12. Data Analysis and Calculations

12.1. Not Applicable

13. Method Performance

13.1. QA maintains control charts for the recovery of surrogate standards and spiked compounds.

13.2. Management periodically reviews the charts to detect and correct any negative trends in analyte recovery.

14. Pollution Prevention

14.1. Do not discard solvent contaminated solid material into trash containers. Place the solids in the designated 5-gallon "satellite accumulation stations" located at various places in the laboratory. This includes spent sodium sulfate, glass wool, solid sample, silica gel and paper wipes. Transfer the solids to a 55-gallon drum in the Hazardous Waste Room when the accumulation pails are full.

14.2. Disposed expired standards into the designated barrel in the hazardous waste room.

14.3. Samples that are designated as hazardous waste by the LIMS "Hazardous Report" must be placed in the designated drum in the Hazardous Waste Storage Area when they are disposed. This process is described in SOP 1003S.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Not Applicable

16. Corrective Actions for Out of Control Events

16.1. Promptly report any events that may compromise the extraction process to the Organic Extractions Supervisor who will take appropriate steps to insure data quality. Corrective actions may include, but are not limited to, notation on the Analyst Notes Form (3056F) or re-extraction of the sample.

16.2. Corrective action procedures for common laboratory issues are provided in the diagrams provided in Section 20.3.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Unacceptable QA data noted during GC or GCMS analysis may result in a request for re-extraction using a 'Request for Re-extraction/Re-analysis Form (Form 0030F). Re-extract

parameters (sample volume, final volume etc.) may be modified from the original extraction based on analytical results.

18. Waste Management

18.1. Place the solids in the designated 5-gallon "satellite accumulation stations" located at various places in the laboratory. This includes spent sodium sulfate, glass wool, solid sample, silica gel and paper wipes. Transfer the solids to the 55-gallon drum labeled "Solvent Contaminated Solids" located in the Hazardous Waste Room when the accumulation pails are full.

18.2. Discard all waste solvent (Hexane and Methylene Chloride) into the 55 gallon drum labeled "Chlorinated Solvents" located in the Hazardous Waste Storage Area

18.3. Disposed expired standards into the designated barrel in the hazardous waste room.

18.4. Place samples that designate as hazardous using the LIMS "Hazardous Report" in the designated drum in the Hazardous Waste Storage Area when they are disposed. SOP 1003S describes the process for disposal of samples. Excess extracts and expired spiking solutions must be disposed of in the container labeled "Chlorinated Solvents" located in the Hazardous Waste Storage Area.

18.5. ARI's Laboratory Chemical Hygiene Plan (CHP) describes internal hazardous waste handling procedures. All analysts must be familiar with these requirements.

18.6. ARI properly profiles and disposes all hazardous waste using an EPA registered TSD (Treatment, Storage and Disposal) facility.

19. Method References

19.1. SW-846, "Ultrasonic Extraction", Method 3550-B, Revision 2, December, 1996. USEPA Test Methods for Evaluating Solid Waste

19.2. SW-846, "Waste Dilution", Method 3580A, Revision 1, July, 1992. USEPA Test Methods for Evaluating Solid Waste

19.3. Method 3546, Revision 0, February, 2007. USEPA Test Methods for Evaluating Solid Waste

19.4. Washington State Department of Ecology, "Extractable Petroleum Hydrocarbons", June 1997.

19.5. Washington State Department of Ecology, "Analytical Methods for Petroleum Hydrocarbons", June 1997.

19.6. Alaska Laboratory Method for the Analysis of Diesel Range Organics (DRO), AK102, November 7, 2002.

19.7. Alaska Laboratory Method for the Analysis of Residual Range Organics (RRO), AK103, November 7, 2002.

19.8. Krone (TBT), 1988.

19.9. SW-846, Alumina Clean-up, Method 3610B, Revision 3, December, 1996.

19.10. SW-846, Silica Gel Clean-up, Method 3630C, Revision 3, December, 1996.

19.11. SW-846, Sulfur Clean-up, Method 3660B, Revision 3, December, 1996.

19.12. SW-846, Sulfuric Acid Clean-up, Method 3665A, Revision 3, December, 1996.

19.13. SW-846, Acid-Base Partition Clean-up, Method 3650, Revision 1, July, 1992.

20. Appendices

20.1. Extractions Performed Using SOP 3304S – Page 20

20.2. Task Instructions (TI)

20.2.1 TI-021 - Preparing for extraction

20.2.2 TI-022 – Weigh sample volume

20.2.3 TI-023 – Spike sample for sonication

20.2.4 TI-024 – Dry sample for sonication

20.2.5 TI-025 – Extract sample by sonication

20.2.6 TI-026 – Dry sample for MARS (microwave)

20.2.7 TI-027 – Spike sample for MARS (microwave)

20.2.8 TI-028 – Extract sample by MARS (microwave)

20.2.9 TI-029- Medium Level-Waste Dilution Extraction

20.2.10 TI-008 - Concentration and Solvent Exchange by Kuderna-Danish

20.2.11 TI-009 - Concentration and Solvent Exchange by Turbo Vap.

20.2.12 TI-010 - Concentration and vialing procedures by Dry Vap.

20.2.13 TI-011S - TPH-D Sulfuric Acid/Silica Gel Clean-up

20.2.14 TI-012 - Pest or PCB Sulfuric Acid Clean-up

20.2.15 TI-013 - Pest or PCB Sulfur removal

20.2.16 TI-014 - Silica Gel by (SPE) Clean-up for Pest, PCB, PNA, and TPH-D.

20.2.17 TI-015 - Alumina Clean-up for TBT.

20.2.18 TI-016 - EPH Fractionation.

20.2.19 TI-017 - Derivitization and Hydrolysis procedures for TBT.

20.2.20 TI-018S- Extraction, Hydrolysis, Clean-up, Back Extraction & Derivitization procedures for Herbicides and PCP

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20.3 Corrective Action Flow Charts
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20.4.25 BAN (PSDDA) by Sonication
20.4.26 SIM BAN (PSDDA) by Sonication
20.4.27 8270 PNA (In house) by Sonication
20.4.28 8270 PNA (In house) by Microwave
20.4.29 SIM PNA (In house) by Sonication
20.4.30 SIM PNA (In house) by Microwave
20.4.31 Analyst Notes

Appendix 20.1 - Extractions Performed using SOP 3304S

Procedure Code	Description of Analysis	Bench Sheet	Extraction Technique	Sample Weight (g)	FEV (mL)	Extraction pH	Solvent	Concentration Technique/TI#	Cleanup/Separation/ Derivatization/Hydrolysis/ (TI#/or SOP)
PESSDCI	Pesticides – In-house (1.7-3.3ppb)	3045F	C	12g dry	4.0	Neutral	Initial=8:2 Hexane/Acetone Exchange=Hexane	K-D/Turbo (TI-008)/(TI-009)	Sulfur/Silica Gel (TI-013)/(TI-014)
PESSDMI	Pesticide – In-house (1.7-3.3ppb)	3061F	M	12g dry	4.0	Neutral	Initial=1:1 Hexane/DCM Exchange=Hexane	Turbo (TI-009)	Sulfur/Silica Gel (TI-013)/(TI-014)
PESSDCP	Pesticide – PSDDA (1.0-2.0ppb)	3046F	C	25g dry	5.0	Neutral	Initial=8:2 Hexane/Acetone Exchange=Hexane	K-D/Turbo (TI-008)/(TI-009)	Sulfur/Silica Gel (TI-013)/(TI-014)
PCBSDCI	PCB – In-house (33ppb)	3025F	C	12g dry	4.0	Neutral	Initial=8:2 Hexane/Acetone Exchange=Hexane	K-D/Turbo (TI-008)/(TI-009)	Acid/Sulfur/Silica Gel (TI-012)/(TI-013)/(TI-014)
PCBSDMI	PCB – In house (33ppb)	3067F	M	12g dry	4.0	Neutral	Initial=1:1 Hexane/DCM Exchange=Hexane	Turbo (TI-009)	Acid/Sulfur/Silica Gel (TI-012)/(TI-013)/(TI-014)
PCBSDCP	PCB – PSDDA (20ppb)	3017F	C	25g dry	5.0	Neutral	Initial=8:2 Hexane/Acetone Exchange=Hexane	K-D/Turbo (TI-008)/(TI-009)	Acid/Sulfur/Silica Gel (TI-012)/(TI-013)/(TI-014)
PCBSDCP	PCB – PSDDA (10ppb)	3026F	C	25g dry	2.5	Neutral	Initial=8:2 Hexane/Acetone Exchange=Hexane	K-D/Turbo (TI-008)/(TI-009)	Acid/Sulfur/Silica Gel (TI-012)/(TI-013)/(TI-014)
PCBSDCP	PCB – PSDDA (4ppb)	3024F	C	25g dry	1.0	Neutral	Initial=8:2 Hexane/Acetone Exchange=Hexane	K-D/Turbo (TI-008)/(TI-009)	Acid/Sulfur/Silica Gel (TI-012)/(TI-013)/(TI-014)
PCBSDVX	PCB – Soil/Sed/Solid Med. Level (800ppb)	3019F	V	5g wet	40.0	Neutral	Initial=1:1 DCM/Hexane Exchange=Hexane	Turbo (TI-009)	Acid/Sulfur/Silica Gel (TI-012)/(TI-013)/(TI-014)
PCBOVX	PCB – Oil Med. Level (1000ppb)	3020F	V	2g As received	20.0	Neutral	Initial=1:1 DCM/Hexane Exchange=Hexane	Turbo (TI-009)	Acid/Sulfur (TI-012)/(TI-013)
PCBIVX	PCB – Wipe Med. Level (1µg/Each)	3031F	V	1 Each As received	10.0	N/A	Initial=Hexane	Turbo (TI-009)	Acid/Sulfur/ Silica Gel (TI-012)/(TI-013)/(TI-014)

Appendix 20.1 - Extractions Performed using SOP 3304S

Procedure Code	Description of Analysis	Bench Sheet	Extraction Technique	Sample Weight (g)	FEV (mL)	Extraction pH	Solvent	Concentration Technique/TI#	Cleanup/Separation/ Derivatization/Hydrolysis/ (TI#/or SOP)
OPPSMI	O P-Pest – OP-Pest In house (10-50ppb)	3081F	M	10g dry	1.0	Neutral	DCM	Turbo (TI-009)	None
PCPSDCI	Chlorophenols – In-house (6.25ppb)	3015F	C	10g wet	25.0	Acidic	Initial=DCM Exchange=Hexane	K-D/Turbo (TI-008)/(TI-009)	Derivitized by GC
HERSDCI	Herbicides – In-house (8.3-8300ppb)	3011F	C	15g wet	50.0	Initial=Acidic Cleanup=Basic Back Extract=Acidic	Initial=1X 1:1DCM/Ether 2X 7:3 Hexane/DCM Cleanup=DCM Back Extract= 1X 7:3 DCM/Ether 2X 7:3 DCM/Hexane	K-D/Turbo (TI-008)/(TI-009)	Hydrolysis (TI-018S) Derivitized by GC
TBTSDCI	TBT – In-house (4-6ppb)	3044F	C	5g dry	0.5	Neutral	Initial=0.10%Tropolone/DCM Exchange=Hexane	K-D/Turbo (TI-008)/(TI-009)	Derivatize (TI-017) Alumina (TI-015)
TBTSDMI	TBT – In-house (4-6ppb)	3064F	M	5g dry	0.5	Neutral	Initial=0.10%Tropolone/DCM Exchange=Hexane	Turbo (TI-009)	Derivatize (TI-017) Alumina (TI-015)
EPHSDCI	EPH – In-house (2000ppb)	3072F	C	10g wet	1.0	Neutral	Initial=DCM Exchange=9:1Pentane/Hexane	K-D/Turbo (TI-008)/(TI-009)	Fractionation (TI-016)
HCISVX	NWHCID In-house (50ppm)	3005F	V	10g wet	5.0	Neutral	Initial=DCM	Turbo (TI-009)	N/A
DIESCI	TPHD- In-house (0.25-0.5ppm)	3013F	C	10g wet	1.0	Neutral	DCM	K-D/Turbo (TI-008)/(TI-009)	Acid/Silica Gel (TI-011S)
DIESMI	TPHD- In-house (0.25-0.5ppm)	3066F	M	10g wet	1.0	Neutral	DCM	Turbo (TI-009)	Acid/Silica Gel (TI-011S)
AK2SCI	AK102-AK103 In-house (0.25-0.5ppm)	3004F	C	10g wet	1.0	Neutral	DCM	K-D/Turbo (TI-008)/(TI-009)	Silica Gel (TI-014)
AK2SMI	AK102-AK103 In-house (0.25-0.5ppm)	3073F	M	10g wet	1.0	Neutral	DCM	Turbo (TI-009)	Silica Gel (TI-014)

Appendix 20.1 - Extractions Performed using SOP 3304S

Procedure Code	Description of Analysis	Bench Sheet	Extraction Technique	Sample Weight (g)	FEV (mL)	Extraction pH	Solvent	Concentration Technique/TI#	Cleanup/Separation/ Derivatization/Hydrolysis/ (TI#/or SOP)
BANSDCI	SVOA – In-house (67ppb)	3009F	C	7.5g dry	0.5	Neutral	Initial=2X 1:1DCM/Acetone 1X DCM	K-D/Turbo (TI-008)/(TI-009)	GPC (SOP 306S)
BANSDMI	SVOA – In-house (67ppb)	3063F	M	7.5g dry	0.5	Neutral	DCM	Turbo (TI-009)	GPC (SOP 306S)
BANSDCP	SVOA – PSDDA (20ppb)	3036F	C	25g dry	0.5	Neutral	Initial=2X 1:1DCM/Acetone 1X DCM	K-D/Turbo (TI-008)/(TI-009)	GPC (SOP 306S)
SBANSDCP	SIM SVOA – PSDDA (6.7ppb)	3037F	C	16g dry	1.0	Neutral	Initial=2X 1:1DCM/Acetone 1X DCM	K-D/Turbo (TI-008)/(TI-009)	GPC (SOP 306S)
PNASDCI	PNA – In-house (67ppb)	3007F	C	7.5g dry	0.5	Neutral	Initial=2X 1:1DCM/Acetone 1X DCM Exchange=Hexane	K-D/Turbo (TI-008)/(TI-009)	Silica Gel (TI-014)
PNASDMI	PNA- In House (67ppb)	3059F	M	7.5g dry	0.5	Neutral	Initial=DCM Exchange=Hexane	Turbo (TI-009)	Silica Gel (TI-014)
PNASDCI	PNA-SIM In-house (5 ppb)	3051F	C	10g dry	0.5	Neutral	Initial=2X 1:1DCM/Acetone 1X DCM Exchange=Hexane	K-D/Turbo (TI-008)/(TI-009)	Silica Gel (TI-014)
PNASDMI	PNA-SIM In-house (5 ppb)	3060F	M	10g dry	0.5	Neutral	Initial=DCM Exchange=Hexane	Turbo (TI-009)	Silica Gel (TI-014)

Appendix 20.2

Task Instructions

Task Instructions (TI-008)

Concentrating and Solvent Exchange procedure using Kuderna-Danish.

Refer to bench sheet for requirements and instructions.

1. Concentrating and solvent exchanging extracts by Kuderna-Danish (KD)

1.1. Rinse a K-D apparatus using methylene chloride or hexane. Add 2-3 clean boiling chips. Note – Rinse glassware with the appropriate Methylene Chloride or Hexane for the low level extractions (PCB or Pesticides). See bench sheet.

1.2. Verify that the sodium sulfate in the sample extract is "free flowing". If it is not, add anhydrous sodium sulfate to adsorb any residual water in the extract. Swirl the flask to assure complete removal of any moisture present.

1.3. For aqueous extract: Pour the extract directly from the flask into the prepared K-D apparatus. Proceed to section 1.4.1.

1.4. For soil/sediment extract: Prepare a drying column by inserting a glasswool plug into the bottom. (See bench sheet for pH of glasswool required). Fill the column with approximately 10cm of anhydrous sodium sulfate. Place the column on top of the KD secured with a clamp.

1.4.1 Rinse the flask three times with the appropriate solvent and add the rinsate to the KD flask as well.

1.4.2 Transfer the colored sample ID labels from the flask to the K-D apparatus.

1.4.3 Attach a rinsed three-ball Snyder column to the KD flask, and place the entire apparatus on the proper temperature water bath using Table 4 below.

1.4.4 Wet the inside of the Snyder column with 1 to 2 mL of the extracting solvent before boiling starts.

1.4.5 **Note: If a solvent exchange is not necessary, proceed to step 1.4.7**

1.4.6 **To exchange the solvent to Hexane:** When the extract reaches ~5mL, add 20 mL Hexane to the K-D through the Snyder Column. Mix well and immediately transfer the K-D to another water bath set at 100°C. <u>Place an "/" on the label to indicate the first Hexane exchange process has taken place.</u> When the extract again reaches ~5mL, add an additional 20mL Hexane to the KD through the Snyder Column. <u>Make an "X" on the label to indicate the second Hexane exchange process has taken place.</u>

1.4.7 When the extract reaches ~5mL, remove the K-D from the water bath and allow it to cool for about 10 minutes on a cooling rack. After cooling the extracts should be approximately 5-10 mL.

1.4.8 Remove the Snyder column from the K-D. Pour the extracts from the K-D into the corresponding labeled Turbo Tube and rinse the K-D apparatus with approximately 5 mL of the appropriate solvent and add the rinsate to the Turbo Tube.

1.4.9 Transfer the colored sample identification labels from the K-D Apparatus to the Turbo Tube.

1.4.10 Extracts are now ready for further concentration by turbo vap, clean-up, hydrolysis, derivitization, or separation as specified on the bench sheet.

1.4.11 **EPH concentration:** The extracts should be placed on the 80ºC water bath. **When the extract has concentrated to no higher than 1mL**, exchange by adding 30mL of 90:10 Pentane/Hexane through the Snyder column. Mix well and immediately transfer the KD to another water bath set at 100º C. Mark the label with an **"X"** to indicate the exchange process has taken place.

1.4.12 **EPH exchange: Once the extract has concentrated to no higher than 0.5mL, remove it from the water bath.** (Removing the extract before it has reached 0.5mL could result in an improper exchange and affect the fractionation process).

1.4.13 When the extract reaches ~0.5mL, remove the K-D from the water bath and allow it to cool for about 10 minutes on a cooling rack. After cooling the extracts should be approximately 5mL.

1.4.14 Remove the Snyder column from the K-D. Pour the extracts from the K-D into the corresponding labeled Turbo Tube and rinse the K-D apparatus with approximately 5 mL of the appropriate solvent and add the rinsate to the Turbo Tube.

1.4.15 Transfer the colored sample identification labels from the K-D Apparatus to the Turbo Tube.

1.4.16 Extracts are now ready for further concentration by turbo vap and separation as specified on the bench sheet.

Table 04: Water Bath Temperatures

Water Bath No.	Temperature Range	Solvent Type
1	80-85º C	DCM only
2	100º C	Hexane only Hexane exchanges 90:10 Pentane/Hexane exchange 80:20 Hexane/Acetone
3	90-95º C	50:50 DCM/Acetone

Task Instructions (TI-009)

Concentrating and Solvent Exchange procedure using by Turbo Vap.

Refer to bench sheet for requirements and instructions.

1.Concentrating and solvent exchanging extracts by Turbo Vap

1.5. Concentrate the extracts to a volume slightly less than the volume to lab specified on the bench sheet.

1.6. Verify that the Turbo Tube was previously labeled at the top with a wide Sharpie pen.

1.7. Position the tray in the Turbo Vap set at 40 °C and check the water level which must be at a level even with the first horizontal tray inside the turbo tube rack.

1.8. Select the row(s) of Nitrogen nozzles to be used by pressing the row panel on the Turbo Vap.

1.9. Start with a lower Nitrogen pressure 10-20 psi minimum to avoid splashing. Insert the turbo tubes into the Turbo Vap tray quickly, one at a time opening and closing the lid between turbo tube insertions.

1.10. When all Turbo tubes are inserted into Turbo Vap, close the lid and as quickly as possible with out splashing and bring the pressure of the nitrogen up to maximum 40 psi by turning the pressure control clockwise. Nitrogen must be continuous. **Complete the concentration as quickly as possible with minimal opening and closing of the TurboVap lid to prevent potential losses of analytes.** When the concentration is complete, remove the extracts from the Turbo Vap immediately. Mix well.

1.11. If a hexane exchange is necessary: follow the bench sheet instructions for volumes.

1.12. Once the extract reaches the specified volume to lab or for further clean-up listed on the bench sheet, transfer it using the appropriate solvent and syringe into an appropriate vial. Clean the syringe between extracts by rinsing three times with the appropriate solvent.

Task Instructions (TI-011S)

Sulfuric Acid/Silica Gel Clean-up procedure for TPH-D Soil/Sediment Extracts

Refer to bench sheet for options or requirements.

1. TPH-D Sulfuric Acid/Silica-Gel Clean-up – When requested by ARI's Client or PM: Acid/Silica gel clean-up may remove naturally occurring non-petroleum organics (e.g. tannins, lignin and other biogenic compounds), which may contribute to chromatographic interferences. Acid clean-up is not suitable for AK-102, AK-103, or HCID analysis.

1.1. Sulfuric Acid clean-up

1.1.1 Turbo vap extracts to approximately 1mL following Turbo vap procedure in Task Instructions (TI-009).

1.1.2 Transfer extracts to a 10-15mL disposable centrifuge tube using a pipette. Rinse turbo tube with 0.5mL Methylene Chloride and transfer rinsate to centrifuge tube. Repeat rinse two more times with 0.5mL Methylene Chloride.

1.1.3 Add 1-4 pipettes of concentrated Sulfuric Acid to the extracts depending on the color of the extract. Generally MB, SB, SBD require only one pipette of acid and extracts with color require 2-4 pipettes of acid. (The darker the color, the more acid is required).

1.1.4 Cap extracts and vortex for one minute. Centrifuge extracts at 2000 rpm for 5 minutes.

1.1.5 Transfer extract Methylene Chloride (Top) to a culture tube for SPE silica gel clean-up. Add 0.5mL Methylene Chloride rinse to acid cleaned extract and transfer rinse to culture tube. Repeat 0.5mL Methylene Chloride rinse two more times for a total of 3mL extract for SPE silica gel clean-up.

1.2. Silica Gel Clean-up

1.2.1 Follow Task Instructions (TI-014) for Silica-gel by (SPE) Clean-up Procedure.

Task Instructions (TI-012)

Sulfuric Acid Clean-up procedure for Pest or PCB

Refer to bench sheet for options or requirements.

1. **Pest or PCB Sulfuric Acid Clean-up: This procedure details the sulfuric acid clean-up portion of solvent extracts analyzed for PCBs and selected Organochlorine Pesticides. Sulfuric Acid: A rigorous clean up process used in the removal of most organic chemicals prior to analysis of PCB's. Use caution when analyzing pesticide since some may be degraded using this procedure. It does not destroy chlorinated benzene or chlorinated naphthalene.**

   1.1. Note: Refer to bench sheet for requirements. Required clean-up for (Low Level extraction) or optional and the extract has any color or the analyst notes (3056F) indicates there were emulsions.

   1.2. Note: Extracts must be exchanged to hexane before initiating acid clean-up.

   1.3. The extract subjected to clean-up must be at least 4 mL in volume. Preferably the extract will be adjusted to final volume prior to acid clean-up. Extracts requiring a final volume less than 4.0 mL will be "transfer rinsed" and re-concentrated after clean-up.

   1.4. Transfer the entire extract to a labeled 20 mL scintillation vial.

   1.5. Carefully add 1.5 to 5.0 mL (1 to 3 pipets) of concentrated sulfuric acid to the vial and cap.

   1.6. Vortex the extract/sulfuric acid mixture for 60 seconds.

   1.7. Centrifuge the vial at ≤ 1000 rpm for 5 minutes to expedite phase separation.

   1.8. If the extract was at final volume prior to this procedure, and no other clean-up is required, transfer 1 mL of the hexane extract to a labeled auto-sampler vial for instrumental analysis. **Caution:** (<u>When working in the acid phase it is extremely important to avoid acid in the vial being sent to GC for analysis</u>).

   1.9. When the acid treated extract requires additional concentration, transfer the entire hexane layer (top) to a turbo vap tube. Rinse the scintillation vial twice with 2 mL aliquots of hexane and combine the rinses with the extract in the turbovap tube. Concentrate the hexane extract to the specified final volume for instrumental analysis.

   1.10. If additional clean-up (sulfur or silica-gel) is required, the hexane extract is transferred to the appropriate container for further treatment. See Appendix 20.2.13 Task Instructions (TI-013 Sulfur Removal) and/or 20.2.14 Task Instructions (TI-014 Silica Gel Clean-up) for details.

Task Instructions (TI-013)

Sulfur Removal for Pest or PCB
Refer to bench sheet for options or requirements.

1. **Pest or PCB Sulfur Clean-up: Elemental sulfur is encountered in many sediment, marine algae, and industrial waste sample matrices. The solubility of sulfur in various solvents is similar to some of the organochlorine and organophosphorous pesticides. Therefore, sulfur remains present with the pesticides through the normal extraction and cleanup techniques and causes interference when samples are analyzed using an electron capture detector (ECD).**
   1.1. The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting pesticides and Aroclors. Sulfur contamination should be expected with sediment samples.
   1.2. Sulfur will be quite evident in gas chromatograms obtained from electron capture detectors. If the gas chromatograph is operated at the normal conditions for organochlorine pesticide analysis, the sulfur interference can completely mask the region from the solvent peak through Aldrin.
   1.3. The use of tetrabutylammonium sulfite (TBAS) for the elimination of sulfur is detailed in this method.
   1.4. **Note: Extracts must be exchanged to hexane before initiating sulfur clean-up.**
   1.5. **Note: An extract treated with TBAS must be at least 4 mL in volume.**
   1.6. **PCB: Proceed to step 1.12.**
   1.7. **Pesticides**: **Note:** Refer to bench sheet for requirements. Required clean-up for (**MTCA level** extraction) or optional and the extract has **any color** or the analyst notes (3056F) indicates there were emulsions.
   1.8. Vial 0.5mL below the final effective volume listed on the bench sheet. Extracts requiring a final volume less than 4.0 mL will be re-concentrated after sulfur removal.
   1.9. Transfer the entire extract to a 20 mL scintillation vial.
   1.10. Add 0.5mL of ethyl acetate.
   1.11. Proceed to step 1.13.
   1.12. PCB'S **(Do not add ethyl acetate)**. Note: Refer to bench sheet for requirements. Required clean-up for (**MTCA level or Low Level** extraction) or optional and the extract has **any color** or the analyst notes (3056F) indicates there were emulsions.
   1.13. Add 1 pipette of TBAS reagent to the 20mL scintillation vial.
   1.14. Vial specified Final Effective Volume stated on the bench sheet into a 20mL scintillation vial.

1.15. Add enough crystalline sodium sulfite to cover the bottom of the 20mL scintillation vial.

1.16. Mix the vial for at least two minutes using a Vortex mixer (or put on the paint shaker for 15 minutes). Crystals of sodium sulfite should be present. If not, add additional 0.2 gram portions of sodium sulfite until crystals are present after repeated mixing.

1.17. After shaking: Add at least 10-15 mL organic-free water to the vial and vortex the solution for two minutes (or put back on paint shaker for 15 minutes).

1.18. After shaking: Allow the sample to stand until the layers have completely separated.

1.19. If there is an emulsion, centrifuge the vial at 1000 rpm for 5 minutes to expedite phase separation

1.20. This completes the sulfur removal process.

1.21. If the extract was at final volume prior to this procedure, and no other clean-up is required, transfer 1 mL of the hexane extract to a labeled auto-sampler vial for instrumental analysis.

1.22. If the extract requires additional concentration, transfer the hexane organic layer (top) to a turbo vap tube. Rinse the scintillation vial twice with 2 mL aliquots of hexane and combine the rinses with the extract in the turbo vap tube. Concentrate the hexane extract to final volume for instrumental analysis.

1.23. If additional clean-up (silica-gel) is required, the hexane extract is transferred to the appropriate container for further treatment. See Appendix 20.2.14 Task Instructions (TI-014 Silica Gel Clean-up) for details.

Task Instructions (TI-014)

Silica Gel by (SPE) clean-up procedure
Refer to bench sheet for options or requirements.

1. Silica-Gel Clean-up by (SPE) for Pest, PCB, PNA, and TPH-D extracts by (SPE)

1.1. Silica gel clean-up of aqueous samples is required when samples are analyzed for SIM-PNA, pesticides or PCB using techniques that provide low detection levels.

1.2. Silica gel clean-up of aqueous samples or TCLP extracts is optional when samples are analyzed for pesticides, PCB, PNA, or SIM-PNA using techniques that provide routine detection levels.

1.3. Silica gel cleanup is performed on TPH-D extracts only when it is requested by ARI's client.

1.4. Procedure: Each RapidTrace workstation to be used must be "primed" with solvents, followed by a verification of its operation daily as follows:

1.4.1 Verify the solvent reservoirs contain adequate levels of solvents before proceeding, if not fill with the appropriate solvent.

1.4.2 Load a SPE cartridge into slot 1 of the cartridge carousel and add a disposable culture tube to the collect and sample slots in position one of the sample rack.

1.4.3 Using the RapidTrace software select the appropriate "prime method" for sample slot 1 and allow the workstation to finish the procedure.

1.4.3.1. These procedures automatically add 2 ml of solvent to the sample tube, followed by 'sipping' the sample and adding it to the SPE cartridge. The eluant is collected into the collection tube and the SPE cartridge is washed with an additional 3 ml of solvent which is again collected into the collection tube.

1.4.3.2. Correct operation is verified if the sample tube contains no residual sample and the collection tube contains 5 ml of eluant.

1.4.4 After priming and verification: up to 10 samples in culture tubes are loaded into the right side of the sample rack with a labeled corresponding collection tube in the left side of the rack.

1.4.4.1. Analysis required volumes and solvents are as follows:

Analysis	Volume	Extract Solvent
Pest	2-3mL	Hexane
PCB	2-3mL	Hexane
PNA	2-3mL	Hexane
TPH-D	2-3mL	Methylene Chloride

1.4.5 Using the RapidTrace software select the RapidTrace module(s) and assign appropriate "run method" to the samples loaded and start the run on the loaded module(s).

1.4.6 Record the prime and extract runs in the SPE Run Log (3078F).

1.4.7 It is possible to initiate runs on multiple modules utilizing multiple methods with the RapidTrace control software. During an analytical run each module runs a single method for each set of samples. Do not mix methods in a single analytical run. Allow the run to finish, and then switch the module over to a different method.

1.4.8 The RapidTrace software will calculate precisely the time for the run to finish after it is initiated based on the "run" procedure selected and the number of samples in the tray. When the run is finished, remove the sample tray and visually inspect the sample tubes and collection tubes to verify proper operation during the run. The sample tubes should be empty and the collection tubes all should contain a consistent volume of eluant. Errors should be noted and the laboratory supervisor notified so as to implement corrective action.

1.4.9 The 16X100 mm collections tubes are transferred to the approriated turbo vap fitted for these tubes (or transferred to a labeled 60mL Turbo Tube) and the extracts are concentrated to the final effective volume specified on the bench sheet.

1.4.10 The extracts are transferred to the appropriate auto sampler vial and delivered to the instrument lab for analysis.

Task Instructions (TI-015)

Alumina Clean-up procedure
Refer to bench sheet for options or requirements.

1. Alumina Clean-up for TBT

1.1. **Note:** The extract must be previously derivitized and hydrolyzed. See Appendix 20.2.17 Task Instructions (TI-017 Derivitization and Hydrolysis procedures for TBT for details).

1.2. Pass the hexane extract through a medium size micro column packed with 5 g of activity 0 alumina. Use a pre-rinsed 60ml Turbo Tube to catch the elution. Rinse the extract Turbo Tube with two 0.5mL portions of hexane, and add to the column. Elute column with hexane and collect 3-4 mL. The collected fraction should be colorless. If the fraction is not colorless, concentrate again to 2.0mL and repeat alumina clean-up once more and note this on the analyst notes (3056F).

1.3. Concentrate extract to 0.5mL and vial into a 2.5mL amber vial with a PTFE-lined screw-cap for analysis by GC/MS-SIM. Transfer red label tape with the sample ID to the vial. Note: Final effective volume is 0.5mL.

Task Instructions (TI-016)

EPH Fractionation
Refer to bench sheet for options or requirements.

1. EPH Fractionation: This method details the procedure to fractionate the collective concentrations of extractable aliphatic and aromatic hydrocarbons. This procedure meets the requirements of WDOE 1997.

1.1. Fractionating EPH Extracts Procedure

1.1.1 Prepare a macro column by packing a small plug of neutral glass wool in the bottom of the column. Add approximately 0.5 cm of anhydrous sodium sulfate on top of the glass wool plug. Suspend the column in a clamp over a 150 mL beaker used to collect process waste.

1.1.2 Place a small funnel at the top of the column. Have <u>four labeled turbo tubes</u> ready for collection of the EPH fractions generated (<u>two for aliphatic and two for aromatic</u> collections of each extract).

1.1.3 Weigh 7.5g of 0% silica gel into a tared 50mL beaker. Add enough methylene chloride to slurry the silica gel.

1.1.4 Mix the silica gel slurry well and pour through a small funnel into the prepared macro column. Once all slurry has been added to the column gently tap the column to settle the silica gel.

1.1.5 Once the silica gel slurry has settled, add approximately 0.5cm of anhydrous sodium sulfate to the top of the silica gel, creating a ‘sandwich’ effect.

1.1.6 Rinse the column with one full column of methylene chloride and elute into a waste beaker.

1.1.7 Once all the methylene chloride has eluted to the waste beaker, immediately add 20mL of pentane to the column. This process exchanges the column and prepares it for the aliphatic collection. The pentane rinse is discarded.

1.1.8 Once the pentane exchange has completely eluted to waste. Remove waster beaker and place the first 60mL turbo tube under the column with an aliphatic label on it.

1.2. **Aliphatic Fraction:**

1.2.1 Using a Pasteur pipette, add 2mL of extract onto column. Let the 2mL fully elute thru the column (collect).

1.2.2 Rinse turbo tube with 2mL of pentane and add this to the column as well. Let rinsate fully elute thru the column (collect).

1.2.3 (See updated QC of how many mL of pentane to use located above the fractionation station) Add 27-33mL of pentane to the column and collect. Split the volume of pentane between two turbo tubes.

1.2.4 Once all pentane has eluted from the column, remove turbo tube and replace it with the first turbo tube that has an aromatic label.

1.3. **Aromatic Fraction:**

1.3.1 Once turbo tube is under column, add 35mL of 60:40 Pentane/DCM to the column. Let the first 2mL get thru the column before adding the rest of the solvent.

1.3.2 Once solvent has fully eluted thru column, remove aromatic turbo tube. Split the volume of solvent between two turbo tubes.

1.3.3 Discard silica gel from the columns into the buckets marked for halogenated waste.

1.3.4 Extracts are now ready for final concentration by Turbo Vap.

1.4. **Final Concentration by Turbo Vap and Vialing**

1.4.1 Refer to Turbo vap procedure in section Appendix 20.2. Task Instructions (TI-009).

1.4.2 Concentrate aliphatic and aromatic fractions separately.

1.4.3 Caution: Constant attention must be given to the turbo vap at all times, pentane concentrates very quickly under nitrogen and leaving extracts unattended could result in loss of extracts.

1.4.4 Concentrate aliphatic fractions first. There are two aliphatic turbo tubes per sample. Concentrate each extract to approximately 5mL and then combine corresponding extract into one turbo tube for final concentration and vialing.

1.4.5 Once the extract reaches 1mL transfer it using a 1000µL syringe into a pre-labled 2.5mL amber vial with PTFE-lined screw cap using Methylene Chloride. Transfer the yellow aliphatic label tape to this vial. Rinse the syringe three times with Methylene Chloride between extracts.

1.4.6 Concentrate aromatic fractions next. There are two aromatic turbo tubes per sample. Concentrate each extract to approximately 5mL and then combine corresponding extract into one turbo tube for final concentration and vialing.

1.4.7 Once the extract reaches 1mL transfer it using a 1000µL syringe into a pre-labled 2.5mL amber vial with PTFE-lined screw cap using Methylene Chloride. Transfer the white aromatic label tape to this vial. Rinse the syringe three times with Methylene Chloride between extracts.

Task Instructions (TI-017)

Derivitization and Hydrolysis procedures for TBT

Refer to bench sheet for options or requirements.

1. TBT Derivatization

1.1. Transfer the 3 – 4mL hexane extract to a 40mL VOA vial with PTFE lined screw cap.

1.2. Rinse the turbo tube with 0.5 mL of hexane and add this to the 40mL VOA vial as well.

1.3. Add 1½ pipette of 1.5M Hexyl Magnesium Bromide (HexylMgBr) in diethyl ether. Add the reagent slowly: start with 3-4 drops of the reagent; slowly add the rest of the pipette. Bubbling may occur during this process. If the extract solidifies during the reaction, add hexane to return the "solid" to solution and note it on the analyst notes form (3056F)

1.4. Mix extracts by hand or on the vortexer for 30 seconds.

1.5. Allow extracts to sit for 40 minutes. Mix the extracts on the vortexer approximately every ten minutes during this 40 minute period.

2. TBT Hydrolysis

2.1. Hydrolyze excess HexylMgBr by adding 1 pipette of concentrated HCL. Caution: add the HCL drop wise slowly while shaking the scintillation vial. Allow bubbling to occur before adding the rest of the acid. Vortex for 10 seconds.

2.2. Draw off the acid layer (bottom) with a pipette and discard. Be careful not to draw off any of the hexane layer.

2.3. Add 5 mL of DI water and vortex.

2.4. Draw off the water/acid layer (bottom layer) with a pipette and discard. Be careful not to draw off any of the hexane layer.

2.5. Repeat 1.6.3 – 1.6.4 a second time.

2.6. The extract is dried by adding a small amount of anhydrous sodium sulfate to the vial.

2.7. Allow extract to dry for 15 minutes, sulfate should be "free flowing".

2.8. The extract is now ready for Alumina Clean-up. See Appendix 20.2.15 Task Instructions (TI-015) Alumina Clean-up for TBT.

Task Instructions (TI-018S)

Sonication Extraction, Hydrolysis, Clean-up, Back Extraction, and Derivitization procedures for Soil/Sediment Herb

Refer to bench sheet for options or requirements.

1. Soil/Sediment Herbicide Sonication/Hydrolysis/Clean-up/Back Extraction

1.1. Label each 100 mL beaker with pink label tape containing the following information: ARI job number, sample ID letter, matrix ID, extraction type (HERB) and, on the method blank (MB) and LCS, use the current date as mmddyy.

1.1.1 Prepare two 100 mL beakers with 15 g of acidified anhydrous sodium sulfate for use as the MB and the LCS. Weigh 15 g of each sample into each corresponding 100 mL beaker. Add 15-30 g of acidified anhydrous sodium sulfate to dry the soil samples and mix well. In a fume hood acidify the samples to pH <2 with approximately 0.25 mL (1/8 pipette) of concentrated HCL and stir well.

1.1.2 Add surrogates to each sample, MB, LCS and MS as specified on the bench sheet Form (3011F). In addition to the surrogates, the LCS and MS receive a full list herbicide spike. To verify that each surrogate and spike addition is accurate, each will be witnessed and documented by another laboratory technician.

1.1.3 After the surrogate and spike solutions have been added, immediately pour approximately 90 mL of 1:1 methylene chloride/ether into each sample.

1.1.4 Rinse a Kuderna-Danish (KD) concentrator for each sample with methylene chloride.

1.1.5 Place a 70mm glass funnel on top of the K-D <u>with no glass wool or sodium sulfat</u>e. The KD will be used to collect the extract generated. Label each K-D with sample ID using a wide permanent sharpie pen.

1.1.6 The samples are now ready for extraction by sonication.

1.1.7 All sonicators must be functioning properly and monitored by the lab technician to make sure of proper disruption.

1.1.8 Sonicate the samples as follows: place the sonic probe into the sample beaker with the tip below the surface of the solvent but above the soil layer. Set the timer for 3 minutes. Power output should be 4-8 depending on matrix. Adjust mode to pulse and duty cycle to 70%.

1.1.9 Once the timer shuts off, remove the beaker, and pour the extraction solvent through the 70 mm glass funnel into corresponding labeled Kurderna-Danish flask.

1.1.10 Repeat steps 2.1.7-2.1.8 two more times using approximately 90 mL of 70:30 hexane/methylene chloride each time.

2. Hydrolysis:

2.1. To 35ml of organic free water add approximately 5 mL of 37% KOH solution. Add this solution and 4-5 boiling chips to each Kuderna-Danish flask. Attach a three ball Snyder column to the flask and place on a water bath set at 100℃.

2.2. Concentrate until boiling starts rapidly (approximately 10 minutes). Remove the Snyder column and let hydrolyze approximately 10-15 minutes more. Periodically, swirl the flask to enhance the solvent evaporation

2.3. When most of the solvent has evaporated, remove the flask from the water bath and allow to cool in the rack. After cooling, (There should be no more than ¼" of solvent left on top of the water. If more than ¼", add 2 more boiling chips and put KD back on the water bath).

3. Clean-up:

3.1. Pour the basic hydrolyzed water and solvent mix from the KD flask into a 250 mL separatory funnel which has been labeled with the sample ID. Rinse the flask two times with approximately 20ml of deionized water and add the rinsate to the corresponding separatory funnel.

3.2. Verify that the pH of the extract is >12. Dip the pointed end of a disposable 5-¾" pipette into the extract and verify with narrow range pH paper. Add 10 N NaOH solution, drop by drop if necessary, and note this on the Analyst Notes form.

3.3. Add 60 mL of methylene chloride to the sample. Seal and shake the separatory funnel vigorously for two minutes, periodically venting. Allow the organic phases to separate. Drain the methylene chloride (bottom layer) into a waste beaker. (If an emulsion is present it must be broken up by whatever mechanical means is necessary.) If the sample is tri-phasic, add an additional 30mL of methylene chloride and shake again. Repeat if the methylene chloride is pulling out any obvious color.

4. Back Extraction:

4.1. Acidify the sample to pH <2 with 3 pipettes of 1:1 sulfuric acid solution. Mix well. Use narrow-range pH paper to check the pH.

4.2. Prepare a labeled 250 ml Erlenmeyer flask for each sample.

4.3. Add 60ml of 7:3 methylene chloride/ether to the separatory funnel and shake for two minutes, venting frequently. Drain the bottom layer, (methylene chloride), into the corresponding Erlenmeyer flask. Extract the sample two more times using 60 ml of 7:3 methylene chloride/hexane. Each time the bottom layer should be drained into the corresponding Erlenmeyer flask.

4.4. Discard the water in the separatory funnel and transfer the sample label to the 250 ml Erlenmeyer containing the extract. Add approximately 10 gm of acidified (Herb) sodium sulfate. Additional acidified (Herb) sodium sulfate may be added to the extract in sufficient quantity so that some sodium sulfate remains "free flowing". Allow the extract to stand for one hour to remove any water in the solvent before KD concentration.

4.5. Transfer the extract to a KD. Rinse Erlenmeyer flask 2-3 times with hexane. Add rinsate to the KD.

4.5.1 Add 40mL Hexane to the KD and put on a water bath set at 100ºC.

4.5.2 Concentrate extract to 5-8mL. Place in KD rack and allow to cool for 10 minutes.

4.5.3 Transfer the extract to a labeled Turbo Tube. Rinse the KD with approximately 5mL of Hexane.

4.5.4 Concentrate the extract to Volume to Lab listed on the bench sheet. See Appendix 20.2.9, Task Instructions (TI-009) Turbo Vap concentration.

5. Derivitization procedures: for Herb and PCP are derivitized in the GC lab and are detailed in GC SOP's:

5.1. Herbicide (405S)

5.2. Phenol (412S)

Task Instructions (TI-019)

Vialing of final extract
Refer to bench sheet for options or requirements.

1. Vialing of final extract

1.1 Once the extract reaches the specified volume to lab for analysis, further clean-up, or specified procedure listed on the bench sheet, transfer the extract using the appropriate syringe and solvent into an appropriate vial. Clean the syringe between extracts by rinsing three times with the appropriate solvent.

1.2 The extract is ready for analysis.

Task Instructions (TI-020)

Reagents and Preparation of Solutions by parameter and matrix:

Preparation of the solutions is documented in the Reagent Preparation Logbook (Form 10076F)

Refer to bench sheet for options or requirements.

1. Pesticide or PCB by Separatory Funnel: Reagents and Solutions for Aqueous extractions

1.1. Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

1.2. Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

1.3. (In house and TCLP): Methylene Chloride, Fisher (D 154-4) or equivalent

1.4. (In house and TCLP): Hexanes, Fisher Optima (H303-4) or equivalent

1.5. (MTCA and Low Level): Methylene Chloride, EMD Omnisolv (DX0831-1) or equivalent

1.6. (MTCA and Low Level): Hexanes, EMD Omnisolv (HX0298-1) or equivalent

1.7. Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours

1.8. Neutral glasswool: prepared by baking in kiln at 400º C for 4 hours

1.9. (PCB acid clean): Concentrated sulfuric acid, Fisher (A300-500) or equivalent

1.10. (Pest or PCB Sulfur Clean): Tetrabutylammonium sulfite (TBAS) reagent:

1.10.1 Tetrabutylammonium hydrogensulfate, ACS Grade, Sigma Aldrich (155837-1KG)

1.10.2 Sodium sulfite, Anhydrous, Fisher, (S430-500)

1.10.3 Prepare (TBAS) reagent by dissolving 3.4 grams tetrabutylammonium hydrogensulfate in 100 mL of organic-free Type 1 water from ARI's water purification system. Extract the resulting solution with Pesticide Grade Hexane (20mL Hexane per 100mL reagent) to remove possible impurities. Discard the hexane extracts. Add 25 grams sodium sulfite to the 100 mL aqueous reagent. The resulting solution is saturated with sodium sulfite. Store the reagent in an amber glass bottle with a PTFE-lined cap. Clearly label the bottle including date prepared, preparer's initials, and the expiration date (30 days after preparation).

1.11. (Pest only): Ethyl Acetate, Fisher, (E191-4)

1.12. Silica Gel (SPE) darts, 3mL/500mg, Supelco Discovery

2. *Pesticide or PCB by Sonication or Microwave: Reagents and Solutions for Soil/Sediment extractions*

1.1 Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

1.2 Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

1.3 (In house and PSDDA): Acetone, EMD (AX0120-8) or equivalent

1.4 (In house and PSDDA): Hexanes, Fisher Optima (H303-4) or equivalent

1.5 (In house and PSDDA): Methylene Chloride, Fisher (D 154-4) or equivalent

1.5.1 80:20 Hexane/Acetone: prepared by mixing 3200mL Hexane/800mL Acetone

1.5.2 50:50 Hexane/Methylene Chloride: prepared by mixing 2000mL Hexane/2000mL Methylene Chloride

1.6 Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours

1.7 Neutral glasswool: prepared by baking in kiln at 400º C for 4 hours

1.8 (PCB acid clean): Concentrated sulfuric acid, Fisher (A300-500) or equivalent

1.9 (Pest or PCB Sulfur Clean): Tetrabutylammonium sulfite (TBAS) reagent:

1.9.1 Tetrabutylammonium hydrogensulfate, ACS Grade, Sigma Aldrich (155837-1KG)

1.9.2 Sodium sulfite, Anhydrous, Fisher, (S430-500)

1.9.3 Prepare (TBAS) reagent by dissolving 3.4 grams tetrabutylammonium hydrogensulfate in 100 mL of organic-free Type 1 water from ARI's water purification system. Extract the resulting solution with Pesticide Grade Hexane (20mL Hexane per 100mL reagent) to remove possible impurities. Discard the hexane extracts. Add 25 grams sodium sulfite to the 100 mL aqueous reagent. The resulting solution is saturated with sodium sulfite. Store the reagent in an amber glass bottle with a PTFE-lined cap. Clearly label the bottle including date prepared, preparer's initials, and the expiration date (30 days after preparation).

1.10 (Pest only): Ethyl Acetate, Fisher, (E191-4)

1.11 Silica Gel (SPE) darts, 3mL/500mg, Supelco Discovery

2. SIM OP Pesticide by Separatory Funnel: Reagents and Solutions for Aqueous extractions

2.1. Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

2.2. Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

2.3. Methylene Chloride, Fisher (D 154-4) or equivalent.

2.4 SIM OP Pesticide by Microwave: Reagents and Solutions for Soil/Sediment extractions

2.5 Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

2.6 Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours.

2.7 (In house): Methylene Chloride, Fisher (D 154-4) or equivalent.

2.8 Neutral glasswool: prepared by baking in kiln at 400º C for 4 hours

3. 8041 PCP by Separatory Funnel: Reagents and Solutions for Aqueous extractions

3.1. Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

3.2. Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

3.3. 1:1 sulfuric acid solution: prepared by diluting 500mL of concentrated sulfuric acid into 500mL of organic free water. **Use extreme caution this process generates heat. Add the acid to a volume of water. Do not add water to a volume of acid.**

3.4. (In house and Low Level): Methylene Chloride, Fisher (D 154-4) or equivalent

3.5. (In house and Low Level): Hexanes, Fisher Optima (H303-4) or equivalent

3.6 8041 PCP by Sonication: Reagents and Solutions for Soil/Sediment extractions

3.7 Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

3.8 Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours.

3.9 Concentrated Sulfuric Acid.

3.10 (In house): Methylene Chloride, Fisher (D 154-4) or equivalent.

3.11 (In house): Hexanes, Fisher Optima (H303-4) or equivalent

4. Herbicide by Separatory Funnel: Reagents and Solutions for Aqueous extractions

4.1. Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

4.2. Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

4.3. 10N sodium hydroxide prepared by dissolving 400g of sodium hydroxide pellets into organic free water and adjusting the volume to 1.0 Liter. **Use extreme caution this process generates heat. Add the NaOH slowly over a period of time to allow the heat to dissipate.**

4.4. (In house-Low Level-TCLP): Methylene Chloride, Fisher (D 154-4) or equivalent

4.5. (In house-Low Level-TCLP): Hexanes, Fisher Optima (H303-4) or equivalent

4.6. Ethyl Ether, Fisher Pesticide Grade (E199-4) or equivalent

4.7. 1:1 sulfuric acid solution: prepared by diluting 500mL of concentrated sulfuric acid into 500mL of organic free water. **Use extreme caution this process generates heat. Add the acid to a volume of water. Do not add water to a volume of acid.**

4.8. 70:30 Methylene Chloride/Ether: prepared by mixing 2800mL Methylene Chloride/1200mL Ether

4.9. 70:30 Methylene Chloride/Hexane: prepared by mixing 2800mL Methylene Chloride/1200mL Hexane

4.10. Acidified (Herb) sodium sulfate:

4.10.1 One 32oz jar of neutral Sodium Sulfate (with 1" headspace)

4.10.2 300mL Ethyl Ether

4.10.3 1mL concentrated Sulfuric Acid

4.10.4 Prepare by using one 32oz jar (with 1" headspace) of kiln baked sodium sulfate.

4.10.5 Under a hood, measure 300mL of Ethyl Ether into a 500mL graduated cylinder.

4.10.6 Measure 1mL of concentrated sulfuric acid into a 1mL volumetric flask.

4.10.7 Using a pipette, transfer the sulfuric acid to the 500mL graduated cylinder containing the Ethyl Ether. Use 1mL of the Ether/Acid mixture to rinse the volumetric flask once and add the rinsate to the 500mL graduated cylinder containing the Ether/Acid solution.

4.10.8 Mix the Ether/Acid solution well by swirling.

4.10.9 Slowly pour the Ether/Acid solution into the 32oz jar of sodium sulfate. Tighten lid on the 32oz jar and shake until homogenized and the sulfate is saturated.

4.10.10 Pour the acidified sodium sulfate into a clean Pyrex baking dish and spread evenly.

4.10.11 Nest the Pyrex dish with acidified sodium sulfate on top of a empty Pyrex dish and place on a hot plate. Turn hot plate on to the lowest setting.

4.10.12 Cover acidified sodium sulfate dish loosely with Aluminum foil venting to a hood. Be sure to allow adequate ventilation for evaporation of Ether and allow to dry overnight.

4.10.13 After all Ethyl Ether has evaporated, transfer acidified sodium sulfate into 16oz jars and label as "Acidified Sulfate for Herbs" with Red and Pink tape.

4.11 Herbicide by Sonication: Reagents and Solutions for Soil/Sediment extractions

4.12 Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

4.13 Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

4.14 10N sodium hydroxide prepared by dissolving 400g of sodium hydroxide pellets into organic free water and adjusting the volume to 1.0 Liter. ***Use extreme caution this process generates heat. Add the NaOH slowly over a period of time to allow the heat to dissipate.***

4.15 Concentrated Hydrochloric Acid.

4.16 1:1 sulfuric acid solution: prepared by diluting 500mL of concentrated sulfuric acid into 500mL of organic free water. ***Use extreme caution this process generates heat. Add the acid to a volume of water. Do not add water to a volume of acid.***

4.17 (In house): Methylene Chloride, Fisher (D 154-4) or equivalent

4.18 (In house): Hexanes, Fisher Optima (H303-4) or equivalent

4.19 Ethyl Ether, Fisher Pesticide Grade (E199-4) or equivalent

4.20 50:50 Methylene Chloride/Ether: prepared by mixing 2000mL Methylene Chloride/2000mL Ether

4.21 70:30 Hexane/Methylene Chloride: prepared by mixing 2800mL Hexane/1200mL Methylene Chloride

4.22 70:30 Methylene Chloride/Ether: prepared by mixing 2800mL Methylene Chloride/1200mL Ether

4.23 70:30 Methylene Chloride/Hexane: prepared by mixing 2800mL Methylene Chloride/1200mL Hexane

4.24 Acidified (Herb) Sodium Sulfate (see herb separatory funnel for preparation)

5. TBT by Separatory Funnel: Reagents and Solutions for Aqueous extractions

5.1. Tropolone, 98%, Aldrich (T89702-25g) or equivalent

5.2. 0.02% Tropolone in Methylene Chloride solution: (1.06g Tropolone/4000mL Methylene Chloride).

5.3. Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

5.4. Sea salts, Sigma, (S9883-1KG) or equivalent

5.5. (Pore Water Blanks): Sea Water solution: (144g Sea Salt/4000mL Organic Free Water)

5.6. (In house and Pore Water): Methylene Chloride, Fisher (D 154-4) or equivalent

5.7. Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

5.8. Concentrated hydrochloric acid, Fisher (A144-212) or equivalent

5.9. 1:1 hydrochloric acid solution: prepared by diluting 500mL of concentrated hydrochloric acid into 500mL of organic free water. **Use extreme caution this process generates heat. Add the acid to a volume of water. Do not add water to a volume of acid.**

5.10. (In house and Pore Water): Hexanes, Fisher Optima (H303-4) or equivalent

5.11. 1.5 M Hexyl magnesium bromide in diethyl ether solution:

5.11.1 1.5L Ethyl Ether, Burdick & Jackson High Purity (107-1) or equivalent

5.11.2 330g 1-Bromohexane, 99%, AlfaAesar

5.11.3 50g Magnesium chips, 99.98%, Aldrich

5.11.4 Preparation of 1.5M Hexyl magnesium bromide solution: Obtain a large grey bin 1/3rd full of ice. Add cold tap water to 2/3rd full.

5.11.5 Weigh 50g of Magnesium chips into a 400mL beaker.

5.11.6 Weigh 330g 1-Bromohexane into a 600mL beaker.

5.11.7 Measure 1000mL of Ethyl Ether into a 1.0L graduated cylinder.

5.11.8 Add the 330g 1-Bromohexane to a 6000mL flat bottom flask with 24/40 joint at the top.

5.11.9 Add the 1000mL of Ethyl Ether to the same 6000mL flask.

5.11.10 Add the 50g Magnesium chips to the same 6000mL flask.

5.11.11 Add 24/40 joint condenser to the top of the 6000mL flask.

5.11.12 Swirl gently to activate reaction.

5.11.13 Place 6000mL flask on a hot plate to activate reaction. (When the reaction starts to turn yellow/brown and bubbles appear-the reaction has started).

5.11.14 Add an additional 500mL of Ethyl Ether to the flask. (Take off the condenser and pour through the top of the flask-NOT top of condenser.)

5.11.15 **CAUTION-STAY IN CONTROL OF THE REACTION during the early stages of the reaction. Use ice bath to control the reaction-But do not stall/stop the reaction! Place the 6000mL flask back and forth from hot plate to ice bath to control the reaction. The solvent should be around the fourth rung of the condenser. If it slows-put it back onto the hot plate. If it gets to the top of the condenser-put in back into the ice bath.**

5.11.16 When the reaction is in control….(after sufficient time in the ice bath). Put back onto hot plate to continue controlled reaction for approximately ½ hour. It may be necessary to turn hot plate “off” and “on”.

5.11.17 After the reaction is complete, take off of the hot plate and let cool for 1 hour.

5.11.18 Bottle reagent into 2oz amber bottles located in the GC cabinet.

5.11.19 Label with reagent ID.

5.11.20 Test the reagent by adding 100µL TBT surrogate and spike to 10mL of hexane. Concentrate to 4mL for derivitization. Perform the Derivitization, Hydrolyzation. Concentrate to 2mL for Alumina Clean-up. Concentrate to 0.5mL and vial for analysis in hexane.

5.12. Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours.

5.13. Neutral glasswool: prepared by baking in kiln at 400º C for 4 hours

5.14. Activity 0 Alumina (neutral alumina baked at 400℃ for 8 hours). Test the alumina with TBT alumina QA spike solution-(Use 100µl for a 0.5mL FEV). Add to 2mL hexane, run through 5g alumina, collect 3-4mL, concentrate to 0.5mL for analysis.

5.15 TBT by Sonication or Microwave: Reagents and Solutions for Soil/Sediment extractions

5.16 Tropolone, 98%, Aldrich (T89702-25g) or equivalent

5.17 0.10% Tropolone in Methylene Chloride solution: (5.25g Tropolone/4000mL Methylene Chloride).

5.18 Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

5.19 (In house): Methylene Chloride, Fisher (D 154-4) or equivalent

5.20 (In house): Hexanes, Fisher Optima (H303-4) or equivalent

5.21 Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

5.22 Concentrated hydrochloric acid, Fisher (A144-212) or equivalent

5.23 1:1 hydrochloric acid solution: prepared by diluting 500mL of concentrated hydrochloric acid into 500mL of organic free water. ***Use extreme caution this process generates heat. Add the acid to a volume of water. Do not add water to a volume of acid.***

5.24 Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours.

5.25 Neutral glasswool: prepared by baking in kiln at 400º C for 4 hours

5.26 Activity 0 Alumina (neutral alumina baked at 400℃ for 8 hours). Test the alumina with TBT alumina QA spike solution-(Use 100µl for a 0.5mL FEV). Add to 2mL hexane, run through 5g alumina, collect 3-4mL, concentrate to 0.5mL for analysis.

5.27 1.5 M Hexyl magnesium bromide in diethyl ether solution:: (See TBT by Separatory Funnel for preparation).

6. EPH by Separatory Funnel: Reagents and Solutions for Aqueous extractions

6.1. Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

6.2. Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

6.3. Concentrated sulfuric acid, Fisher (A300-500) or equivalent

6.4. 1:1 sulfuric acid solution: prepared by diluting 500mL of concentrated sulfuric acid into 500mL of organic free water. **Use extreme caution this process generates heat. Add the acid to a volume of water. Do not add water to a volume of acid.**

6.5. (In house): Methylene Chloride, Fisher (D 154-4) or equivalent

6.6. Pentane, Fisher HPLC grade, (P399-4) or equivalent

6.7. (In house): Hexanes, Fisher Optima (H303-4) or equivalent

6.8. 90:10 Pentane/Hexane: prepared by mixing 3600mL Pentane/400mL Hexane

6.9. 60:40 Pentane/Methylene Chloride: prepared by mixing 2400mL Pentane/1600mL Methylene Chloride

6.10. Neutral glasswool: prepared by baking in kiln at 400º C for 4 hours

6.11. Silica Gel, 60-200 mesh, Fisher, (S704-10) or equivalent

6.12. 0% Silica Gel (activated by baking in kiln at 400º C for 8 hours). Test the silica gel by adding 100µL EPH surrogate and spike solutions to 2mL of hexane. Elute and collect 1mL increments and checked by OE GC for proper Aliphatic/Aromatic elution volumes.

6.12 EPH by Sonication: Reagents and Solutions for Soil/Sediment extractions

6.13 Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

6.14 (In house): Methylene Chloride, Fisher (D 154-4) or equivalent

6.15 Pentane, Fisher HPLC grade, (P399-4) or equivalent

6.16 (In house): Hexanes, Fisher Optima (H303-4) or equivalent

6.17 90:10 Pentane/Hexane: prepared by mixing 3600mL Pentane/400mL Hexane

6.18 60:40 Pentane/Methylene Chloride: prepared by mixing 2400mL Pentane/1600mL Methylene Chloride

6.19 Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours.

6.20 Neutral glasswool: prepared by baking in kiln at 400º C for 4 hours

6.21 Silica Gel, 60-200 mesh, Fisher, (S704-10) or equivalent

6.22 0% Silica Gel (activated by baking in kiln at 400º C for 8 hours). Test the silica gel by adding 100µL EPH surrogate and spike solutions to 2mL of hexane. Elute and collect 1mL increments and checked by OE GC for proper Aliphatic/Aromatic elution volumes.

7. NWTPHD/HCID by Separatory Funnel: Reagents and Solutions for Aqueous extractions

7.1. Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

7.2. Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

7.3. Concentrated sulfuric acid, Fisher (A300-500) or equivalent

7.4. 1:1 sulfuric acid solution: prepared by diluting 500mL of concentrated sulfuric acid into 500mL of organic free water. **Use extreme caution this process generates heat. Add the acid to a volume of water. Do not add water to a volume of acid.**

7.5. (In house): Methylene Chloride, Fisher (D 154-4) or equivalent

7.6. Silica Gel, 60-200 mesh, Fisher, (S704-10) or equivalent

7.7. 0% Silica Gel (activated by baking in kiln at 400º C for 8 hours).

7.8 NWTPHD/HCID by Sonication or Microwave: Reagents and Solutions for Soil/Sediment extractions

7.9 Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

7.10 Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours.

7.11 Neutral glasswool: prepared by baking in kiln at 400º C for 4 hours

7.12 Concentrated sulfuric acid, Fisher (A300-500) or equivalent

7.13 1:1 sulfuric acid solution: prepared by diluting 500mL of concentrated sulfuric acid into 500mL of organic free water. ***Use extreme caution this process generates heat. Add the acid to a volume of water. Do not add water to a volume of acid.***

7.14 (In house): Methylene Chloride, Fisher (D 154-4) or equivalent

7.15 Silica Gel (SPE) darts, 3mL/500mg, Supelco Discovery

8. AK102/103 by Separatory Funnel: Reagents and Solutions for Aqueous extractions

8.1. Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

8.2. Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

8.3. Concentrated hydrochloric acid, Fisher (A144-212) or equivalent

8.4. 1:1 hydrochloric acid solution: prepared by diluting 500mL of concentrated hydrochloric acid into 500mL of organic free water. **Use extreme caution this process generates heat. Add the acid to a volume of water. Do not add water to a volume of acid.**

8.5. (In house): Methylene Chloride, Fisher (D 154-4) or equivalent

8.6. Silica Gel, 60-200 mesh, Fisher, (S704-10) or equivalent

8.7. 0% Silica Gel (activated by baking in kiln at 400º C for 8 hours).

8.8 AK102/103 by Sonication or Microwave: Reagents and Solutions for Soil/Sediment extractions

8.9 Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

8.10 Neutral glasswool: prepared by baking in kiln at 400º C for 4 hours

8.11 Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours.

8.12 (In house): Methylene Chloride, Fisher (D 154-4) or equivalent

8.13 Silica Gel (SPE) darts, 3mL/500mg, Supelco Discovery

9. BAN by Separatory Funnel: Reagents and Solutions for Aqueous extractions

9.1. Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

9.2. Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

9.3. Concentrated sulfuric acid, Fisher (A300-500) or equivalent

9.4. 1:1 sulfuric acid solution: prepared by diluting 500mL of concentrated sulfuric acid into 500mL of organic free water. **Use extreme caution this process generates heat. Add the acid to a volume of water. Do not add water to a volume of acid.**

9.5. (In house and TCLP): Methylene Chloride, Fisher (D 154-4) or equivalent

9.6. Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours.

9.7. Sodium hydroxide, Fisher, (S318-500) or equivalent

9.8. 10N sodium hydroxide prepared by dissolving 400g of sodium hydroxide pellets into organic free water and adjusting the volume to 1.0 Liter. **Use extreme caution this process generates heat. Add the NaOH slowly over a period of time to allow the heat to dissipate.**

9.9 BAN (In house/PSDDA/SIM) by Sonication: Reagents and Solutions for Soil/Sediment extractions

9.10 Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

9.11 Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours.

9.12 (In house): Methylene Chloride, Fisher (D 154-4) or equivalent

9.13 (In house): Acetone, EMD (AX0120-8) or equivalent

9.14 50:50 Methylene Chloride/Acetone prepared by mixing prepared by mixing 2000mL Methylene Chloride/2000mL Acetone

9.15 Pre-deactivated anhydrous sodium sulfate:

9.15.1 One 32oz jar of neutral sodium sulfate

9.15.2 100mL Acetone

9.15.3 1mL Organic Free Water

9.15.4 Ten drops concentrated HCL

9.15.5 Prepare by using one 32oz jar (with 1" headspace) of kiln baked sodium sulfate.

9.15.6 <u>Under a hood</u>, using a pipette, add ten drops of concentrated HCL to 1mL of Organic Free Water.

9.15.7 Measure 100mL of Acetone into a graduated cylinder.

9.15.8 Add the acidified water to the Acetone and mix well.

9.15.9 Add the acidified Acetone solution to the 32oz jar of sodium sulfate. Mix well by shaking until homogenized and the sulfate is saturated.

9.15.10 Pour the acidified sodium sulfate into a clean pyrex dish and spread evenly.

9.15.11 Cover acidified sodium sulfate dish loosely with Aluminum foil venting to a hood. Be sure to allow adequate ventilation for evaporation of Acetone allow to dry overnight.

9.15.12 After all Acetone has evaporated, transfer acidified sodium sulfate into a 1.0L Amber Boston Round Wide Mouth Bottle and label "Pre-deactivated sodium sulfate for BAN".

9.16 Pre-deactivated glass wool.

9.16.1 One Designated Grey Bin (Above Sep Funnel Hood)

9.16.2 Green dishwashing gloves (clean)

9.16.3 8.0L Organic Free Water

9.16.4 100mL Concentrated HCL

9.16.5 Six 32oz jars Neutral glass wool

9.16.6 Two clean Pyrex dishes

9.16.7 One hot plate

9.16.8 Aluminum Foil

9.16.9 Prepare by adding approximately 8.0L Organic Free Water to designated Grey Bin (1/2 full).

9.16.10 Add 100mL concentrated HCL to the water in the bin. Mix Well.

9.16.11 Use six 32oz jars of neutral glasswool that has been baked in the kiln. Put glass wool in the acid bath and soak for a few seconds. Make sure it all gets saturated.

9.16.12 Wring the glass wool out well. Pull apart as much as possible and place into a clean Pyrex dish.

9.16.13 Nest glass wool Pyrex dish on top of another empty Pyrex dish. Set on hot plate set to the lowest setting. Tent dish with Aluminum Foil venting to a hood. Make sure the hood is drawing air over the glass wool.

9.16.14 Leave overnight or until dry.

9.16.15 Place dry glass wool into 16oz clear jars and label "Pre-deactivated glass wool for BAN"

9.17 BAN (In house) by Microwave: Reagents and Solutions for Soil/Sediment extractions

9.18 Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

9.19 Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours.

9.20 (In house): Methylene Chloride, Fisher (D 154-4) or equivalent

9.21 Pre-deactivated anhydrous sodium sulfate: (See BAN sonication for preparation).

9.22 Neutral glasswool: prepared by baking in kiln at 400º C for 4 hours

18. BAN and 1,4 Dioxane by Continuous Liquid Liquid: Reagents and Solutions for Aqueous extractions

18.1 Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

18.2 Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

18.3 Concentrated sulfuric acid, Fisher (A300-500) or equivalent

18.4 1:1 sulfuric acid solution: prepared by diluting 500mL of concentrated sulfuric acid into 500mL of organic free water. **Use extreme caution this process generates heat. Add the acid to a volume of water. Do not add water to a volume of acid.**

18.5 (In house): Methylene Chloride, Fisher (D 154-4) or equivalent

19 (8270) PNA and In house SIM PNA by Separatory Funnel and Continuous Liquid Liquid: Reagents and Solutions for Aqueous extractions

19.1 Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

19.2 Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

19.3 (In house): Methylene Chloride, Fisher (D 154-4) or equivalent

19.4 (In house): Hexanes, Fisher Optima (H303-4) or equivalent

19.5 Silica Gel (SPE) darts, 3mL/500mg, Supelco Discovery

9.9. (8270) PNA and SIM PNA by Sonication: Reagents and Solutions for Soil/Sediment extractions

9.10. Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

9.11. Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours.

9.12. Neutral glasswool: prepared by baking in kiln at 400º C for 4 hours

9.13. (In house): Hexanes, Fisher Optima (H303-4) or equivalent

9.14. (In house): Methylene Chloride, Fisher (D 154-4) or equivalent

9.15. (In house): Acetone, EMD (AX0120-8) or equivalent

9.16. 50:50 Methylene Chloride/Acetone prepared by mixing prepared by mixing 2000mL Methylene Chloride/2000mL Acetone

9.17. Silica Gel (SPE) darts, 3mL/500mg, Supelco Discovery

9.18.

9.19. (8270) PNA and SIM PNA by Microwave: Reagents and Solutions for Soil/Sediment extractions

9.20. Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

9.21. Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours.

9.22. Neutral glasswool: prepared by baking in kiln at 400º C for 4 hours

9.23. (In house): Hexanes, Fisher Optima (H303-4) or equivalent

9.24. (In house): Methylene Chloride, Fisher (D 154-4) or equivalent

9.25. Silica Gel (SPE) darts, 3mL/500mg, Supelco Discovery

20 Low Level SIM PNA by Separatory Funnel and Continuous Liquid Liquid: Reagents and Solutions for Aqueous extractions

20.1 Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

20.2 Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

20.3 (Low Level): Methylene Chloride, EMD Omnisolv (DX0831-1) or equivalent

20.4 (Low Level): Hexanes, EMD Omnisolv (HX0298-1) or equivalent

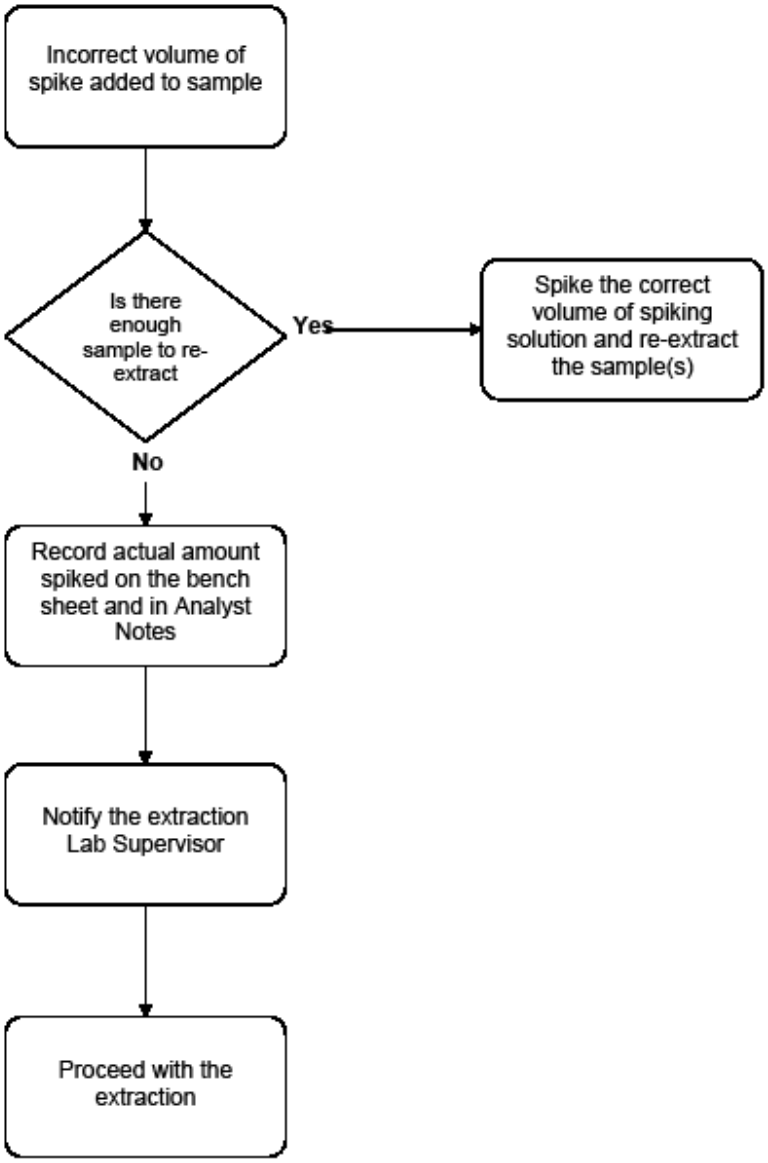
20.5 Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours.

Silica Gel (SPE) darts, 3mL/500mg, Supelco Discovery

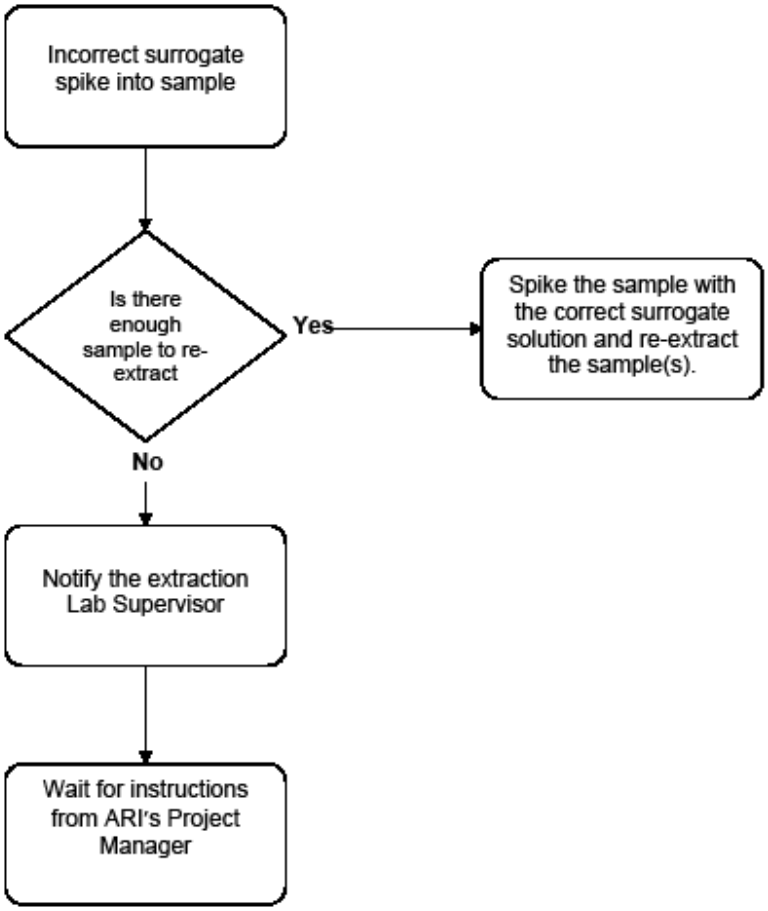
Appendix 20.3

Corrective Action Charts

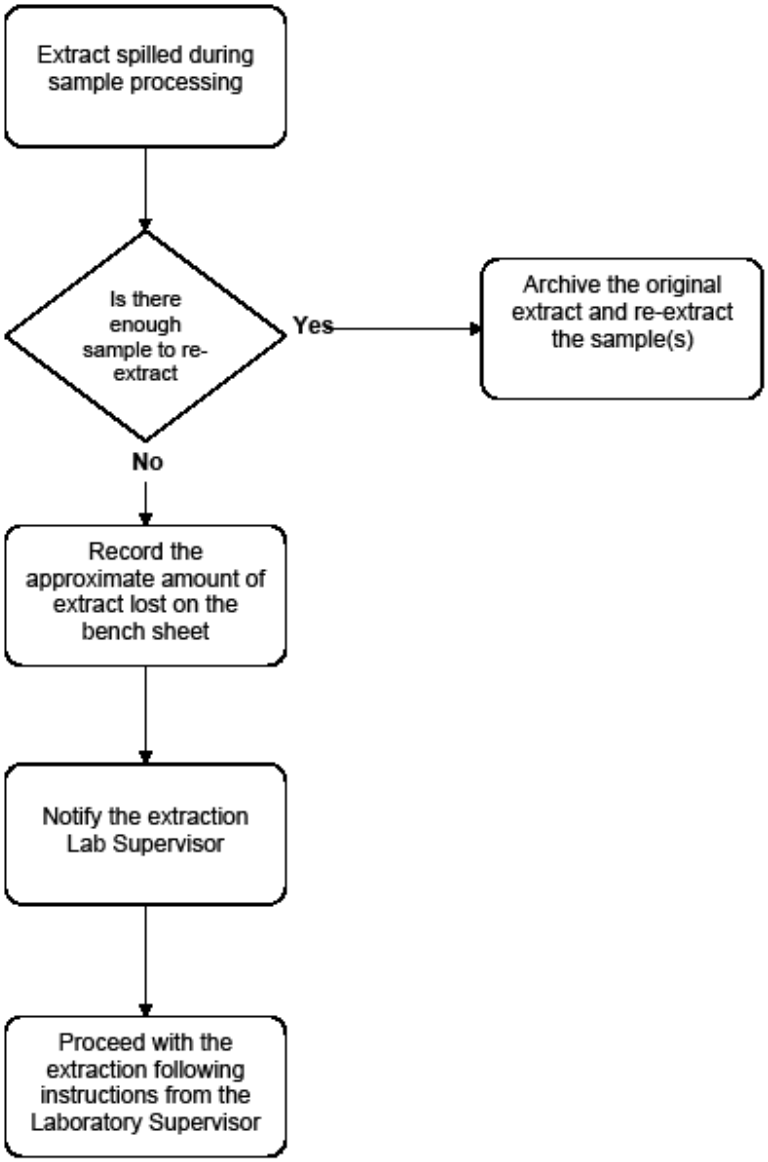
Corrective Action for Incorrect Spike Volume



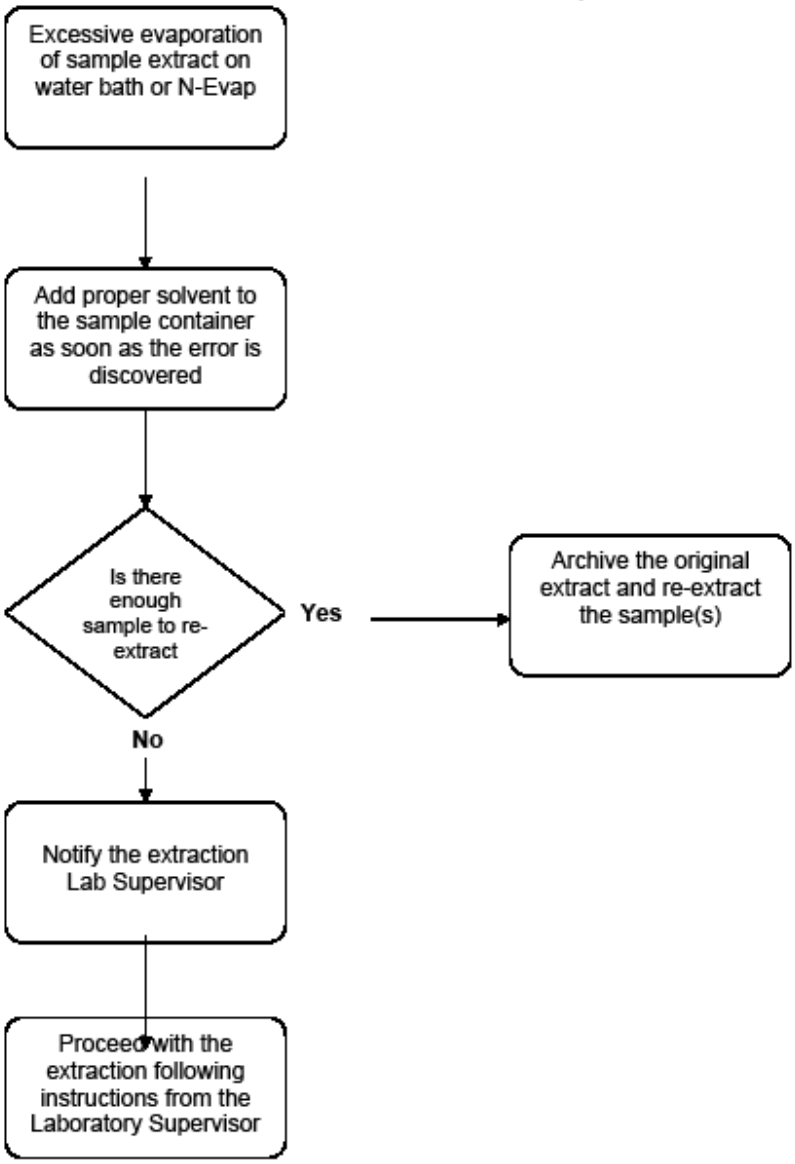
Corrective Action for Incorrect Surrogate Addition



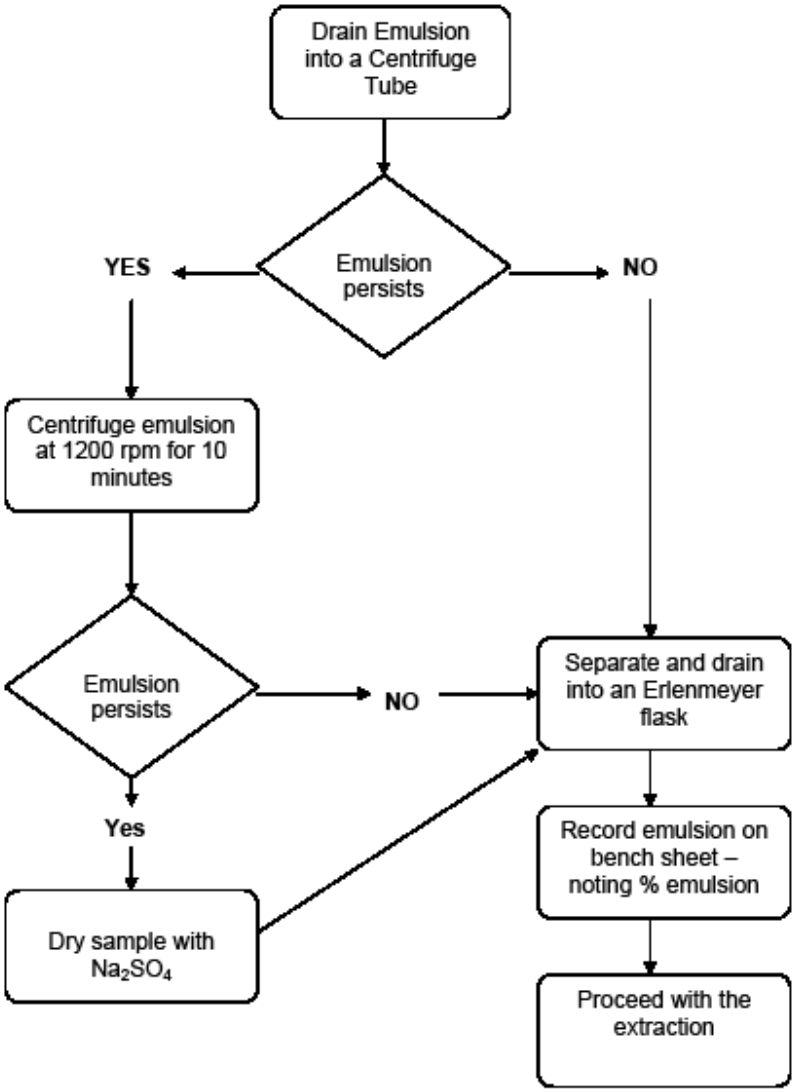
Corrective Action for an Extract Spill



Corrective Action for Loss of Sample on Water Bath or Turbo-Vap



Corrective Action for Emulsions



Appendix 20.4

Forms

Analytical Resources, Incorporated
Analytical Chemists and Consultants

Organic Extractions Laboratory
Analyst Notes

ARI Job No.: _____ Client ID: _____

Parameter: Client Project:

Note problems, concerns, corrective actions	Analyst/Date
Screens: Soil/Sediment/Solid/Other:	
☐ **No Anomalies (standard soil/sediment)**	
☐ **Wet sediment/sludge=**	
☐ **Standing Water Decanted=**	
☐ **Standing Water Homogenized (Shared samples)=**	
☐ **Clay (Difficult to homogenize/Mixed with Kitchen Aid)=**	
☐ **Rocks/Organics=**	
☐ **Oily, obvious fuel/sulfur odors=**	
☐ **Other (Details)=**	
Aqueous:	
☐ **No Anomalies**	
☐ **Turbid/Color=**	
☐ **Particulates=**	
☐ **Emulsions=**	
☐ **Other (Details)=**	
☐ **Other Notes/Comments=**	

3056F

Revision 007
02/25/10

Appendix 20.5

Bench Sheets

Analytical Resources, Incorporated
Analytical Chemists and Consultants

Standard Operating Procedure

Extraction of Aqueous Samples using Separatory Funnel or Continuous Liquid-Liquid Extraction (EPA Methods 3510C and 3520C)

SOP 3311S
Revision 000
Revision Date: 3/12/10
Effective Date: 3/12/10

Prepared By:

Tarry Hawk

Approvals:

Laboratory / Section Manager

Quality Assurance

4611 South 134th Place, Suite 100 • Tukwila WA 98168 • **206-695-6200** • **206-695-6201** fax

Annual Review

SOP Number:	3311S
Title:	Extraction of Aqueous Samples Using Separatory Funnel or Continuous Liquid-Liquid Extraction (EPA Methods 3510C and 3520C)

The ARI employee named below certifies that this SOP is accurate, complete and requires no revisions

Name	Reviewer's Signature	Date

Standard Operating Procedure 3311S - Extraction of Aqueous Samples Using Separatory Funnel or Continuous Liquid-Liquid Extraction (EPA Methods 3510C and 3520C)

1. Scope and Application

1.1. This document describes the procedures used at ARI to perform solvent extraction of aqueous environmental samples for semi-volatile organics analyses. Aqueous samples include ground water, surface water, marine water, industrial wastewater, pore water and TCLP extracts. The procedures described are used for GC and GC-MS analyses listed in Appendix 20.1. Procedures in this document are based on EPA Methods 3500C, 3510C and 3520C. Samples are extracted by either separatory funnel or continuous liquid-liquid extractors. Sample volumes, extraction solvents and extract final volumes vary depending on client or project specific requirements.

1.2. This document provides an outline of procedures and/or tasks for each analysis listed in Appendix 20.1. The specific requirements for each analysis are provided in a Bench Sheet. A unique Bench Sheet is available for each analysis. Bench Sheets define the extraction protocol and are used to document the extraction procedure. Appendix 20.3 includes all current Bench Sheets applicable to this SOP.

1.3. An outline or brief summary of the tasks required to complete the analyses in Appendix 20.1 are provided in this document. Step by step instructions are documented in the task specific "Task Instructions" (TI-001-TI-020) reproduced in Appendix 20.2. Training of Organic Extractions Analysts is based on the Analytical Bench Sheets and Task Instructions. Analysts proficiency is documented for each Task Instruction.

2. Summary of the Procedure

2.1. A specified volume of aqueous sample is measured into a separatory funnel or continuous liquid-liquid extractor and the pH is adjusted as necessary. Samples are spiked with analysis specific surrogate standards and extracted with a specified organic solvent.

2.2. The extracts are concentrated to a designated final volume using a Kuderna-Danish (KD) concentrator apparatus on a water bath and/or a turbo-vap system or a dry vap system.

2.3. Extracts may be subjected to method specific solvent exchange, cleanup or separation procedures before they are relinquished to the GC laboratory for analysis.

2.4. Project specified Quality Assurance samples are prepared and extracted using identical procedures.

3. Definitions

3.1. Final Effective Volume (FEV) –

3.2. Laboratory Control Sample (LCS) – A sample matrix, free from the analytes of interest, spiked with verified amounts of analyes or a material containing known amounts of analytes. It is generally used to establish intra-laboratory or analyst-specific precision or to assess the performance of all or a portion of the measurement system.

3.3. Matrix Spike (MS)- A sample prepared by adding a known mass of target analyte to a specified amount of sample matrix for which an independent estimate of target analyte concentration is available. Matrix spikes are used, to determine the effect of the sample matrix on a method's recovery efficiency.

3.4. Matrix Spike Duplicate (MSD)- A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

3.5. Method Blank (MB) – A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

3.6. Surrogate – A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

3.7. TCLP – Toxicity Characteristic Leaching Procedure as defined by EPA SW-846 Method 1311.

3.8. Sulfuric Acid/Silica gel for TPH-D: clean-up can be used to remove naturally occurring non-petroleum organics (e.g. leaf litter, bark, etc.), which may contribute biogenic interferences and assist in the reduction or elimination of these interferences. Acid Clean-up procedure is strictly designated for TPH-D analyses. This method of clean up is not suitable for AK-102, AK-103 or HCID analyses.

3.9. Silica-gel - A regenerative adsorbent with weakly acidic properties which can be used in column chromatography for the separation of analytes from interfering compounds.

3.10. Sulfuric Acid Clean up for PCB: A rigorous clean up process used in the removal of most organic chemicals prior to analysis of PCB's. It does not destroy chlorinated benzene or chlorinated naphthalene.

3.11. Tetra Butyl Ammonium Sulfite (TBAS)- reagent used to remove elemental sulfur from solvent extracts. Preparation of the reagent is described in ARI SOP 334S Sulfur Removal.

4. Interferences

4.1. Standard solutions, solvents or supports materials must not contain extraneous compounds or other chemical interferences. All standards are verified by GC/ECD, GC-FID or GC/MS prior to use. All solvent lots and support materials are checked for purity prior to use.

4.2. Glassware used in the procedure is cleaned and kiln fired as described in ARI SOP 301S. Glassware is rinsed with clean solvent just prior to use.

4.3. Extracting solvent may contain impurities that could interfere with analyses. Use solvents from a supplier's lot that have are verified free of such contaminates.

4.4. Laboratory supplies and equipment are potential sources of interfering contamination

4.5. Items such as gloves, bench paper, rubber stoppers should not come into contact with samples or extracts.

4.6. Airborne dust and other debris may contaminate samples. Samples and extracts must be covered at all times.

5. Safety

5.1. The toxicity or carcinogenicity of each reagent used in this SOP are not precisely defined. Treat each compound as a potential health hazard. Reduce exposure to all chemicals to the lowest possible level by whatever means available.

5.2. Always wear appropriate PPE (personal protective equipment) when working in the Organics Extraction Laboratory. Gloves, safety glasses, ear protection, lab coats, respirators, face shields, etc. are provided for your protection.

5.3. DO NOT attempt to cleanup solvent spills in the laboratory. Immediately evacuate the area and contact a member of the Emergency Response Team (ERT) for assistance.

5.4. Material Safety Data Sheets (MSDS) that outline hazards, exposure limits, treatments and regulatory guidelines are available for all chemicals used in this procedure and should be consulted when such information is required. The MSDS file is located in the central project management area. MSDS are also available online, at http//hazard.com/MSDS/.

5.5. Environmental Samples may contain hazardous material; treat them as potential health hazards.

5.6. Dispose of all unwanted, broken glassware into a broken glassware disposal box. Inspect every piece of glassware prior to use. Do not use glassware that is chipped, cracked, etched, or scratched. Set aside glassware with minor damage for repair.

5.7. Use nitrile gloves when working with organic solvents; latex gloves are not appropriate for working with methylene chloride or other solvents.

6. Equipment and Supplies

6.1. Equipment

6.1.1. Personal protective equipment (PPE)

6.1.1.1. Gloves

6.1.1.2. Laboratory coat

6.1.1.3. Goggles

6.1.2. Glass etcher / engraver

6.1.3. Centrifuge Beckmann GP or equivalent

6.1.4. Heating mantle, six place, Electrothermal Electromantle ME or equivalent

6.1.5. Portable Paint Shaker-Blair Turbo II or equivalent

6.1.6. RapidTrace Workstation-(SPE)

6.1.7. Water Bath, Precision Scientific or equivalent

6.1.8. Turbo Vap LV, Zymark, (set at 40° C)

6.1.9. Dry Vap concentrator system-Horizon

6.1.10. Vortex Mixer-Fisher Scientific or equivalent

6.1.11. Hamilton gastight syringes: 25µL, 50µL, 100µL, 250µL, 1.0mL, 5.0mL, and 10.0mL

6.2. Glassware

6.2.1. Graduated cylinder, glass, pre-cleaned graduated 'to contain'. One 100, 1000 or 2000 mL cylinder per sample depending on volume of ample to be extracted

6.2.2. Separatory funnel (SF), glass with PTFE stopper and stopcock plug (one for each sample)

6.2.2.1. 2000 mL SF for 1 Liter samples

6.2.2.2. 1000 mL SF for 500 mL samples

6.2.2.3. 250mL SF for 100 mL (TCLP) samples

6.2.3. Erlenmeyer flask, Glass, 250 mL (one for each sample)

6.2.4. Continuous liquid-liquid extractor: 1000 mL round-bottom flask, extractor body, condenser, and a clip ring.

6.2.4.1. 500 mL extractor body for SIM PNA

6.2.4.2. 1000 mL extractor body (with solvent saver insert) for (8270) BAN, (8270) PNA, and (8270) 1,4 Dioxane

6.2.5. Centrifuge bottle, glass, 200mL

6.2.6. 'Tilt-a-pet" dispenser, glass, 30 or 60 mL used to add a measured volume of extracting solvent to the sample in a separatory funnel

6.2.7. Pasteur Pipette, borosilicate glass, 5 ¾ inch, disposable

6.2.8. Glass stir rod

6.2.9. Kuderna-Danish (KD) concentrating apparatus, glass, 500mL, and three ball Snyder Column

6.2.10. EV Dry Vap tube, glass, 200mL

6.2.11. Turbo Tube, glass, 60mL

6.2.12. VOA vial, glass, 40mL, with PTFE lined screw cap

6.2.13. Scintillation vial, glass, 20mL, with foil lined screw cap

6.2.14. Centrifuge tube, borosilicate glass, 15mL, with PTFE lined screw cap

6.2.15. Disposable culture tube, borosilicate glass, 16 X 100mm

6.2.16. Medium Micro Column, glass, 16 X 297mm

6.2.17. Beaker, glass, 50mL

6.2.18. Vial, auto sampler, clear glass, 1.5mL with PTFE lined crimp cap

6.2.19. Vial, amber glass, 1.5mL with PTFE lined screw cap

6.3. Supplies

6.3.1. Wide Sharpie Permanent Pen

6.3.2. Boiling Chips-PTFE (Chemware)

6.3.3. Yellow, Pink, Red, Blue, Blue-Green, Purple, White, and Grey label tapes

6.3.4. Kim wipe

6.3.5. Narrow range pH paper

6.3.6. Rubber bulb, amber latex, 1mL

6.3.7. Filter, 0.20µm, (13mm PTFE), Whatman Puridisc

6.3.8. Filter, 0.45µm, (13-25mm PTFE), Whatman Puridisc

6.3.9. Filter, 0.45µm, GDX HPF (25mm), Whatman

7. Reagents and Standards for aqueous extractions: Reagents by Parameter, Matrix, Task Instruction, and Preparation of the solutions is located in Appendix 20.2, (TI-020)

7.1. Acetone, EMD (AX0120-8) or equivalent

7.2. Hexanes, Fisher Optima (H303-4) or equivalent

7.3. Hexanes, EMD Omnisolv (HX0298-1) or equivalent

7.4. Methylene Chloride, Fisher (D 154-4) or equivalent

7.5. Methylene Chloride, EMD Omnisolv (DX0831-1) or equivalent

7.5.1 70:30 Methylene Chloride/Hexane solution

7.5.2 70:30 Methylene Chloride/Ether solution

7.6. Pentane, Fisher HPLC grade, (P399-4) or equivalent

7.6.1. 90:10 Pentane/Hexane solution

7.6.2. 60:40 Pentane/Methylene Chloride solution

7.7. Ethyl Ether, Fisher Pesticide Grade (E199-4) or equivalent

7.8. Concentrated hydrochloric acid, Fisher (A144-212) or equivalent

7.8.1. 1:1 hydrochloric acid solution

7.9. Concentrated sulfuric acid, Fisher (A300-500) or equivalent

7.9.1. 1:1 sulfuric acid solution

7.10. Sodium hydroxide, Fisher, (S318-500) or equivalent

7.10.1. 10N sodium hydroxide solution

7.11. Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours

7.12. Acidified (Herb) sodium sulfate

7.13. Neutral glasswool: prepared by baking in kiln at 400° C for 4 hours

7.14. Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

7.15. Sea Water solution

7.15.1. Sea salts, Sigma, (S9883-1KG) or equivalent

7.16. 0.02% Tropolone in Methylene Chloride solution

7.16.1. Tropolone, 98%, Aldrich (T89702-25g) or equivalent

7.17. 1.5 M Hexyl magnesium bromide in diethyl ether reagent

7.17.1. 1-Bromohexane, 99%, AlfaAesar, 2500g

7.17.2. Magnesium, chips, 99.98%, Aldrich

7.17.3. Ethyl Ether, Burdick & Jackson High Purity (107-4) or equivalent

7.18. Tetrabutylammonium sulfite (TBAS) reagent

7.18.1. Tetrabutylammonium hydrogensulfate, ACS Grade, Sigma Aldrich (155837-1KG)

7.18.2. Sodium sulfite, Anhydrous, Fisher, (S430-500)

7.19. Ethyl Acetate, Fisher, (E191-4)

7.20. Silica Gel (SPE) darts, 3mL/500mg, Supelco Discovery

7.21. Silica Gel, 60-200 mesh, Fisher, (S704-10) or equivalent

7.21.1. 0% Silica Gel

7.22. Activity 0 Alumina

7.23. Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions. Preparation of the solutions is documented in the Reagent Preparation Logbook (Form 10076F)

8. Sample Collection, Preservation, Shipment and Storage

8.1. Required holding times for all water samples is 7 days from date and time of sample collection. Extract aqueous samples within seven days of collection. When holding times are or may be compromised inform your supervisor immediately.

8.2. TCLP and SPLP preparations are considered water samples with a 7 day holding time from date the leachate is filtered.

8.3. All aqueous samples are placed in bins in Refrigerator 05 between 2°C to 6°C and recorded in the Refrigerator 05 Logbook by Sample Receiving staff. When retrieving samples from

Refrigerator 05 you must record your action in the Logbook. Allow samples to equilibrate to ambient temperature prior to extraction.

8.4. Following extraction, waters are archived in walk-in Refrigerator 01 and stored between 2°C to 6°C until disposal date.

8.5. Some samples are shared with the metals and or the conventionals laboratories. These samples are placed in a share bin in Refrigerator 36. Procedures for sharing samples are outlined in SOP 1019S.

9. Quality Control

9.1. Quality Control requirements are specified on the analysis specific bench sheet

9.2. Generally, all samples are spiked with surrogate standard(s), and each extraction batch of 20 or fewer samples include a Method Blank (MB) and a Lab Control Sample (LCS).

9.3. When requested by ARI's client's or required by published analytical protocol other QA samples such as Matrix Spikes, Replicates, Standard Reference Materials are prepared and extracted.

9.4. To verify that surrogate and matrix spikes is accurate; all spike additions are witnessed and documented by a second laboratory technician.

9.5. QC samples including Method Blanks and Laboratory Control Samples (LCS) must be subjected to the clean-up procedures along with any samples that require that technique.

9.6. Prior to beginning the extraction process, determine what procedures are required by reviewing the bench sheet. Follow all information listed for extraction volumes, surrogate and spike additions, pH requirements and special instructions listed at the bottom of all bench sheets. Bench sheet Forms are listed in Appendix 20.1 and examples are contained in Appendix 20.3.

9.7. Proceed slowly and carefully while extracting samples. Note all problems, concerns, errors or deviations from the standard procedure on an Analyst Notes Form (Form 3056F). What seem to be minor errors or deviations may have a significant impact on the final results. Analyst must report all deviations from standard procedures to the laboratory supervisor as soon as possible. Failure to do so may result in a disciplinary process.

9.8. Label glassware and vials with permanent markers.

9.9. Verify sample identification is transferred correctly when transferring or vialing samples and extracts.

9.10. The LIMS Data entry person will review all bench sheets before distribution.

9.11. The laboratory supervisor reviews all logbooks for completeness and accuracy monthly.

9.12. The QA section periodically reviews the standard preparation process, including standard bottles, logbooks and standard certificates and traceability to standardized sources.

10. Calibration and Standardization

10.1. Not Applicable

11. Procedure – The procedure includes a series of tasks performed sequentially. The tasks are listed in Table 1. A different analyst may be assigned to any task. Detailed procedures for each task are contained in specific "Task Instructions" (TI). All tasks are listed in Table 01. Details of the first five task instructions are included in this section. (TI 001 thru TI 007). "Task Instructions" (TI 008 thru TI 020) are detailed in Appendix 20.2.

Table 01 – Task for Extraction of Aqueous samples

	Preparation Step	Optional Tasks	Task Instruction
1	Prepare for extraction	Separatory Funnel	TI 001
		Liquid-Liquid	TI 002
2	Measure sample Volume		TI 003
3	Adjust sample pH		TI 004
4	Spike sample		TI 005
5.	Extract sample	Separatory Funnel	TI 006
		Liquid-Liquid	TI 007
6	Concentrate Extract	Kuderna-Danish	TI 008
		Turbo-vap	TI 009
		DryVap	TI 010
7	Extract Clean-up / Hydrolysis / Derivatization	Sulfuric Acid / Silica Gel for TPH-D	TI 011W
		Sulfuric acid for Pesticide or PCB	TI 012
		Sulfur removal	TI 013
		SPE – Silica gel	TI 014
		Alumina	TI 015
		EPH Fractionation	TI 016
		Hydrolysis / derivitization for TBT	TI 017
		Hydrolysis / derivitization for Herbicides & PCP	TI 018W
8	Vialing of final extract		TI 019

11.1. Preparing for extraction

11.1.1. Review the appropriate bench sheet Form listed in Appendix 20.1 to determine sample volume to be extracted, surrogate and spike additions, pH requirements, final effective volume, reporting limits requested and any other special requirements or instructions.

11.1.2. Obtain an Analyst Notes Form (3056F) and enter required data on the forms. Add header and sample identifications to the forms as appropriate. Example form is provided in Appendix 20.5.

11.1.3. Remove samples from Extractions Incoming Samples Refrigerator 5 and allow them to warm to room temperature.

11.1.4. Verify the client Identification's with the ARI labels. If the identifications do not match, inform the Organic Extractions Supervisor immediately.

11.1.5. Preparing Extraction by Separatory Funnel **(TI-001)**

11.1.5.1. Place a separatory funnel (SF) for each sample, matrix spike (MS), method blank (MB) and laboratory control sample (LCS) in a ring stand. The SF must have a capacity approximately two times the volume to be extracted.

11.1.5.2. Label each separatory funnel with the appropriate color label tape for the analysis containing the following information: ARI job number, sample ID letter, matrix ID, and type of extraction eq: (Pesticide or PCB). Method blank and LCS labels must also include the date of extraction. **See Table 2 below for label tape color by matrix and analysis.**

Table 02: Color coded Label Tape by Matrix and Analysis

Analysis	Matrix	Label Tape Color
Pest	Water/TCLP	Yellow
PCB	Water/TCLP	Yellow
SIM OP Pest	Water	Blue
PCP	Water	Purple
Herb	Water/TCLP	Pink
TBT	Water/Pore Water	Red
EPH	Water	Yellow (Aliphatic), White (Aromatic)
TPHD	Water	Grey
HCID	Water	Grey
AK102/103	Water	Grey
8270 PNA	Water	Blue-Green
SIM PNA	Water	Blue-Green
BAN or 1,4 Dioxane	Water/TCLP	Blue

11.1.5.3. Use a clean graduated cylinder to measure the specified volume of organic free water to the MB and LCS separatory funnels. Use the TCLP extraction fluid prepared by the metals laboratory for TCLP MB and LCS. Use Sea Water for TBT Pore Water for MB and LCS. (Pre-wash all TBT blanks with 30mL Methylene Chloride and discard the Methylene Chloride. **Note: All glassware associated with TBT must be pre-rinsed with 0.02% Tropolone in Methylene Chloride prior to use.**

11.1.5.4. Proceed to section 11.2 Measuring sample volumes **(TI 003).**

11.1.6. Preparing Extraction by Liquid/Liquid **(TI-002)**

11.1.6.1. Prepare one liquid/liquid extractor for each sample, matrix spike (MS), method blank (MB), and laboratory control sample (LCS).

11.1.6.2. Place a methylene chloride-rinsed 1000 mL round bottom (RB) flask into a heating mantle.

11.1.6.3. Add 2–3 boiling chips to each 1000 mL RB flask.

11.1.6.4. Place and seal a methylene chloride-rinsed extractor body 500mL or (1000mL with solvent saver insert), onto each RB flask and secure it with a green clip ring.

11.1.6.5. Use a clean 500 mL graduated cylinder to measure 200 mL of methylene chloride. Pour the methylene chloride into each extractor body.

11.1.6.6. Label each RB flask with the appropriate color label tape for the analysis containing the following information: ARI job number, sample ID letter, matrix ID, type of extraction (BAN, SIM PNA, etc.), and the date on the method blank (MB) and the LCS. **See Table 2 above for label tape color by matrix and analysis.**

11.1.6.7. Add (1000mL for 1000mL extractor body) or (500mL for 500mL extractor body) of deionized water from ARI's centralized water purification system to each extractor body designated for a MB or LCS. (Some of the solvent will siphon into the RB flask).

11.1.6.8. Turn on the cooling water for the condensers for each station being used.

11.2. Measuring sample volumes **(TI-003)**

11.2.1. <u>Full (to the neck) 0.5 or 1 L bottles</u> are assumed to contain 500 or 1000mL <u>respectively</u>. Pour the full bottle contents into the labeled separatory funnel or extractor body.

11.2.2. Record the volume on the bench sheet.

11.2.3. Note: sample description of each sample on Analyst Notes form 3056F. (No anomalies, turbid, color of sample or particulates).

11.2.4. *Set the empty sample bottle aside for later bottle rinsing (Section 11.5.1.2 thru 11.5.1.2.2 for Separatory Funnel) or (Section 11.5.2.1 thru 11.5.2.2 for Liquid/Liquid).*

11.2.4.1. For liquid/liquid extractions: The size of extractor bodies used are "to contain" 500mL or 1000mL sample and deionized water combined volumes to cycle properly with the 200mL methylene chloride.

11.2.4.2. If using 500mL of sample volume: add 500mL deionized water for the 1000mL extractor bodies.

11.2.5. When extracting contents of a partially filled sample container (500 or 1000 mL bottles).

11.2.5.1. Etch a line on the sample bottle at the sample meniscus.

11.2.5.2. Pour the entire sample into the separatory funnel or extractor body.

11.2.5.3. Note: sample description of each sample on Analyst Notes form 3056F. (No anomalies, turbid, color of sample or particulates).

11.2.5.4. *Perform bottle rinsing procedure in (Section 11.5.1.2 thru 11.5.1.2.2 for Separatory Funnel) or (Section 11.5.2.1-11.5.2.2 for Liquid/Liquid). Before determining sample volume.*

11.2.6. Determine each sample volume.

11.2.6.1. Fill each empty sample container to the previously etched line with tap water.

11.2.6.2. Pour the tap water into a graduated cylinder to measure the actual sample volume.

11.2.6.3. Record the actual volume on the bench sheet as the sample volume extracted.

11.2.6.4. For liquid/liquid extractions: The type of extractor bodies used are to contain 1000mL or 500mL combined sample and deionized water volumes to cycle properly with the 200mL methylene chloride.

11.2.6.5. Example: If using 300mL of sample volume: add 700mL deionized water for the 1000mL extractor bodies or 200mL deionized water for the 500mL extractor bodies.

11.2.7. When extracting less than the entire contents of a sample container.

11.2.7.1. Measure each sample using a clean glass graduated cylinder.

11.2.7.2. When there are insufficient pre-cleaned cylinders to measure an entire batch of samples, clean the cylinders thoroughly between measurements as follows:

11.2.7.3. Clean the cylinders using, in sequence, a detergent wash, a dilute acid rinse, an organic free water rinse, an acetone rinse and a final methylene chloride rinse. The procedure is described in ARI SOP 301S.

11.2.7.4. Record the "actual" volume of sample used on the bench sheet.

11.2.7.5. For liquid/liquid extractions: The type of extractor bodies used are to contain 1000mL or 500mL combined sample and deionized water volumes to cycle properly with the 200mL methylene chloride.

11.2.7.6. Example: If using 300mL of sample volume: add 700mL deionized water for the 1000mL extractor bodies or 200mL deionized water for the 500mL extractor bodies.

11.2.7.7. *No bottle rinse will be preformed when extracting less than entire contents of the sample container. (Note on Analyst Notes). Remaining sample will be archived.*

11.3. Adjust sample pH **(TI-004)**: See appropriate bench sheet listed in Appendix 20.1 and Table 3 for pH requirements and adjusting pH for specific analysis.

11.3.1. Measuring the pH of each sample.

11.3.1.1. Dip a clean glass stir rod or Pasteur Pipette in a sample and touch the adsorbed sample to a piece of narrow range pH paper. If using a glass stir rod for Liquid/Liquid: Rinse the stir rod with DI water, acetone and methylene chloride between samples.

11.3.1.2. When the pH must be in the range 5 < pH < 9 for Separatory funnel extraction.

11.3.1.2.1. When the pH is < 5 add sufficient 10 N sodium hydroxide solution drop-wise to increase the pH to 5 < pH < 9.

11.3.1.2.2. Stir the sample to disperse the10 N sodium hydroxide solution.

11.3.1.2.3. Allow the samples to sit for 10 minutes. Stir the samples again and verify the pH once more.

11.3.1.2.4. When the pH is > 9 add sufficient 1:1 Sulfuric Acid drop-wise to decrease the pH to 5 < pH < 9.

11.3.1.2.5. Stir the sample to disperse the Sulfuric Acid solution.

11.3.1.2.6. Allow the samples to sit for 10 minutes. Stir the samples again and verify the pH once more.

11.3.1.3. When the pH must be in the range <2.

11.3.1.3.1. When the pH is >2 use a Pasteur pipette to add sufficient 1:1 Sulfuric Acid or 1:1 HCL solution to the sample. See Table 2 for correct type of acid solution to be used. (usually ¼ to one full pipette for samples) (Use only ¼ pipette for the MB and LCS).

11.3.1.3.2. Stir the sample to disperse the Sulfuric Acid solution and decrease the pH to < 2.

11.3.1.3.3. Allow the samples to sit for 10 minutes. Stir the samples again and verify the pH is < 2 once more.

11.3.1.3.4. When more than one portion of Sulfuric Acid is required to reduce the pH to < 2, record the approximate volume of acid used on the Analysts Notes Form (3056F).

11.3.1.4. When the pH of a sample is adjusted, document the original pH and the amount of acid or base added to the sample on the Analyst Notes Form (Form 3056F).

Table 03: pH requirements per analysis		
Analysis	**Solutions**	**Ph Requirement**
Pest	1:1 Sulfuric Acid 10 N sodium hydroxide	5 < pH < 9
PCB	1:1 Sulfuric Acid 10 N sodium hydroxide	5 < pH < 9
SIM OP Pest	1:1 Sulfuric Acid 10 N sodium hydroxide	5 < pH < 9
PCP	1:1 Sulfuric Acid	pH<2
Herbicides	10 N sodium hydroxide 10 N sodium hydroxide 1:1 Sulfuric Acid	Initial extraction=pH 12> Clean-up=pH 12> Back extraction=pH<2
TBT	1:1 HCL	pH<2
EPH	1:1 Sulfuric Acid	pH<2
TPH	1:1 Sulfuric Acid	pH<2
HCID	1:1 Sulfuric Acid	pH<2
AK102/103	1:1 HCL	pH<2
8270 PNA	1:1 Sulfuric Acid	5 < pH < 9
SIM PNA	1:1 Sulfuric Acid 10 N sodium hydroxide	5 < pH < 9
BAN	1:1 Sulfuric Acid 10 N sodium hydroxide	SeparatoryFunnel Initial extraction=pH<2 (3xDCM) pH 12> (3XDCM) Liquid/Liquid=pH<2

11.4. Spiking Samples **(TI-005)**

11.4.1. Add the appropriate volume of surrogate solution specified on the bench sheet to all samples including MB, LCS, LSD, MS, and MSD. To verify that the surrogate spiking is accurate, the surrogate addition will be witnessed and documented by another laboratory technician. If the sample is an LCS, LCS Duplicate, or MS, MSD, proceed to sections 11.4.2.

11.4.2. Add the appropriate volume of spike solution specified on the bench sheet to the LCS, LCS Duplicate and any MS, MSD sample. To verify that the matrix spiking is accurate, the spike addition will be witnessed and documented by another laboratory technician.

11.4.3. The samples are now ready for extraction.

11.5. Extract Samples

11.5.1. Extracting samples by Separatory Funnel **(TI-006)**

11.5.2. **Note: Separatory Funnel extraction, hydrolysis, clean-up, and back extraction procedure for Herbicides is detailed in Appendix 20.2.18 Task Instructions (TI-018W).**

11.5.2.1. Extract each sample two to three times with the required solvent specified on the bench sheet. Use 30 mL Methylene Chloride for 100 and 500 mL samples and 60 mL Methylene Chloride for 1000 mL samples. Use 60 mL of the appropriate Hexane for low level extractions (1000 to 0.5 mL or 1500 to 0.25 mL). Final extracts have nominal volumes of either a 90 or 180 mL.

11.5.2.2. *Rinse each empty sample container with the initial portion of extracting solvent and pour the rinsate into its corresponding separatory funnel.*

11.5.2.2.1. *No bottle rinse will be preformed when extracting less than entire contents of the sample container or bottles containing residual sample or TCLP leachate. (Note on Analyst Notes form 3056F). Remaining sample will be archived.*

11.5.2.3. Mixing the sample and solvent will increase the vapor pressure in the separatory funnel. Prevent excessive pressure by gently inverting the funnel two or three times then with the stop cock end pointed up slowly open the stop cock to release pressure. Caution: Point the funnel into a designated hood away from yourself and co-workers, solvent may be ejected from the separatory funnel.

11.5.2.4. Seal and shake the separatory funnel vigorously for 2 minutes, periodically venting through the stopcock to release internal pressure.

11.5.2.5. Place the separatory funnel in a ring stand and allow organic and aqueous layers to separate for several minutes.

11.5.2.6. When the extracting solvent is Methylene Chloride (bottom), drain the entire Methylene Chloride organic layer (including any emulsion) into a labeled 250 mL Erlenmeyer flask.

11.5.2.7. When the extracting solvent is Hexane (top), drain the water into the original or clean sample container, then drain the entire Hexane organic layer (including any emulsion) into a labeled 250mL Erlenmeyer flask. Return the water from the sample bottle to the separatory funnel.

11.5.2.8. Repeat steps 11.5.2.3 through 11.5.2.7 two more times.

11.5.2.9. The 250 mL Erlenmeyer flask will contain a 90 or 180 mL extract plus any emulsion that may have formed.

11.5.2.10. When an emulsion is present, it must be broken up by whatever mechanical means necessary until all the inorganic material (water, silt, etc.) is removed from the extract.

11.5.2.11. More than 10% emulsion is centrifuged at 1000rpm for 5 minutes. Note centrifuge action on Analyst Notes form (3056F)

11.5.2.12. Dry the extract by adding anhydrous sodium sulfate to the flask. Use sufficient sodium sulfate so it is "free flowing".

11.5.2.13. Transfer the label tape from the separatory funnel to the corresponding 250 mL Erlenmeyer flask.

11.5.2.14. Submit the sample extract for concentration as specified on the bench sheet. Proceed to section 11.6.

11.5.3. Extracting samples by Continuous Liquid/Liquid **(TI-007)**

11.5.2.1 Rinse each empty sample container with 30 mL of Methylene Chloride and pour the rinsate into its corresponding extractor body.

11.5.2.2 No bottle rinse will be preformed when extracting less than entire contents of the sample container or bottles containing residual sample or TCLP leachate. (Note on Analyst Notes form 3056F). Remaining sample will be archived.

11.5.2.3 Rinse each neck of the L/L body with methylene chloride and attach and seal a condenser to each L/L body.

11.5.2.4 Turn on the heating mantle: make sure the control knob for each mantle is set at the calibrated mark.

11.5.2.5 Record the start time on the bench sheet

11.5.2.6 Allow samples to extract for a minimum of 8 hours (usually overnight)

11.5.2.7 After the extraction is completed: turn off the heating mantles while leaving on the cold water condensers, and allow the samples to cool for one-half hour

11.5.2.8 Record the end time and date on the bench sheet.

11.5.2.9 Turn off the cold water to the condensers. Remove condensers from the extractor body.

11.5.2.10 Carefully remove each extractor body from the 1000mL (RB) flask so that sample is not siphoned into the RB flask.

11.5.2.11 Pour the remaining water (extracted sample) into the 10 gallon waste pot.

11.5.2.12 Remove the flask from the mantle.

11.5.2.13 If necessary: Dry the extract by adding anhydrous sodium sulfate to the RB flask. Use sufficient sodium sulfate so it is "free flowing".

11.5.2.14 Record the analyst name, date and time for disassemble on the bench sheet.

11.5.2.15 Submit the sample extract for concentration task as specified on the bench sheet.

11.6. Concentrate sample as specified on the bench sheet. Options include

11.6.1. **Concentration and solvent exchange by Kuderna-Danish using TI-008**

11.6.2. **Concentration and solvent exchange by Turbo Vap using TI-009**

11.6.3. **Concentration and vialing extracts by DryVap using TI-010**

11.7. Concentrated extracts may require clean-up, hydrolysis, derivitization or separation as specified on the bench sheet. Extract treatment options/requirements are: (See benchsheet details).

11.7.1. **TPH-D Sulfuric Acid/Silica Gel Clean-up using TI-011W**

11.7.2. **Pest or PCB Sulfuric Acid Clean-up using TI-012**

11.7.3. **Pest or PCB Sulfur removal using TI-013**

11.7.4. **Silica Gel (SPE) Clean-up for Pest, PCB, PNA, and TPH-D using TI-014**

11.7.5. **Alumina Clean-up for TBT using TI-015**

11.7.6. **EPH Fractionation using TI-016**

11.7.7. **Derivitization and Hydrolysis for TBT using TI-017**

11.7.8. **Hydrolysis and Derivitization procedures for Herbicide and PCP using TI-018W**

12. Data Analysis and Calculations

12.1. Not Applicable

13. Method Performance

13.1. QA maintains control charts for the recovery of surrogate standards and spiked compounds.

13.2. Management periodically reviews the charts to detect and correct any negative trends in analyte recovery.

14. Pollution Prevention

14.1. Do not discard solvent contaminated solid material into trash containers. Place the solids in the designated 5-gallon "*satellite accumulation stations*" located at various places in the laboratory. This includes spent sodium sulfate, glass wool, solid sample, silica gel and paper wipes. Transfer the solids to a 55-gallon drum in the Hazardous Waste Room when the accumulation pails are full.

14.2. Pour any residual wastewater into the container in the Water Extraction Lab fume hood.

14.3. Do not discard organic solvent into the sink. Pour all waste solvent into the labeled 55-gallon drum located in the Hazardous Waste Storage Area.

14.4. Disposed expired standards into the designated barrel in the hazardous waste room.

14.5. Samples that are designated as hazardous waste by the LIMS "Hazardous Report" must be placed in the designated drum in the Hazardous Waste Storage Area when they are disposed. This process is described in SOP 1003S.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Not Applicable

16. Corrective Actions for Out of Control Events

16.1. Promptly report any events that may compromise the extraction process to the Organic Extractions Supervisor who will take appropriate steps to insure data quality. Corrective actions may include, but are not limited to, notation on the Analyst Notes Form (3056F) or re-extraction of the sample.

16.2. Corrective action procedures for common laboratory issues are provided in the diagrams provided in Section 20.14.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Unacceptable QA data noted during GC or GCMS analysis may result in a request for re-extraction using a 'Request for Re-extraction/Re-analysis Form (Form 0030F). Re-extract parameters (sample volume, final volume etc.) may be modified from the original extraction based on analytical results.

18. Waste Management

18.1. Place the solids in the designated 5-gallon "satellite accumulation stations" located at various places in the laboratory. This includes spent sodium sulfate, glass wool, solid sample, silica gel and paper wipes. Transfer the solids to the 55-gallon drum labeled "Solvent Contaminated Solids" located in the Hazardous Waste Room when the accumulation pails are full.

18.2. Discard all waste solvent (Hexane and Methylene Chloride) into the 55 gallon drum labeled "Chlorinated Solvents" located in the Hazardous Waste Storage Area.

18.3. Disposed expired standards into the designated barrel in the hazardous waste room.

18.4. Place samples that designate as hazardous using the LIMS "Hazardous Report" in the designated drum in the Hazardous Waste Storage Area when they are disposed. SOP 1003S describes the process for disposal of samples. Excess extracts and expired spiking solutions must be disposed of in the container labeled "Chlorinated Solvents" located in the Hazardous Waste Storage Area.

18.5. ARI's Laboratory Chemical Hygiene Plan (CHP) describes internal hazardous waste handling procedures. All analysts must be familiar with these requirements.

18.6. ARI properly profiles and disposes all hazardous waste using an EPA registered TSD (Treatment, Storage and Disposal) facility.

19. Method References

19.1. SW-846, "Separatory Funnel Extraction", Method 3510-C, Revision 3, December, 1996. USEPA Test Methods for Evaluating Solid Waste, (SW-846, 3rd Edition)

19.2. SW-846, "Continuous Liquid-Liquid Extraction", Method 3520-C, Revision 3, December, 1996. USEPA Test Methods for Evaluating Solid Waste, (SW-846, 3rd Edition)

19.3. Washington State Department of Ecology, "Extractable Petroleum Hydrocarbons", June 1997.

19.4. Washington State Department of Ecology, "Analytical Methods for Petroleum Hydrocarbons", June 1997.

19.5. Alaska Laboratory Method for the Analysis of Diesel Range Organics (DRO), AK102, November 7, 2002.

19.6. Alaska Laboratory Method for the Analysis of Residual Range Organics (RRO), AK103, November 7, 2002.

19.7. Krone (TBT), 1988.

19.8. SW-846, Alumina Clean-up, Method 3610B, Revision 3, December, 1996

19.9. SW-846, Silica Gel Clean-up, Method 3630C, Revision 3, December, 1996

19.10. SW-846, Sulfur Clean-up, Method 3660B, Revision 3, December, 1996

19.11. SW-846, Sulfuric Acid Clean-up, Method 3665A, Revision 3, December, 1996

19.12. SW-846, Acid-Base Partition Clean-up, Method 3650, Revision 1, July, 1992

20. Appendices

20.1. Appendix 20.1 - Extractions Performed Using SOP 3311S

20.2. Appendix 20.2 – Task Instructions (TI)

- 20.2.1. TI-001 - Preparation for aqueous sample extraction using a separatory funnel
- 20.2.2. TI-002 - Preparation for aqueous sample extraction using liquid-liquid extraction
- 20.2.3. TI-003 - Measure aqueous sample volume
- 20.2.4. TI-004 - Adjust pH of aqueous samples
- 20.2.5. TI-005 - Spike samples
- 20.2.6. TI-006 - Sample extraction using separatory funnel
- 20.2.7. TI-007 - Sample extraction using liquid–liquid extraction
- 20.2.8. TI-008 - Concentration and Solvent Exchange by Kuderna-Danish
- 20.2.9. TI-009 - Concentration and Solvent Exchange by Turbo Vap.
- 20.2.10. TI-010 - Concentration and vialing procedures by Dry Vap.
- 20.2.11. TI-011W - TPH-D Sulfuric Acid/Silica Gel Clean-up
- 20.2.12. TI-012 - Pest or PCB Sulfuric Acid Clean-up
- 20.2.13. TI-013 - Pest or PCB Sulfur removal
- 20.2.14. TI-014 - Silica Gel by (SPE) Clean-up for Pest, PCB, PNA, and TPH-D.
- 20.2.15. TI-015 - Alumina Clean-up for TBT.
- 20.2.16. TI-016 - EPH Fractionation.

20.2.17. TI-017 - Derivitization and Hydrolysis procedures for TBT.

20.2.18. TI-018W – Extraction, Hydrolysis, Clean-up, Back Extraction and Derivitization procedures for Herb and PCP.

20.2.19. TI-019- Vialing of final extract.

20.2.20. TI-020 - Preparation of Reagents and Solutions by parameter and matrix.

20.3. Appendix 20.3 – Corrective Action Flow Charts

20.4. Appendix 20.4 – Bench Sheets

20.4.1. SIM-PNA-Water – Low Level (0.01 ppb) Liq-Liq

20.4.2. SIM-PNA-Water – In-house (0.1 ppb) Liq-Liq

20.4.3. SIM-PNA-Water – Liq-Liq (1.0 ppb) Liq-Liq

20.4.4. 1,4-Dioxane – Water - Liq-Liq

20.4.5. 8270 BAN – Water Liq-Liq

20.4.6. SIM-PNA-Water – Low Level (0.01 ppb) SepFunnel

20.4.7. SIM-PNA-Water – In-house (0.1 ppb) SepFunnel

20.4.8. SIM-PNA-Water – Liq-Liq (1.0 ppb) SepFunnel

20.4.9. 8270 BAN – TCLP Extract SepFunnel

20.4.10. 8270 BAN – Water – SepFunnel

20.4.11. NWTPHD/HCID by SepFunnel

20.4.12. EPH by SepFunnel

20.4.13. TBT – Water – SepFunnel

20.4.14. TBT – Pore Water – SepFunnel

20.4.15. Herbicide – TCLPExtract – SepFunnel

20.4.16. Herbicide – Water-Low Level – SepFunnel

20.4.17. Herbicide – Water – SepFunnel

20.4.18. 8041 PCP – Water-Low Level– SepFunnel

20.4.19. 8041 PCP – Water – SepFunnel

20.4.20. SIM OP Pesticide by SepFunnel

20.4.21. PCB – TCLP Extract – SepFunnel

20.4.22. PCB – Water – Low Level (0.01ppb) – SepFunnel

20.4.23. PCB – Water – MTCA (0.1ppb) – SepFunnel

20.4.24. PCB – Water – (1.0 ppb) – SepFunnel

20.4.25. Pesticide – TCLP Extract – SepFunnel

20.4.26. Pesticide – Water MTCA – SepFunnel

20.4.27. Pesticide – Water – SepFunnel

20.4.28. SIM – PNA/Phenol – Water – SepFunnel

20.5. Forms

Appendix 20.1 - Extractions Performed using SOP 3311S

Procedure Code	Description of Analysis	Bench Sheet	Extraction Technique	Sample volume (mL)	FEV (mL)	Extraction pH	Solvent	Concentration Technique/TI#	Cleanup/Separation/ Derivatization/Hydrolysis/ TI#
PESWSI	Pesticides – In-house (0.05-0.1ppb)	3038F	SF	500	5.0	5 < pH < 9	Initial=DCM Exchange=Hexane	K-D/Turbo (TI-008)/(TI-009)	Sulfur/Silica Gel (TI-013)/(TI-014)
PESWSM	Pesticide – MTCA (0.005-0.01ppb)	3041F	SF	1000	1.0	5 < pH < 9	Initial=DCM Exchange=Hexane	K-D/Turbo (TI-008)/(TI-009)	Sulfur/Silica Gel (TI-013)/(TI-014)
PESWST	Pesticide – TCLP (0.5-1.0ppb)	3039F	SF	100	10.0	5 < pH < 9	Initial=DCM Exchange=Hexane	K-D/Turbo (TI-008)/(TI-009)	Silica Gel (TI-014)
PCBWSI	PCB – In-house (1.0ppb)	3018F	SF	500	5.0	5 < pH < 9	Initial=DCM Exchange=Hexane	K-D/Turbo (TI-008)/(TI-009)	Acid/Sulfur/Silica Gel (TI-012)/(TI-013)/(TI-014)
PCBWSM	PCB – MTCA (0.1ppb)	3021F	SF	500	1.0	5 < pH < 9	Initial=DCM Exchange=Hexane	K-D/Turbo (TI-008)/(TI-009)	Acid/Sulfur/Silica Gel (TI-012)/(TI-013)/(TI-014)
PCBWSL	PCB – Low level (0.01ppb)	3022F	SF	1000	0.5	5 < pH < 9	Initial=Hexane	K-D/Turbo (TI-008)/(TI-009)	Acid/Sulfur/Silica Gel (TI-012)/(TI-013)/(TI-014)
PCBWST	PCB – TCLP (10ppb)	3023F	SF	100	10.0	5 < pH < 9	Initial=DCM Exchange=Hexane	K-D/Turbo (TI-008)/(TI-009)	Acid/Silica Gel (TI-012)/(TI-014)
OPPWSI	o,p-Pest – In house (0.2-1.0ppb)	3082F	SF	500	1.0	5 < pH < 9	DCM	KD/DryVap/Turbo (TI-008) /(TI-010) /(TI-009)	None
PCPWSI	Chlorophenols – In-house (0.25ppb)	3016F	SF	500	50.0	pH < 2	Initial=DCM Exchange=Hexane	K-D/Turbo (TI-008)/(TI-009)	Derivitized by GC
PCPWSL	Chlorophenols – Low Level (0.05ppb)	3050F	SF	500	10.0	pH < 2	Initial=DCM Exchange=Hexane	K-D/Turbo (TI-008)/(TI-009)	Derivitized by GC
HERWSI	Herbicides – In-house (0.25-250ppb)	3012F	SF	500	50.0	Initial pH > 12 CleanuppH>12 Back Extract pH < 2	Cleanup=DCM Back Extract= 1XDCM/Ether 2X DCM/Hexane	K-D/Turbo (TI-008)/(TI-009)	Hydrolysis (TI-018W) Derivitized by GC
HERWSL	Herbicides – Low Level (0.05-50ppb)	3049F	SF	500	10.0	Initial pH > 12 Cleanup pH 12 Back Extract pH < 2	Cleanup=DCM Back Extract= 1XDCM/Ether 2X DCM/Hexane	K-D/Turbo (TI-008)/(TI-009)	Hydrolysis (TI-018W) Derivitized by GC

Appendix 20.1 - Extractions Performed using SOP 3311S

Procedure Code	Description of Analysis	Bench Sheet	Extraction Technique	Sample volume (mL)	FEV (mL)	Extraction pH	Solvent	Concentration Technique/TI#	Cleanup/Separation/ Derivatization/Hydrolysis/ TI#
HERWST	Herbicides – TCLP(1.25-1250ppb)	3048F	SF	100	50.0	Initial pH > 12 Cleanup pH >12 Back Extract pH < 2	Cleanup=DCM Back Extract= 1XDCM/Ether 2X DCM/Hexane	K-D/Turbo (TI-008)/(TI-009)	Hydrolysis (TI-018W) Derivitized by GC
TBTWSI	TBT – In-house (0.2-0.3ppb)	3043F	SF	100	0.5	pH < 2	Initial=Tropolone/DCM Exchange=Hexane	K-D/Turbo (TI-008)/(TI-009)	Derivatize (TI-017) Alumina (TI-015)
TBTPSI	TBT – Pore Water (0.0075-0.01ppb)	3047F	SF	100	0.5	pH < 2	Initial=Tropolone/DCM Exchange=Hexane	K-D/Turbo (TI-008)/(TI-009)	Derivatize (TI-017) Alumina (TI-015)
EPHWSI	EPH – In-house (40ppb)	3070F	SF	500	1.0	pH < 2	Initial=DCM Exchange=Pentane/Hexane	K-D/Turbo (TI-008)/(TI-009)	Fractionation (TI-016)
DIEWSI	TPHD-HCID – In-house (0.25-0.5ppm)	3014F	SF	500	1.0	pH < 2	DCM	KD/DryVap/Turbo (TI-008) /(TI-010) /(TI-009)	Acid/Silica Gel (TI-011W)
AK2WSI	AK102-AK103 In-house (0.25-0.5ppm)	3003F	SF	500	1.0	pH < 2	DCM	DryVap/Turbo (TI-010)/(TI-009)	Silica Gel (TI-014)
BANWSI	SVOA – In-house (1.0-5.0ppb)	3010F	SF	500	0.5	1st Extraction pH < 2 2nd Extraction pH > 12	DCM	K-D/Turbo (TI-008)/(TI-009)	None
BANWST	SVOA – TCLP (10.0-50.0ppb)	3035F	SF	100	1.0	1st Extraction pH < 2 2nd Extraction pH > 12	DCM	K-D/Turbo (TI-008)/(TI-009)	None
PNAWSI	PNA – In-house (1.0ppb)	3008F	SF	500	0.5	5 < pH < 9	Initial=DCM Exchange=Hexane	K-D/Turbo (TI-008)/(TI-009)	Silica Gel (TI-014)
PNSWSL	PNA-SIM-In House (0.1ppb)	3053F	SF	500	0.5	5 < pH < 9	Initial=DCM Exchange=Hexane	K-D/Turbo (TI-008)/(TI-009)	Silica Gel (TI-014)
PNLWSL	PNA-SIM Low Level (0.01ppb)	3071F	SF	500	0.5	5 < pH < 9	Initial=DCM Exchange=Hexane	K-D/Turbo (TI-008)/(TI-009)	Silica Gel (TI-014)

Appendix 20.1 - Extractions Performed using SOP 3311S

Procedure Code	Description of Analysis	Bench Sheet	Extraction Technique	Sample volume (mL)	FEV (mL)	Extraction pH	Solvent	Concentration Technique/TI#	Cleanup/Separation/ Derivatization/Hydrolysis/ TI#
BANWLI	SVOA – In-house (1.0-10.0ppb)	3006F	LL	500	0.5	pH < 2	DCM	KD/DryVap/Turbo (TI-008) /(TI-010) /(TI-009)	None
DIOWLI	SVOA-Dioxane In House (2.0ppb)	3033F	LL	500	1.0	pH < 2	DCM	K-D/Turbo (TI-008)/(TI-009)	None
PNAWLI	PNA – In-house (1.0ppb)	3052F	LL	500	0.5	5 < pH < 9	Initial=DCM Exchange=Hexane	K-D/Turbo (TI-008)/(TI-009)	Silica Gel (TI-014)
PNSWLL	PNA-SIM In House (0.1ppb)	3054F	LL	500	0.5	5 < pH < 9	Initial=DCM/Hexane Exchange=Hexane	K-D/Turbo (TI-008)/(TI-009)	Silica Gel (TI-014)
PNLWLL	PNA-SIM Low Level (0.01ppb)	3055F	LL	500	0.5	5 < pH < 9	Initial=DCM/Hexane Exchange=Hexane	K-D/Turbo (TI-008)/(TI-009)	Silica Gel (TI-014)

Appendix 20.2

Task Instructions

Task Instructions (TI-008)

Concentrating and Solvent Exchange procedure using Kuderna-Danish.

Refer to bench sheet for requirements and instructions.

1. Concentrating and solvent exchanging extracts by Kuderna-Danish (KD)

1.1. Rinse a K-D apparatus using methylene chloride or hexane. Add 2-3 clean boiling chips. Note – Rinse glassware with the appropriate Methylene Chloride or Hexane for the low level extractions (PCB or Pesticides). See bench sheet.

1.2. Verify that the sodium sulfate in the sample extract is "free flowing". If it is not, add anhydrous sodium sulfate to adsorb any residual water in the extract. Swirl the flask to assure complete removal of any moisture present.

1.3. For aqueous extract: Pour the extract directly from the flask into the prepared K-D apparatus. Proceed to section 1.4.1.

1.4. For soil/sediment extract: Prepare a drying column by inserting a glasswool plug into the bottom. (See bench sheet for pH of glasswool required). Fill the column with approximately 10cm of anhydrous sodium sulfate. Place the column on top of the KD secured with a clamp.

1.4.1. Rinse the flask three times with the appropriate solvent and add the rinsate to the KD flask as well.

1.4.2. Transfer the colored sample ID labels from the flask to the K-D apparatus.

1.4.3. Attach a rinsed three-ball Snyder column to the KD flask, and place the entire apparatus on the proper temperature water bath using Table 4 below.

1.4.4. Wet the inside of the Snyder column with 1 to 2 mL of the extracting solvent before boiling starts.

1.4.5. **Note: If a solvent exchange is not necessary, proceed to step 1.4.7**

1.4.6. **To exchange the solvent to Hexane:** When the extract reaches ~5mL, add 20 mL Hexane to the K-D through the Snyder Column. Mix well and immediately transfer the K-D to another water bath set at 100°C. Place an "/" on the label to indicate the first Hexane exchange process has taken place. When the extract again reaches ~5mL, add an additional 20mL Hexane to the KD through the Snyder Column. Make an **"X"** on the label to indicate the second Hexane exchange process has taken place.

1.4.7. When the extract reaches ~5mL, remove the K-D from the water bath and allow it to cool for about 10 minutes on a cooling rack. After cooling the extracts should be approximately 5-10 mL.

1.4.8. Remove the Snyder column from the K-D. Pour the extracts from the K-D into the corresponding labeled Turbo Tube and rinse the K-D apparatus with approximately 5 mL of the appropriate solvent and add the rinsate to the Turbo Tube.

1.4.9. Transfer the colored sample identification labels from the K-D Apparatus to the Turbo Tube.

1.4.10. Extracts are now ready for further concentration by turbo vap, clean-up, hydrolysis, derivitization, or separation as specified on the bench sheet.

1.4.11. **EPH concentration:** The extracts should be placed on the 80°C water bath. **When the extract has concentrated to no higher than 1mL**, exchange by adding 30mL of 90:10 Pentane/Hexane through the Snyder column. Mix well and immediately transfer the KD to another water bath set at 100° C. Mark the label with an **"X"** to indicate the exchange process has taken place.

1.4.12. **EPH exchange: Once the extract has concentrated to no higher than 0.5mL, remove it from the water bath.** (Removing the extract before it has reached 0.5mL could result in an improper exchange and affect the fractionation process).

1.4.13. When the extract reaches ~0.5mL, remove the K-D from the water bath and allow it to cool for about 10 minutes on a cooling rack. After cooling the extracts should be approximately 5mL.

1.4.14. Remove the Snyder column from the K-D. Pour the extracts from the K-D into the corresponding labeled Turbo Tube and rinse the K-D apparatus with approximately 5 mL of the appropriate solvent and add the rinsate to the Turbo Tube.

1.4.15. Transfer the colored sample identification labels from the K-D Apparatus to the Turbo Tube.

1.4.16. Extracts are now ready for further concentration by turbo vap and separation as specified on the bench sheet.

Table 04: Water Bath Temperatures

Water Bath No.	Temperature Range	Solvent Type
1	80-85° C	DCM only
2	100° C	Hexane only Hexane exchanges 90:10 Pentane/Hexane exchange 80:20 Hexane/Acetone
3	90-95° C	50:50 DCM/Acetone

Task Instructions (TI-009)

Concentrating and Solvent Exchange procedure using by Turbo Vap.

Refer to bench sheet for requirements and instructions.

1. Concentrating and solvent exchanging extracts by Turbo Vap

1.1. Concentrate the extracts to a volume slightly less than the volume to lab specified on the bench sheet.

1.2. Verify that the Turbo Tube was previously labeled at the top with a wide Sharpie pen.

1.3. Position the tray in the Turbo Vap set at 40 °C and check the water level which must be at a level even with the first horizontal tray inside the turbo tube rack.

1.4. Select the row(s) of Nitrogen nozzles to be used by pressing the row panel on the Turbo Vap.

1.5. Start with a lower Nitrogen pressure 10-20 psi *minimum to avoid splashing.* Insert the turbo tubes into the Turbo Vap tray quickly, one at a time opening and closing the lid between turbo tube insertions.

1.6. When all Turbo tubes are inserted into Turbo Vap, close the lid and as quickly as possible with out splashing and bring the pressure of the nitrogen up to maximum 40 psi by turning the pressure control clockwise. Nitrogen must be continuous. **Complete the concentration as quickly as possible with minimal opening and closing of the TurboVap lid to prevent potential losses of analytes.** When the concentration is complete, remove the extracts from the Turbo Vap immediately. Mix well.

1.7. If a hexane exchange is necessary: follow the bench sheet instructions for volumes.

1.8. Once the extract reaches the specified volume to lab or for further clean-up listed on the bench sheet, transfer it using the appropriate solvent and syringe into an appropriate vial. Clean the syringe between extracts by rinsing three times with the appropriate solvent.

Task Instructions (TI-010)

Concentrating and vialing procedure using by Dry Vap.

Refer to bench sheet for requirements and instructions.

1. Concentrating and vialing extracts by Dry Vap

1.1. Pre-Clean Dry Vap station using Methylene Chloride before use.

1.1.1. Power up the Dry Vap by turning on the vacuum.

1.1.1.1. The settings are as follows: The vacuum regulator should read 12 inches Hg. The vacuum pump guage should read 25 inches Hg. The Dry Volume knob is set at 0. Heater power knob is set at 1. Heat Timer is set at 0. Auto Rinse is off.

1.1.2. Make sure the rinse tube is in line and pointed towards the sparge tube to ensure proper rinse coverage of the station lid and EV tube.

1.1.3. For each station to be used: Place the EV tube into the cradle. When the EV tube is inserted, the *"Ready" LED illuminates*. With the station lid pushed down on the EV tube (make sure the EV tube is centered with the lid for best surface contact with the lid gasket for a vacuum seal. (Vacuum regulator should read between 20-25 for a good seal). Press the "Manual Rinse" button and wait a few seconds until the rinse has finished. Repeat this step until the Methylene Chloride rinse is being streamed to the underside of the lid and running down the sides of the EV tube. Rinse the EV tube 3 more times by pressing the "Manual Rinse". Lift the station lid and rinse the sparge tube and heater coil with methylene chloride. Remove the EV tube from cradle and discard the methylene chloride rinse into a waste beaker. Rinse the EV tube with methylene chloride.

1.1.4. Replace the EV tube back into cradle. Add 15mL methylene chloride to the EV tube and re-seal the station lid. Press the "Manual Rinse" button 4 X. Lift the station lid and rinse the sparge tube and heater coil with methylene chloride. Press the "Start" button to purge any solvent from the sparge tube. (5-10 seconds). Press the "Stop" button after the solvent has emptied from the sparge tube. Remove the EV tube from the cradle and discard methylene chloride rinse into a waster beaker. **(Note: Make sure to empty the liquid trap bottles located under the units when they are full).**

1.1.5. The Dry Vap station is now ready for use.

1.1.6. Using a clean 200mL EV tube with 0.4mL tip, fill the tube (to the max fill-line) with the extract. Transfer the colored label tape to the EV tube. Seal the station lid. (Check pressure for proper seal). Press the "Start" button. *The "Heat", "Vacuum", and "Sparge" LED's will illuminate during operation.*

1.1.7. When the extract is concentrated to 0.4mL, *the station "Finish" LED will Illuminate*. Allow the EV tube to cool to room temp (22-23°C) before lifting the station lid.

1.1.8. Lift the station lid and remove the EV tube. The extract is ready to vial.

1.1.9. Transfer the extract using a 1000µL syringe into a pre-labeled with sample ID 2.5mL vial with PTFE-lined cap using methylene chloride. Rinse the syringe three times with methylene chloride between extracts.

Task Instructions (TI-011W)

Sulfuric Acid/Silica Gel Clean-up procedure for TPH-D Aqueous Extracts

Refer to bench sheet for options or requirements.

1. **TPH-D Sulfuric Acid/Silica-Gel Clean-up – When requested by ARI's Client or PM: Acid/Silica gel clean-up may remove naturally occurring non-petroleum organics (e.g. tannins, lignin and other biogenic compounds), which may contribute to chromatographic interferences. Acid clean-up is not suitable for AK-102, AK-103, or HCID analysis.**

1.1. Aqueous extracts need to be vialed at exactly 1mL before clean-ups. Vial into a pre-labeled with sample ID, 2.5mL clear vial with PTFE-lined crimp cap using methylene chloride

1.2. Add ~100 µL of concentrated sulfuric acid to the 1.0mL extract using a pipet.

1.3. Mix the extract well using a Vortex Mixer.

1.4. Centrifuge the extract to separate the acid from the solvent. Uncap the extract and using a 1.0mL syringe, remove the solvent layer (top) and place into a labeled 2.5 mL clear auto sampler vial.

1.5. Add ~0.04 g silica gel.

1.6. Mix well using a Vortex Mixer. Centrifuge the extract.

1.7. Uncap the extract and using a 1.0mL syringe, remove the solvent layer (top) and place into a labeled clear vial.

1.8. If the extract still has color: Repeat 1.2-1.7 using the same amounts of acid and silica gel. Centrifuge the extract.

1.9. Attach a 0.2 µm filter (13mm) to a 1000µL luer lock syringe and rinse the filter with 1mL of methylene chloride. Dry the filter with 1mL volume of air.

1.10. Filter the extract to remove any remaining silica gel into a pre-labeled 2.5 mL clear auto ampler vial with a PTFE lined crimp cap for GC-FID analysis. (*Solvent loss will occur during clean-ups; this is normal and reporting limits are based on the 1mL FEV before clean-ups.*)

1.11. Rinse the syringe thoroughly three times with methylene chloride between each extract.

Task Instructions (TI-012)

Sulfuric Acid Clean-up procedure for Pest or PCB

Refer to bench sheet for options or requirements.

1. **Pest or PCB Sulfuric Acid Clean-up: This procedure details the sulfuric acid clean-up portion of solvent extracts analyzed for PCBs and selected Organochlorine Pesticides. Sulfuric Acid: A rigorous clean up process used in the removal of most organic chemicals prior to analysis of PCB's. Use caution when analyzing pesticide since some may be degraded using this procedure. It does not destroy chlorinated benzene or chlorinated naphthalene.**

1.1. Note: Refer to bench sheet for requirements. Required clean-up for (Low Level extraction) or optional and the extract has any color or the analyst notes (3056F) indicates there were emulsions.

1.2. **Note:** Extracts must be exchanged to hexane before initiating acid clean-up.

1.3. The extract subjected to clean-up must be at least 4 mL in volume. Preferably the extract will be adjusted to final volume prior to acid clean-up. Extracts requiring a final volume less than 4.0 mL will be "transfer rinsed" and re-concentrated after clean-up.

1.4. Transfer the entire extract to a labeled 20 mL scintillation vial.

1.5. Carefully add 1.5 to 5.0 mL (1 to 3 pipets) of concentrated sulfuric acid to the vial and cap.

1.6. Vortex the extract/sulfuric acid mixture for 60 seconds.

1.7. Centrifuge the vial at ≤ 1000 rpm for 5 minutes to expedite phase separation.

1.8. If the extract was at final volume prior to this procedure, and no other clean-up is required, transfer 1 mL of the hexane extract to a labeled auto-sampler vial for instrumental analysis. **Caution:** (*When working in the acid phase it is extremely important to avoid acid in the vial being sent to GC for analysis*).

1.9. When the acid treated extract requires additional concentration, transfer the entire hexane layer (top) to a turbo vap tube. Rinse the scintillation vial twice with 2 mL aliquots of hexane and combine the rinses with the extract in the turbovap tube. Concentrate the hexane extract to the specified final volume for instrumental analysis.

1.10. If additional clean-up (sulfur or silica-gel) is required, the hexane extract is transferred to the appropriate container for further treatment. See Appendix 20.2.13 Task Instructions (TI-013 Sulfur Removal) and/or 20.2.14 Task Instructions (TI-014 Silica Gel Clean-up) for details.

Task Instructions (TI-013)

Sulfur Removal for Pest or PCB
Refer to bench sheet for options or requirements.

1. **Pest or PCB Sulfur Clean-up:** Elemental sulfur is encountered in many sediment, marine algae, and industrial waste sample matrices. The solubility of sulfur in various solvents is similar to some of the organochlorine and organophosphorous pesticides. Therefore, sulfur remains present with the pesticides through the normal extraction and cleanup techniques and causes interference when samples are analyzed using an electron capture detector (ECD).

1.1. The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting pesticides and Aroclors. Sulfur contamination should be expected with sediment samples.

1.2. Sulfur will be quite evident in gas chromatograms obtained from electron capture detectors. If the gas chromatograph is operated at the normal conditions for organochlorine pesticide analysis, the sulfur interference can completely mask the region from the solvent peak through Aldrin.

1.3. The use of tetrabutylammonium sulfite (TBAS) for the elimination of sulfur is detailed in this method.

1.4. **Note: Extracts must be exchanged to hexane before initiating sulfur clean-up.**

1.5. **Note: An extract treated with TBAS must be at <u>least</u> 4 mL in volume.**

1.6. **PCB: Proceed to step 1.12.**

1.7. **Pesticides**: **Note:** Refer to bench sheet for requirements. Required clean-up for (**MTCA level** extraction) or optional and the extract has **any color** or the analyst notes (3056F) indicates there were emulsions.

1.8. Vial 0.5mL below the final effective volume listed on the bench sheet. Extracts requiring a final volume less than 4.0 mL will be re-concentrated after sulfur removal.

1.9. Transfer the entire extract to a 20 mL scintillation vial.

1.10. Add 0.5mL of ethyl acetate.

1.11. Proceed to step 1.13.

1.12. PCB'S **(Do not add ethyl acetate)**. Note: Refer to bench sheet for requirements. Required clean-up for (**MTCA level or Low Level** extraction) or optional and the extract has **any color** or the analyst notes (3056F) indicates there were emulsions.

1.13. Add 1 pipette of TBAS reagent to the 20mL scintillation vial.

1.14. Vial specified Final Effective Volume stated on the bench sheet into a 20mL scintillation vial.

1.15. Add enough crystalline sodium sulfite to cover the bottom of the 20mL scintillation vial.

1.16. Mix the vial for at least two minutes using a Vortex mixer (or put on the paint shaker for 15 minutes). Crystals of sodium sulfite should be present. If not, add additional 0.2 gram portions of sodium sulfite until crystals are present after repeated mixing.

1.17. After shaking: Add at least 10-15 mL organic-free water to the vial and vortex the solution for two minutes (or put back on paint shaker for 15 minutes).

1.18. After shaking: Allow the sample to stand until the layers have completely separated.

1.19. If there is an emulsion, centrifuge the vial at 1000 rpm for 5 minutes to expedite phase separation

1.20. This completes the sulfur removal process.

1.21. If the extract was at final volume prior to this procedure, and no other clean-up is required, transfer 1 mL of the hexane extract to a labeled auto-sampler vial for instrumental analysis.

1.22. If the extract requires additional concentration, transfer the hexane organic layer (top) to a turbo vap tube. Rinse the scintillation vial twice with 2 mL aliquots of hexane and combine the rinses with the extract in the turbo vap tube. Concentrate the hexane extract to final volume for instrumental analysis.

1.23. If additional clean-up (silica-gel) is required, the hexane extract is transferred to the appropriate container for further treatment. See Appendix 20.2.14 Task Instructions (TI-014 Silica Gel Clean-up) for details.

Task Instructions (TI-014)

Silica Gel by (SPE) clean-up procedure
Refer to bench sheet for options or requirements.

1. Silica-Gel Clean-up by (SPE) for Pest, PCB, PNA, and TPH-D extracts by (SPE)

1.1. Silica gel clean-up of aqueous samples is required when samples are analyzed for SIM-PNA, pesticides or PCB using techniques that provide low detection levels.

1.2. Silica gel clean-up of aqueous samples or TCLP extracts is optional when samples are analyzed for pesticides, PCB, PNA, or SIM-PNA using techniques that provide routine detection levels.

1.3. Silica gel cleanup is performed on TPH-D extracts only when it is requested by ARI's client.

1.4. Procedure: Each RapidTrace workstation to be used must be "primed" with solvents, followed by a verification of its operation daily as follows:

1.4.1. Verify the solvent reservoirs contain adequate levels of solvents before proceeding, if not fill with the appropriate solvent.

1.4.2. Load a SPE cartridge into slot 1 of the cartridge carousel and add a disposable culture tube to the collect and sample slots in position one of the sample rack.

1.4.3. Using the RapidTrace software select the appropriate "prime method" for sample slot 1 and allow the workstation to finish the procedure.

1.4.3.1. These procedures automatically add 2 ml of solvent to the sample tube, followed by 'sipping' the sample and adding it to the SPE cartridge. The eluant is collected into the collection tube and the SPE cartridge is washed with an additional 3 ml of solvent which is again collected into the collection tube.

1.4.3.2. Correct operation is verified if the sample tube contains no residual sample and the collection tube contains 5 ml of eluant.

1.4.4. After priming and verification: up to 10 samples in culture tubes are loaded into the right side of the sample rack with a labeled corresponding collection tube in the left side of the rack.

1.4.4.1. Analysis required volumes and solvents are as follows:

Analysis	Volume	Extract Solvent
Pest	2-3mL	Hexane
PCB	2-3mL	Hexane
PNA	2-3mL	Hexane
TPH-D	2-3mL	Methylene Chloride

1.4.5. Using the RapidTrace software select the RapidTrace module(s) and assign appropriate "run method" to the samples loaded and start the run on the loaded module(s).

1.4.6. Record the prime and extract runs in the SPE Run Log (3078F).

1.4.7. It is possible to initiate runs on multiple modules utilizing multiple methods with the RapidTrace control software. During an analytical run each module runs a single method for each set of samples. Do not mix methods in a single analytical run. Allow the run to finish, and then switch the module over to a different method.

1.4.8. The RapidTrace software will calculate precisely the time for the run to finish after it is initiated based on the "run" procedure selected and the number of samples in the tray. When the run is finished, remove the sample tray and visually inspect the sample tubes and collection tubes to verify proper operation during the run. The sample tubes should be empty and the collection tubes all should contain a consistent volume of eluant. Errors should be noted and the laboratory supervisor notified so as to implement corrective action.

1.4.9. The 16X100 mm collections tubes are transferred to the approriated turbo vap fitted for these tubes (or transferred to a labeled 60mL Turbo Tube) and the extracts are concentrated to the final effective volume specified on the bench sheet.

1.4.10. The extracts are transferred to the appropriate auto sampler vial and delivered to the instrument lab for analysis.

Task Instructions (TI-015)

Alumina Clean-up procedure
Refer to bench sheet for options or requirements.

1. Alumina Clean-up for TBT

1.1. **Note:** The extract must be previously derivitized and hydrolyzed. See Appendix 20.2.17 Task Instructions (TI-017 Derivitization and Hydrolysis procedures for TBT for details).

1.2. Pass the hexane extract through a medium size micro column packed with 5 g of activity 0 alumina. Use a pre-rinsed 60ml Turbo Tube to catch the elution. Rinse the extract Turbo Tube with two 0.5mL portions of hexane, and add to the column. Elute column with hexane and collect 3-4 mL. The collected fraction should be colorless. If the fraction is not colorless, concentrate again to 2.0mL and repeat alumina clean-up once more and note this on the analyst notes (3056F).

1.3. Concentrate extract to 0.5mL and vial into a 2.5mL amber vial with a PTFE-lined screw-cap for analysis by GC/MS-SIM. Transfer red label tape with the sample ID to the vial. Note: Final effective volume is 0.5mL.

Task Instructions (TI-016)

EPH Fractionation
Refer to bench sheet for options or requirements.

1. **EPH Fractionation:** This method details the procedure to fractionate the collective concentrations of extractable aliphatic and aromatic hydrocarbons. This procedure meets the requirements of WDOE 1997.

1.1. Fractionating EPH Extracts Procedure

1.1.1. Prepare a macro column by packing a small plug of neutral glass wool in the bottom of the column. Add approximately 0.5 cm of anhydrous sodium sulfate on top of the glass wool plug. Suspend the column in a clamp over a 150 mL beaker used to collect process waste.

1.1.2. Place a small funnel at the top of the column. Have four labeled turbo tubes ready for collection of the EPH fractions generated (two for aliphatic and two for aromatic collections of each extract).

1.1.3. Weigh 7.5g of 0% silica gel into a tared 50mL beaker. Add enough methylene chloride to slurry the silica gel.

1.1.4. Mix the silica gel slurry well and pour through a small funnel into the prepared macro column. Once all slurry has been added to the column gently tap the column to settle the silica gel.

1.1.5. Once the silica gel slurry has settled, add approximately 0.5cm of anhydrous sodium sulfate to the top of the silica gel, creating a 'sandwich' effect.

1.1.6. Rinse the column with one full column of methylene chloride and elute into a waste beaker.

1.1.7. Once all the methylene chloride has eluted to the waste beaker, immediately add 20mL of pentane to the column. This process exchanges the column and prepares it for the aliphatic collection. The pentane rinse is discarded.

1.1.8. Once the pentane exchange has completely eluted to waste. Remove waster beaker and place the first 60mL turbo tube under the column with an aliphatic label on it.

1.2. **Aliphatic Fraction:**

1.2.1. Using a Pasteur pipette, add 2mL of extract onto column. Let the 2mL fully elute thru the column (collect).

1.2.2. Rinse turbo tube with 2mL of pentane and add this to the column as well. Let rinsate fully elute thru the column (collect).

1.2.3. (See updated QC of how many mL of pentane to use located above the fractionation station) Add 27-33mL of pentane to the column and collect. Split the volume of pentane between two turbo tubes.

1.2.4. Once all pentane has eluted from the column, remove turbo tube and replace it with the first turbo tube that has an aromatic label.

1.3. **Aromatic Fraction:**

1.3.1. Once turbo tube is under column, add 35mL of 60:40 Pentane/DCM to the column. Let the first 2mL get thru the column before adding the rest of the solvent.

1.3.2. Once solvent has fully eluted thru column, remove aromatic turbo tube. Split the volume of solvent between two turbo tubes.

1.3.3. Discard silica gel from the columns into the buckets marked for halogenated waste.

1.3.4. Extracts are now ready for final concentration by Turbo Vap.

1.4. **Final Concentration by Turbo Vap and Vialing**

1.4.1. Refer to Turbo vap procedure in section Appendix 20.2. Task Instructions (TI-009).

1.4.2. Concentrate aliphatic and aromatic fractions separately.

1.4.3. Caution: Constant attention must be given to the turbo vap at all times, pentane concentrates very quickly under nitrogen and leaving extracts unattended could result in loss of extracts.

1.4.4. Concentrate aliphatic fractions first. There are two aliphatic turbo tubes per sample. Concentrate each extract to approximately 5mL and then combine corresponding extract into one turbo tube for final concentration and vialing.

1.4.5. Once the extract reaches 1mL transfer it using a 1000µL syringe into a pre-labled 2.5mL amber vial with PTFE-lined screw cap using Methylene Chloride. Transfer the yellow aliphatic label tape to this vial. Rinse the syringe three times with Methylene Chloride between extracts.

1.4.6. Concentrate aromatic fractions next. There are two aromatic turbo tubes per sample. Concentrate each extract to approximately 5mL and then combine corresponding extract into one turbo tube for final concentration and vialing.

1.4.7. Once the extract reaches 1mL transfer it using a 1000µL syringe into a pre-labled 2.5mL amber vial with PTFE-lined screw cap using Methylene Chloride. Transfer the white aromatic label tape to this vial. Rinse the syringe three times with Methylene Chloride between extracts.

Task Instructions (TI-017)

Derivitization and Hydrolysis procedures for TBT

Refer to bench sheet for options or requirements.

1. TBT Derivatization

1.1. Transfer the 3 – 4mL hexane extract to a 40mL VOA vial with PTFE lined screw cap.

1.2. Rinse the turbo tube with 0.5 mL of hexane and add this to the 40mL VOA vial as well.

1.3. Add 1½ pipette of 1.5M Hexyl Magnesium Bromide (HexylMgBr) in diethyl ether. Add the reagent slowly: start with 3-4 drops of the reagent; slowly add the rest of the pipette. Bubbling may occur during this process. If the extract solidifies during the reaction, add hexane to return the "solid" to solution and note it on the analyst notes form (3056F)

1.4. Mix extracts by hand or on the vortexer for 30 seconds.

1.5. Allow extracts to sit for 40 minutes. Mix the extracts on the vortexer approximately every ten minutes during this 40 minute period.

1.6. **TBT Hydrolysis**

1.6.1. Hydrolyze excess HexylMgBr by adding 1 pipette of concentrated HCL. Caution: add the HCL drop wise slowly while shaking the scintillation vial. Allow bubbling to occur before adding the rest of the acid. Vortex for 10 seconds.

1.6.2. Draw off the acid layer (bottom) with a pipette and discard. Be careful not to draw off any of the hexane layer.

1.6.3. Add 5 mL of DI water and vortex.

1.6.4. Draw off the water/acid layer (bottom layer) with a pipette and discard. Be careful not to draw off any of the hexane layer.

1.6.5. Repeat 1.6.3 – 1.6.4 a second time.

1.6.6. The extract is dried by adding a small amount of anhydrous sodium sulfate to the vial.

1.6.7. Allow extract to dry for 15 minutes, sulfate should be "free flowing".

1.6.8. The extract is now ready for Alumina Clean-up. See Appendix 20.2.15 Task Instructions (TI-015) Alumina Clean-up for TBT.

Task Instructions (TI-018W)

Separatory Funnel Extraction, Hydrolysis, Clean-up, Back Extraction, and Derivitization procedures for Aqueous Herb and PCP

Refer to bench sheet for options or requirements.

1. Aqueous Herbicide Separatory Funnel/Hydrolysis/Clean-up/Back Extraction

1.1. Transfer the sample to a labeled Separatory Funnel. The bottles are rinsed with 30 mL of DI water containing one pipette of 10N sodium hydroxide. The rinsate is added to the separatory funnel and the contents are mixed well. Verify that the pH of the sample is > 12 using a pipette and narrow range pH paper. Add additional 10N sodium hydroxide drop wise as needed until pH ≥ 12.

1.2. Add surrogates to each sample, MB, LCS and MS as specified on the bench sheet form, 3012F or 3049F. In addition to the surrogates, the LCS and MS receive a full list herbicide spike. To verify that each surrogate and spike addition is accurate, each will be witnessed and documented by another laboratory technician.

1.3. **Hydrolysis:** The samples are mixed well and allowed to stand at room temperature for one hour to allow for the hydrolysis of herbicide esters to the free acids.

1.4. **Clean-up:** After one hour add 60 ml of methylene chloride to the samples and shake the separatory funnel vigorously for two minutes. Allow the phases to separate and drain off the bottom layer, (methylene chloride), to a waste beaker.

1.5. **Back Extraction:** Acidify the sample to pH <2 with 3 pipettes of 1:1 sulfuric acid solution. Mix well. Use narrow-range pH paper to check the pH.

1.6. Prepare a labeled 250 ml Erlenmeyer flask for each sample.

1.7. Add 60ml of 7:3 methylene chloride/ether to the separatory funnel and shake for two minutes, venting frequently. Drain the bottom layer, (methylene chloride), into the corresponding Erlenmeyer flask. Extract the sample <u>two more times</u> using 60 ml of 7:3 methylene chloride/hexane. Each time the bottom layer should be drained into the corresponding Erlenmeyer flask.

1.8. Discard the water in the separatory funnel and transfer the sample label to the 250 ml Erlenmeyer containing the extract. Add approximately 10 gm of acidified (Herb) sodium sulfate. Additional acidified (Herb) sodium sulfate may be added to the extract in sufficient quantity so that some sodium sulfate remains "free flowing". <u>Allow the extract to stand for one hour to remove any water in the solvent before KD concentration.</u>

1.9. Transfer the extract to a KD. Rinse Erlenmeyer flask 2-3 times with hexane. Add rinsate to the KD.

1.10. Add 40mL Hexane to the KD and put on a water bath set at 100°C.

1.11. Concentrate extract to 5-8mL. Place in KD rack and allow to cool for 10 minutes.

1.12. Transfer the extract to a labeled Turbo Tube. Rinse the KD with approximately 5mL of Hexane.

1.13. Concentrate the extract to Volume to Lab listed on the bench sheet. See Appendix 20.2.9, Task Instructions (TI-009) Turbo Vap concentration.

1.14. **Derivitization procedures: for Herb and PCP are derivitized in the GC lab and are detailed in GC SOP's:**

1.14.1. Herbicide (405S)

1.14.2. 8040 Phenol (412S)

Task Instructions (TI-019)

Vialing of final extract
Refer to bench sheet for options or requirements.

1.0 Vialing of final extract

1.1 Once the extract reaches the specified volume to lab for analysis, further clean-up, or specified procedure listed on the bench sheet, transfer the extract using the appropriate syringe and solvent into an appropriate vial. Clean the syringe between extracts by rinsing three times with the appropriate solvent.

1.2 The extract is ready for analysis.

Task Instructions (TI-020)

Reagents and Preparation of Solutions by parameter and matrix:

Preparation of the solutions is documented in the Reagent Preparation Logbook (Form 10076F)
Refer to bench sheet for options or requirements.

1. Pesticide or PCB by Separatory Funnel: Reagents and Solutions for Aqueous extractions

1.1 Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

1.2 Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

1.3 (In house and TCLP): Methylene Chloride, Fisher (D 154-4) or equivalent

1.4 (In house and TCLP): Hexanes, Fisher Optima (H303-4) or equivalent

1.5 (MTCA and Low Level): Methylene Chloride, EMD Omnisolv (DX0831-1) or equivalent

1.6 (MTCA and Low Level): Hexanes, EMD Omnisolv (HX0298-1) or equivalent

1.7 Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours

1.8 Neutral glasswool: prepared by baking in kiln at 400° C for 4 hours

1.9 (PCB acid clean): Concentrated sulfuric acid, Fisher (A300-500) or equivalent

1.10(Pest or PCB Sulfur Clean): Tetrabutylammonium sulfite (TBAS) reagent:

1.10.1 Tetrabutylammonium hydrogensulfate, ACS Grade, Sigma Aldrich (155837-1KG)

1.10.2 Sodium sulfite, Anhydrous, Fisher, (S430-500)

1.10.3 Prepare (TBAS) reagent by dissolving 3.4 grams tetrabutylammonium hydrogensulfate in 100 mL of organic-free Type 1 water from ARI's water purification system. Extract the resulting solution with Pesticide Grade Hexane (20mL Hexane per 100mL reagent) to remove possible impurities. Discard the hexane extracts. Add 25 grams sodium sulfite to the 100 mL aqueous reagent. The resulting solution is saturated with sodium sulfite. Store the reagent in an amber glass bottle with a PTFE-lined cap. Clearly label the bottle including date prepared, preparer's initials, and the expiration date (30 days after preparation).

1.11(Pest only): Ethyl Acetate, Fisher, (E191-4)

1.12Silica Gel (SPE) darts, 3mL/500mg, Supelco Discovery

1.13 Pesticide or PCB by Sonication or Microwave: Reagents and Solutions for Soil/Sediment extractions

1.14 Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

1.15 Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

1.16 (In house and PSDDA): Acetone, EMD (AX0120-8) or equivalent

1.17 (In house and PSDDA): Hexanes, Fisher Optima (H303-4) or equivalent

1.18 (In house and PSDDA): Methylene Chloride, Fisher (D 154-4) or equivalent

1.18.1 80:20 Hexane/Acetone: prepared by mixing 3200mL Hexane/800mL Acetone

1.18.2 50:50 Hexane/Methylene Chloride: prepared by mixing 2000mL Hexane/2000mL Methylene Chloride

1.19 Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours

1.20 Neutral glasswool: prepared by baking in kiln at 400° C for 4 hours

1.21 (PCB acid clean): Concentrated sulfuric acid, Fisher (A300-500) or equivalent

1.22 (Pest or PCB Sulfur Clean): Tetrabutylammonium sulfite (TBAS) reagent:

1.22.1 Tetrabutylammonium hydrogensulfate, ACS Grade, Sigma Aldrich (155837-1KG)

1.22.2 Sodium sulfite, Anhydrous, Fisher, (S430-500)

1.22.3 Prepare (TBAS) reagent by dissolving 3.4 grams tetrabutylammonium hydrogensulfate in 100 mL of organic-free Type 1 water from ARI's water purification system. Extract the resulting solution with Pesticide Grade Hexane (20mL Hexane per 100mL reagent) to remove possible impurities. Discard the hexane extracts. Add 25 grams sodium sulfite to the 100 mL aqueous reagent. The resulting solution is saturated with sodium sulfite. Store the reagent in an amber glass bottle with a PTFE-lined cap. Clearly label the bottle including date prepared, preparer's initials, and the expiration date (30 days after preparation).

1.23 (Pest only): Ethyl Acetate, Fisher, (E191-4)

1.24 Silica Gel (SPE) darts, 3mL/500mg, Supelco Discovery

2. SIM OP Pesticide by Separatory Funnel: Reagents and Solutions for Aqueous extractions

2.1. Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

2.2. Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

2.3. Methylene Chloride, Fisher (D 154-4) or equivalent.

2.4 SIM OP Pesticide by Microwave: Reagents and Solutions for Soil/Sediment extractions

2.5 Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

2.6 Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours.

2.7 (In house): Methylene Chloride, Fisher (D 154-4) or equivalent.

2.8 Neutral glasswool: prepared by baking in kiln at 400° C for 4 hours

3. 8041 PCP by Separatory Funnel: Reagents and Solutions for Aqueous extractions

3.1. Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

3.2. Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

3.3. 1:1 sulfuric acid solution: prepared by diluting 500mL of concentrated sulfuric acid into 500mL of organic free water. **Use extreme caution this process generates heat. Add the acid to a volume of water. Do not add water to a volume of acid.**

3.4. (In house and Low Level): Methylene Chloride, Fisher (D 154-4) or equivalent

3.5. (In house and Low Level): Hexanes, Fisher Optima (H303-4) or equivalent

3.6 8041 PCP by Sonication: Reagents and Solutions for Soil/Sediment extractions

3.7 Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

3.8 Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours.

3.9 Concentrated Sulfuric Acid.

3.10 (In house): Methylene Chloride, Fisher (D 154-4) or equivalent.

3.11 (In house): Hexanes, Fisher Optima (H303-4) or equivalent

4. Herbicide by Separatory Funnel: Reagents and Solutions for Aqueous extractions

4.1. Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

4.2. Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

4.3. 10N sodium hydroxide prepared by dissolving 400g of sodium hydroxide pellets into organic free water and adjusting the volume to 1.0 Liter. **Use extreme caution this process generates heat. Add the NaOH slowly over a period of to allow the heat to dissipate.**

4.4. (In house-Low Level-TCLP): Methylene Chloride, Fisher (D 154-4) or equivalent

4.5. (In house-Low Level-TCLP): Hexanes, Fisher Optima (H303-4) or equivalent

4.6. Ethyl Ether, Fisher Pesticide Grade (E199-4) or equivalent

4.7. 1:1 sulfuric acid solution: prepared by diluting 500mL of concentrated sulfuric acid into 500mL of organic free water. **Use extreme caution this process generates heat. Add the acid to a volume of water. Do not add water to a volume of acid.**

4.8. 70:30 Methylene Chloride/Ether: prepared by mixing 2800mL Methylene Chloride/1200mL Ether

4.9. 70:30 Methylene Chloride/Hexane: prepared by mixing 2800mL Methylene Chloride/1200mL Hexane

4.10. Acidified (Herb) sodium sulfate:

4.10.1. One 32oz jar of neutral Sodium Sulfate (with 1" headspace)

4.10.2. 300mL Ethyl Ether

4.10.3. 1mL concentrated Sulfuric Acid

4.10.4. Prepare by using one 32oz jar (with 1" headspace) of kiln baked sodium sulfate.

4.10.5. Under a hood, measure 300mL of Ethyl Ether into a 500mL graduated cylinder.

4.10.6. Measure 1mL of concentrated sulfuric acid into a 1mL volumetric flask.

4.10.7. Using a pipette, transfer the sulfuric acid to the 500mL graduated cylinder containing the Ethyl Ether. Use 1mL of the Ether/Acid mixture to rinse the volumetric flask once and add the rinsate to the 500mL graduated cylinder containing the Ether/Acid solution.

4.10.8. Mix the Ether/Acid solution well by swirling.

4.10.9. Slowly pour the Ether/Acid solution into the 32oz jar of sodium sulfate. Tighten lid on the 32oz jar and shake until homogenized and the sulfate is saturated.

4.10.10. Pour the acidified sodium sulfate into a clean Pyrex baking dish and spread evenly.

4.10.11. Nest the Pyrex dish with acidified sodium sulfate on top of a empty Pyrex dish and place on a hot plate. Turn hot plate on to the lowest setting.

4.10.12. Cover acidified sodium sulfate dish loosely with Aluminum foil venting to a hood. Be sure to allow adequate ventilation for evaporation of Ether and allow to dry overnight.

4.10.13. After all Ethyl Ether has evaporated, transfer acidified sodium sulfate into 16oz jars and label as "Acidified Sulfate for Herbs" with Red and Pink tape.

4.11 Herbicide by Sonication: Reagents and Solutions for Soil/Sediment extractions

4.12 Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

4.13 Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

4.14 10N sodium hydroxide prepared by dissolving 400g of sodium hydroxide pellets into organic free water and adjusting the volume to 1.0 Liter. ***Use extreme caution this process generates heat. Add the NaOH slowly over a period of time to allow the heat to dissipate.***

4.15 Concentrated Hydrochloric Acid.

4.16 1:1 sulfuric acid solution: prepared by diluting 500mL of concentrated sulfuric acid into 500mL of organic free water. ***Use extreme caution this process generates heat. Add the acid slowly over a period of time to allow the heat to dissipate. Add the acid to a volume of water. Do not add water to a volume of acid.***

4.17 (In house): Methylene Chloride, Fisher (D 154-4) or equivalent

4.18 (In house): Hexanes, Fisher Optima (H303-4) or equivalent

4.19 Ethyl Ether, Fisher Pesticide Grade (E199-4) or equivalent

4.20 50:50 Methylene Chloride/Ether: prepared by mixing 2000mL Methylene Chloride/2000mL Ether

4.21 70:30 Hexane/Methylene Chloride: prepared by mixing 2800mL Hexane/1200mL Methylene Chloride

4.22 70:30 Methylene Chloride/Ether: prepared by mixing 2800mL Methylene Chloride/1200mL Ether

4.23 70:30 Methylene Chloride/Hexane: prepared by mixing 2800mL Methylene Chloride/1200mL Hexane

4.24 Acidified (Herb) Sodium Sulfate (see herb separatory funnel for preparation)

5. TBT by Separatory Funnel: Reagents and Solutions for Aqueous extractions

5.1. Tropolone, 98%, Aldrich (T89702-25g) or equivalent

5.2. 0.02% Tropolone in Methylene Chloride solution: (1.06g Tropolone/4000mL Methylene Chloride).

5.3. Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

5.4. Sea salts, Sigma, (S9883-1KG) or equivalent

5.5. (Pore Water Blanks): Sea Water solution: (144g Sea Salt/4000mL Organic Free Water)

5.6. (In house and Pore Water): Methylene Chloride, Fisher (D 154-4) or equivalent

5.7. Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

5.8. Concentrated hydrochloric acid, Fisher (A144-212) or equivalent

5.9. 1:1 hydrochloric acid solution: prepared by diluting 500mL of concentrated hydrochloric acid into 500mL of organic free water. **Use extreme caution this process generates heat. Add the acid to a volume of water. Do not add water to a volume of acid.**

5.10. (In house and Pore Water): Hexanes, Fisher Optima (H303-4) or equivalent

5.11. 1.5 M Hexyl magnesium bromide in diethyl ether solution:

5.11.1. 1.5L Ethyl Ether, Burdick & Jackson High Purity (107-1) or equivalent

5.11.2. 330g 1-Bromohexane, 99%, AlfaAesar

5.11.3. 50g Magnesium chips, 99.98%, Aldrich

5.11.4. Preparation of 1.5M Hexyl magnesium bromide solution: Obtain a large grey bin 1/3rd full of ice. Add cold tap water to 2/3rd full.

5.11.5. Weigh 50g of Magnesium chips into a 400mL beaker.

5.11.6. Weigh 330g 1-Bromohexane into a 600mL beaker.

5.11.7. Measure 1000mL of Ethyl Ether into a 1.0L graduated cylinder.

5.11.8. Add the 330g 1-Bromohexane to a 6000mL flat bottom flask with 24/40 joint at the top.

5.11.9. Add the 1000mL of Ethyl Ether to the same 6000mL flask.

5.11.10. Add the 50g Magnesium chips to the same 6000mL flask.

5.11.11. Add 24/40 joint condenser to the top of the 6000mL flask.

5.11.12. Swirl gently to activate reaction.

5.11.13. Place 6000mL flask on a hot plate to activate reaction. (When the reaction starts to turn yellow/brown and bubbles appear-the reaction has started).

5.11.14. Add an additional 500mL of Ethyl Ether to the flask. (Take off the condenser and pour through the top of the flask-NOT top of condenser.)

5.11.15. **CAUTION-STAY IN CONTROL OF THE REACTION during the early stages of the reaction. Use ice bath to control the reaction-But do not stall/stop the reaction! Place the 6000mL flask back and forth from hot plate to ice bath to control the reaction. The solvent should be around the fourth rung of the condenser. If it slows-put it back onto the hot plate. If it gets to the top of the condenser-put in back into the ice bath.**

5.11.16. When the reaction is in control....(after sufficient time in the ice bath). Put back onto hot plate to continue controlled reaction for approximately ½ hour. It may be necessary to turn hot plate "off" and "on".

5.11.17. After the reaction is complete, take off of the hot plate and let cool for 1 hour.

5.11.18. Bottle reagent into 2oz amber bottles located in the GC cabinet.

5.11.19. Label with reagent ID.

5.11.20. Test the reagent by adding 100µL TBT surrogate and spike to 10mL of hexane. Concentrate to 4mL for derivitization. Perform the Derivitization, Hydrolyzation. Concentrate to 2mL for Alumina Clean-up. Concentrate to 0.5mL and vial for analysis in hexane.

5.12. Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours.

5.13. Neutral glasswool: prepared by baking in kiln at 400° C for 4 hours

5.14. Activity 0 Alumina (neutral alumina baked at 400°C for 8 hours). Test the alumina with TBT alumina QA spike solution-(Use 100µl for a 0.5mL FEV). Add to 2mL hexane, run through 5g alumina, collect 3-4mL, concentrate to 0.5mL for analysis.

*5.15 **TBT by Sonication or Microwave: Reagents and Solutions for Soil/Sediment extractions***

5.16 Tropolone, 98%, Aldrich (T89702-25g) or equivalent

5.17 0.10% Tropolone in Methylene Chloride solution: (5.25g Tropolone/4000mL Methylene Chloride).

5.18 Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

5.19 (In house): Methylene Chloride, Fisher (D 154-4) or equivalent

5.20 (In house): Hexanes, Fisher Optima (H303-4) or equivalent

5.21 Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

5.22 Concentrated hydrochloric acid, Fisher (A144-212) or equivalent

5.23 1:1 hydrochloric acid solution: prepared by diluting 500mL of concentrated hydrochloric acid into 500mL of organic free water. ***Use extreme caution this process generates heat. Add the acid to a volume of water. Do not add water to a volume of acid.***

5.24 Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours.

5.25 Neutral glasswool: prepared by baking in kiln at 400° C for 4 hours

5.26 Activity 0 Alumina (neutral alumina baked at 400°C for 8 hours). Test the alumina with TBT alumina QA spike solution-(Use 100µl for a 0.5mL FEV). Add to 2mL hexane, run through 5g alumina, collect 3-4mL, concentrate to 0.5mL for analysis.

5.27 1.5 M Hexyl magnesium bromide in diethyl ether solution:: (See TBT by Separatory Funnel for preparation).

6. EPH by Separatory Funnel: Reagents and Solutions for Aqueous extractions

6.1. Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

6.2. Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

6.3. Concentrated sulfuric acid, Fisher (A300-500) or equivalent

6.4. 1:1 sulfuric acid solution: prepared by diluting 500mL of concentrated sulfuric acid into 500mL of organic free water. **Use extreme caution this process generates heat. Add the acid to a volume of water. Do not add water to a volume of acid.**

6.5. (In house): Methylene Chloride, Fisher (D 154-4) or equivalent

6.6. Pentane, Fisher HPLC grade, (P399-4) or equivalent

6.7. (In house): Hexanes, Fisher Optima (H303-4) or equivalent

6.8. 90:10 Pentane/Hexane: prepared by mixing 3600mL Pentane/400mL Hexane

6.9. 60:40 Pentane/Methylene Chloride: prepared by mixing 2400mL Pentane/1600mL Methylene Chloride

6.10. Neutral glasswool: prepared by baking in kiln at 400° C for 4 hours

6.11. Silica Gel, 60-200 mesh, Fisher, (S704-10) or equivalent

6.12. 0% Silica Gel (activated by baking in kiln at 400° C for 8 hours). Test the silica gel by adding 100µL EPH surrogate and spike solutions to 2mL of hexane. Elute and collect 1mL increments and checked by OE GC for proper Aliphatic/Aromatic elution volumes.

6.12 EPH by Sonication: Reagents and Solutions for Soil/Sediment extractions

6.13 Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

6.14 (In house): Methylene Chloride, Fisher (D 154-4) or equivalent

6.15 Pentane, Fisher HPLC grade, (P399-4) or equivalent

6.16 (In house): Hexanes, Fisher Optima (H303-4) or equivalent

6.17 90:10 Pentane/Hexane: prepared by mixing 3600mL Pentane/400mL Hexane

6.18 60:40 Pentane/Methylene Chloride: prepared by mixing 2400mL Pentane/1600mL Methylene Chloride

6.19 Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours.

6.20 Neutral glasswool: prepared by baking in kiln at 400° C for 4 hours

6.21 Silica Gel, 60-200 mesh, Fisher, (S704-10) or equivalent

6.22 0% Silica Gel (activated by baking in kiln at 400° C for 8 hours). Test the silica gel by adding 100µL EPH surrogate and spike solutions to 2mL of hexane. Elute and collect 1mL increments and checked by OE GC for proper Aliphatic/Aromatic elution volumes.

7. NWTPHD/HCID by Separatory Funnel: Reagents and Solutions for Aqueous extractions

7.1. Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

7.2. Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

7.3. Concentrated sulfuric acid, Fisher (A300-500) or equivalent

7.4. 1:1 sulfuric acid solution: prepared by diluting 500mL of concentrated sulfuric acid into 500mL of organic free water. **Use extreme caution this process generates heat. Add the acid to a volume of water. Do not add water to a volume of acid.**

7.5. (In house): Methylene Chloride, Fisher (D 154-4) or equivalent

7.6. Silica Gel, 60-200 mesh, Fisher, (S704-10) or equivalent

7.7. 0% Silica Gel (activated by baking in kiln at 400° C for 8 hours).

*7.8 **NWTPHD/HCID by Sonication or Microwave: Reagents and Solutions for Soil/Sediment extractions***

7.9 Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

7.10 Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours.

7.11 Neutral glasswool: prepared by baking in kiln at 400° C for 4 hours

7.12 Concentrated sulfuric acid, Fisher (A300-500) or equivalent

*7.13 1:1 sulfuric acid solution: prepared by diluting 500mL of concentrated sulfuric acid into 500mL of organic free water. **Use extreme caution this process generates heat. Add the acid to a volume of water. Do not add water to a volume of acid.***

7.14 (In house): Methylene Chloride, Fisher (D 154-4) or equivalent

7.15 Silica Gel (SPE) darts, 3mL/500mg, Supelco Discovery

8. AK102/103 by Separatory Funnel: Reagents and Solutions for Aqueous extractions

8.1. Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

8.2. Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

8.3. Concentrated hydrochloric acid, Fisher (A144-212) or equivalent

8.4. 1:1 hydrochloric acid solution: prepared by diluting 500mL of concentrated hydrochloric acid into 500mL of organic free water. **Use extreme caution this process generates heat. Add the acid to a volume of water. Do not add water to a volume of acid.**

8.5. (In house): Methylene Chloride, Fisher (D 154-4) or equivalent

8.6. Silica Gel, 60-200 mesh, Fisher, (S704-10) or equivalent

8.7. 0% Silica Gel (activated by baking in kiln at 400° C for 8 hours).

*8.8 **AK102/103 by Sonication or Microwave: Reagents and Solutions for Soil/Sediment extractions***

8.9 Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

8.10 Neutral glasswool: prepared by baking in kiln at 400° C for 4 hours

8.11 Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours.

8.12 (In house): Methylene Chloride, Fisher (D 154-4) or equivalent

8.13 Silica Gel (SPE) darts, 3mL/500mg, Supelco Discovery

9. BAN by Separatory Funnel: Reagents and Solutions for Aqueous extractions

9.1. Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

9.2. Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

9.3. Concentrated sulfuric acid, Fisher (A300-500) or equivalent

9.4. 1:1 sulfuric acid solution: prepared by diluting 500mL of concentrated sulfuric acid into 500mL of organic free water. **Use extreme caution this process generates heat. Add the acid to a volume of water. Do not add water to a volume of acid.**

9.5. (In house and TCLP): Methylene Chloride, Fisher (D 154-4) or equivalent

9.6. Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours.

9.7. Sodium hydroxide, Fisher, (S318-500) or equivalent

9.8. 10N sodium hydroxide prepared by dissolving 400g of sodium hydroxide pellets into organic free water and adjusting the volume to 1.0 Liter. **Use extreme caution this process generates heat. Add the NaOH slowly over a period time of to allow the heat to dissipate.**

9.9 BAN (In house/PSDDA/SIM) by Sonication: Reagents and Solutions for Soil/Sediment extractions

9.10 Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

9.11 Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours.

9.12 (In house): Methylene Chloride, Fisher (D 154-4) or equivalent

9.13 (In house): Acetone, EMD (AX0120-8) or equivalent

9.14 50:50 Methylene Chloride/Acetone prepared by mixing prepared by mixing 2000mL Methylene Chloride/2000mL Acetone

9.15 Pre-deactivated anhydrous sodium sulfate:

9.15.1 One 32oz jar of neutral sodium sulfate

9.15.2 100mL Acetone

9.15.3 1mL Organic Free Water

9.15.4 Ten drops concentrated HCL

9.15.5 Prepare by using one 32oz jar (with 1" headspace) of kiln baked sodium sulfate.

9.15.6 Under a hood, using a pipette, add ten drops of concentrated HCL to 1mL of Organic Free Water.

9.15.7 Measure 100mL of Acetone into a graduated cylinder.

9.15.8 Add the acidified water to the Acetone and mix well.

9.15.9 Add the acidified Acetone solution to the 32oz jar of sodium sulfate. Mix well by shaking until homogenized and the sulfate is saturated.

9.15.10 Pour the acidified sodium sulfate into a clean pyrex dish and spread evenly.

9.15.11 Cover acidified sodium sulfate dish loosely with Aluminum foil venting to a hood. Be sure to allow adequate ventilation for evaporation of Acetone allow to dry overnight.

9.15.12 After all Acetone has evaporated, transfer acidified sodium sulfate into a 1.0L Amber Boston Round Wide Mouth Bottle and label "Pre-deactivated sodium sulfate for BAN".

9.16 Pre-deactivated glass wool.

9.16.1 One Designated Grey Bin (Above Sep Funnel Hood)

9.16.2 Green dishwashing gloves (clean)

9.16.3 8.0L Organic Free Water

9.16.4 100mL Concentrated HCL

9.16.5 Six 32oz jars Neutral glass wool

9.16.6 Two clean Pyrex dishes

9.16.7 One hot plate

9.16.8 Aluminum Foil

9.16.9 Prepare by adding approximately 8.0L Organic Free Water to designated Grey Bin (1/2 full).

9.16.10 Add 100mL concentrated HCL to the water in the bin. Mix Well.

9.16.11 Use six 32oz jars of neutral glasswool that has been baked in the kiln. Put glass wool in the acid bath and soak for a few seconds. Make sure it all gets saturated.

9.16.12 Wring the glass wool out well. Pull apart as much as possible and place into a clean Pyrex dish.

9.16.13 Nest glass wool Pyrex dish on top of another empty Pyrex dish. Set on hot plate set to the lowest setting. Tent dish with Aluminum Foil venting to a hood. Make sure the hood is drawing air over the glass wool.

9.16.14 Leave overnight or until dry.

9.16.15 Place dry glass wool into 16oz clear jars and label "Pre-deactivated glass wool for BAN"

9.17 BAN (In house) by Microwave: Reagents and Solutions for Soil/Sediment extractions

9.18 Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

9.19 Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours.

9.20 (In house): Methylene Chloride, Fisher (D 154-4) or equivalent

9.21 Pre-deactivated anhydrous sodium sulfate: (See BAN sonication for preparation).

9.22 Neutral glasswool: prepared by baking in kiln at 400° C for 4 hours

10. BAN and 1,4 Dioxane by Continuous Liquid Liquid: Reagents and Solutions for Aqueous extractions

10.1. Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

10.2. Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

10.3. Concentrated sulfuric acid, Fisher (A300-500) or equivalent

10.4. 1:1 sulfuric acid solution: prepared by diluting 500mL of concentrated sulfuric acid into 500mL of organic free water. **Use extreme caution this process generates heat. Add the acid to a volume of water. Do not add water to a volume of acid.**

10.5. (In house): Methylene Chloride, Fisher (D 154-4) or equivalent

11. (8270) PNA and In house SIM PNA by Separatory Funnel and Continuous Liquid Liquid: Reagents and Solutions for Aqueous extractions

11.1. Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

11.2. Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

11.3. (In house): Methylene Chloride, Fisher (D 154-4) or equivalent

11.4. (In house): Hexanes, Fisher Optima (H303-4) or equivalent

11.5. Silica Gel (SPE) darts, 3mL/500mg, Supelco Discovery

11.6 (8270) PNA and SIM PNA by Sonication: Reagents and Solutions for Soil/Sediment extractions

11.7 Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

11.8 Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours.

11.9 Neutral glasswool: prepared by baking in kiln at 400° C for 4 hours

11.10 (In house): Hexanes, Fisher Optima (H303-4) or equivalent

11.11 (In house): Methylene Chloride, Fisher (D 154-4) or equivalent

11.12 (In house): Acetone, EMD (AX0120-8) or equivalent

11.13 *50:50 Methylene Chloride/Acetone prepared by mixing prepared by mixing 2000mL Methylene Chloride/2000mL Acetone*

11.14 *Silica Gel (SPE) darts, 3mL/500mg, Supelco Discovery*

11.15 ***(8270) PNA and SIM PNA by Microwave: Reagents and Solutions for Soil/Sediment extractions***

11.16 *Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.*

11.17 *Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours.*

11.18 *Neutral glasswool: prepared by baking in kiln at 400° C for 4 hours*

11.19 *(In house): Hexanes, Fisher Optima (H303-4) or equivalent*

11.20 *(In house): Methylene Chloride, Fisher (D 154-4) or equivalent*

11.21 *Silica Gel (SPE) darts, 3mL/500mg, Supelco Discovery*

12. Low Level SIM PNA by Separatory Funnel and Continuous Liquid Liquid: Reagents and Solutions for Aqueous extractions

12.1. Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.

12.2. Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions.

12.3. (Low Level): Methylene Chloride, EMD Omnisolv (DX0831-1) or equivalent

12.4. (Low Level): Hexanes, EMD Omnisolv (HX0298-1) or equivalent

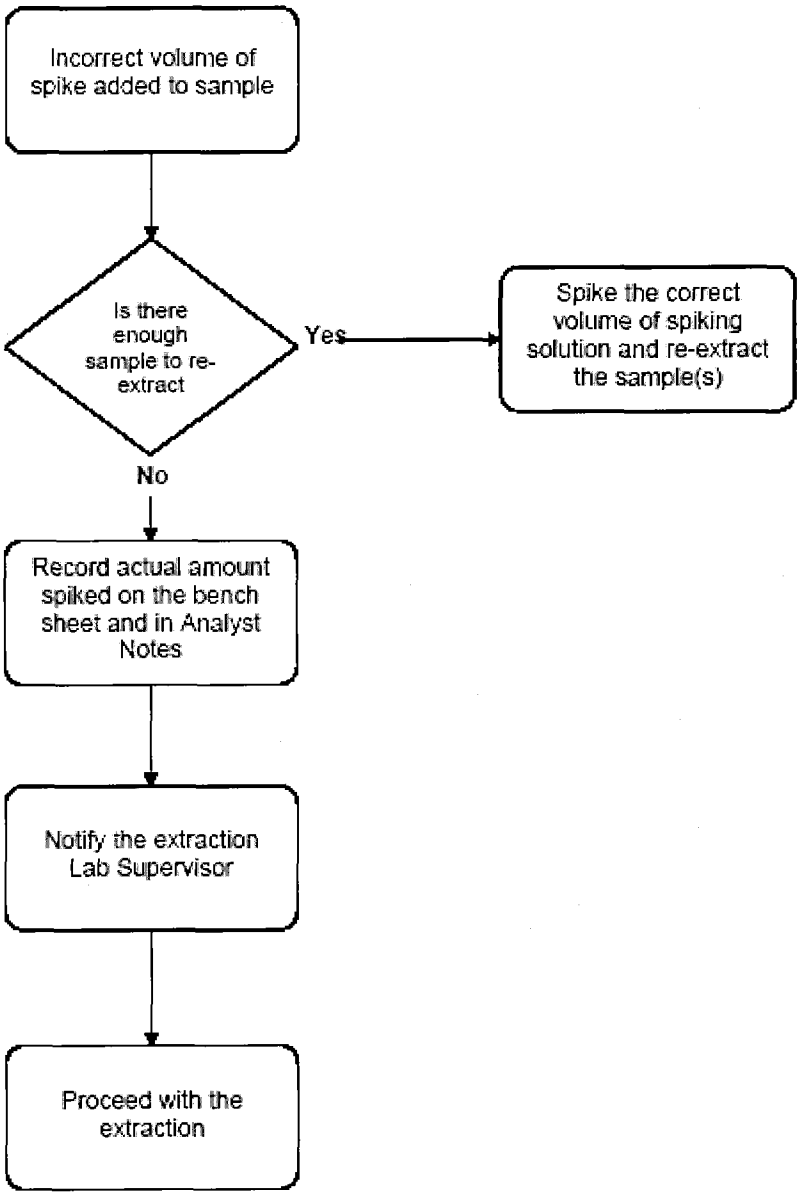
12.5. Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours.

12.6. Silica Gel (SPE) darts, 3mL/500mg, Supelco Discovery

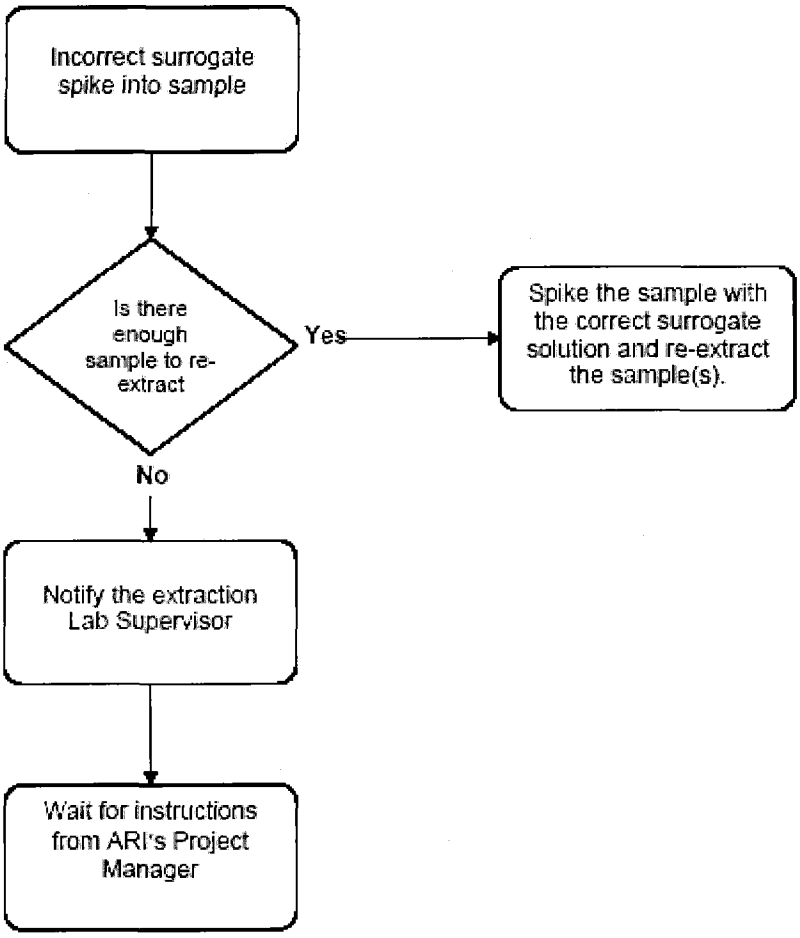
Appendix 20.3

Corrective Action Charts

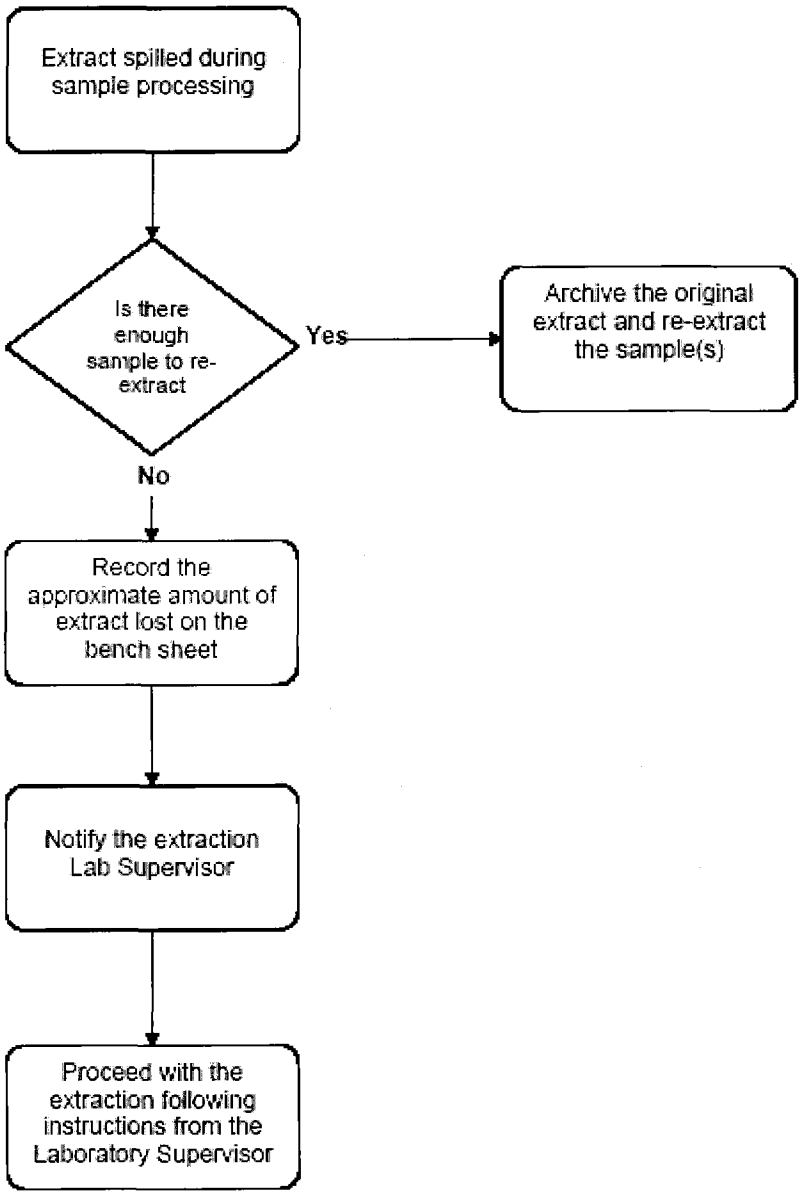
Corrective Action for Incorrect Spike Volume



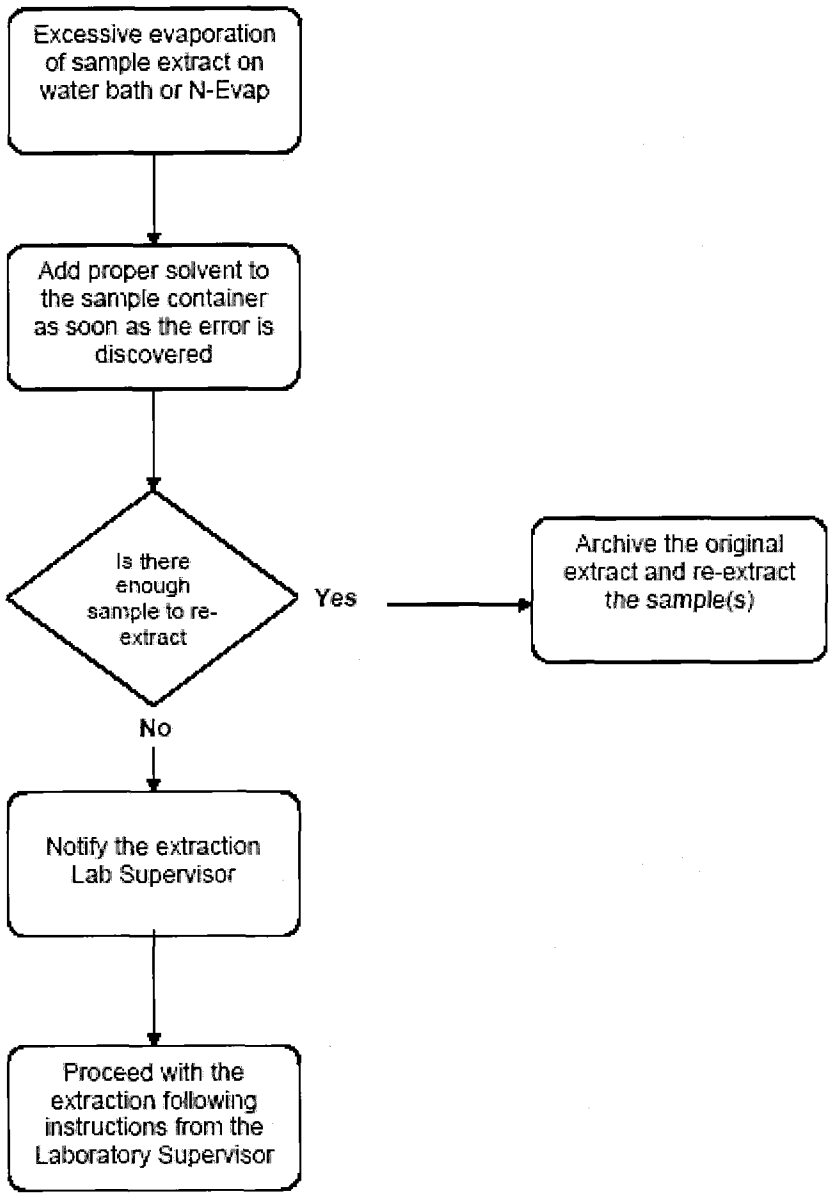
Corrective Action for Incorrect Surrogate Addition



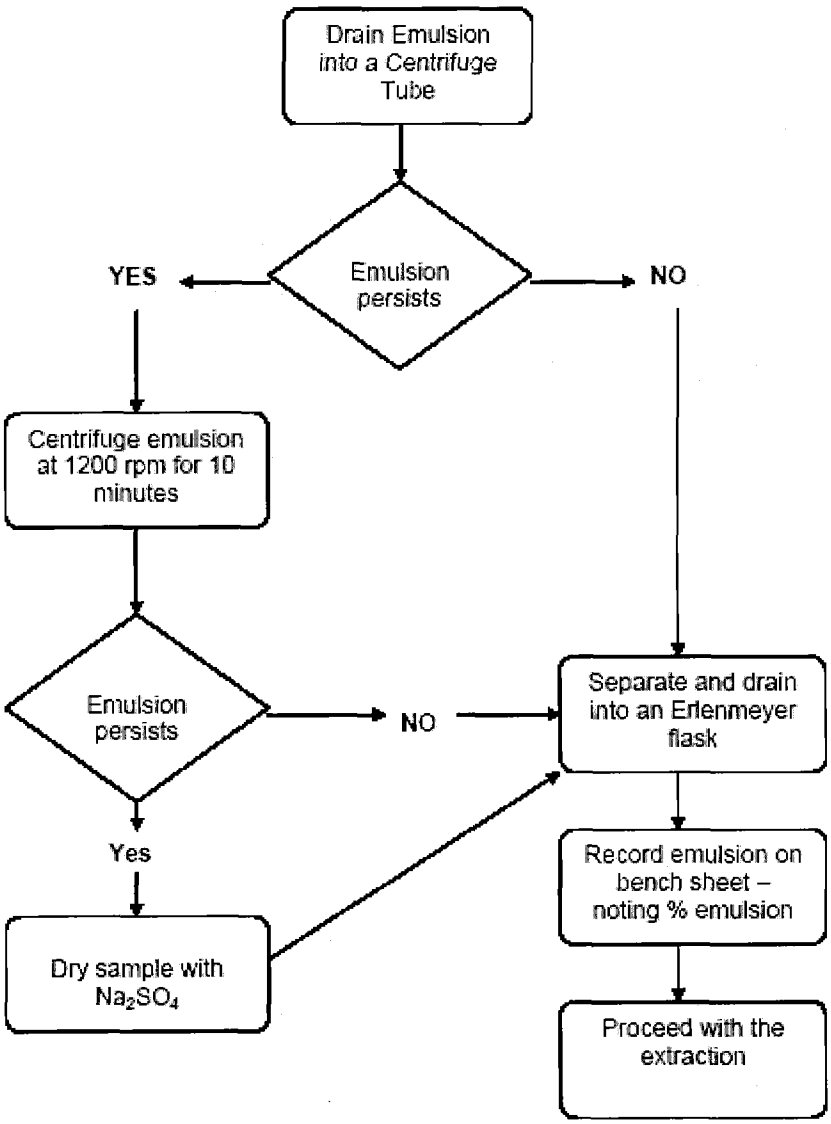
Corrective Action for an Extract Spill



Corrective Action for Loss of Sample on water Bath or N-Evap



Corrective Action for Emulsions



Appendix 20.4

Bench Sheets

Analytical Resources, Incorporated
Analytical Chemists and Consultants

Organic Extractions Benchsheet

SIM PNA-Water
Liquid/Liquid (3520C) (SOP # 3311S)

Preparation Test SIM PNA # 4
ARI Job No(s)_____

Low Level (0.01ppb)
Batch set up by:_____

Bottle #	Extraction Requirements	Verify Client ID	Volume Extracted	Disassemble Liq/Liq	KD Hex X	TurboVap 1 2 3	(REQ) Silica Gel Clean (1:1)	TurboVap 1 2 3	Final Effective Volume	Volume to Lab	Comments
	MBW	Date	500mL						0.5mL	0.5mL	
	SBW		↓						↓	↓	
	SBW Dup.		↓						↓	↓	
Analyst/Date:											

Standard	Standard ID	Volume	Expiration Date	Analyst	Witness
Surrogate	**I**	**100µL**			
Spike	**18**	**100µL**			
Extraction Time:		**Liq/Liq Start:**		**Liq/Liq Stop:**	

SPECIAL INSTRUCTIONS: 1. Rinse all glassware with Low Level DCM. 2. Use 500mL Liq/Liq Body 3. Add 20-25mL Low Level Hexane. 4. Add ~200mL Low Level DCM to Liq/Liq. 5. Add surr/spike. 6. Extract minimum 8 hrs. 7. KD (no drying column) to ~8mL at 80°. 8. Exchange (2 X with 10mL) to Low Level Hexane at 100°. 9. TurboVap. 10. Silica Clean-up=REQUIRED. 11. TurboVap. 12. Vial in Low Level DCM. 13. Post Screen extracts with any color noted for Silica Gel Clean-up.

A. Archive Y / N

Analytical Resources, Incorporated
Analytical Chemists and Consultants

Organic Extractions Benchsheet

SIM PNA-Water
Liquid/Liquid (3520C) (SOP # 3311S)

Preparation Test SIM PNA # 3
ARI Job No(s)_____

In-House (0.1ppb)
Batch set up by:_____

Bottle #	Extraction Requirements	Verify Client ID	Volume Extracted	Disassemble Liq/Liq	KD **Hex X**	Turbo Vap 1 2 3	**(Opt)** Silica Gel Clean **(1:1)** Y / N	Turbo Vap 1 2 3	Final Effective Volume	Volume to Lab	Comments
	MBW	Date	500mL						0.5mL	0.5mL	
	SBW		↓						↓	↓	
	SBW Dup.		↓						↓	↓	
Analyst/Date:											

Standard	Standard ID	Volume	Expiration Date	Analyst	Witness
Surrogate	**B**	**100µL**			
Spike	**15**	**100µL**			
Extraction Time:		**Liq/Liq Start:**		**Liq/Liq Stop:**	

SPECIAL INSTRUCTIONS: 1. Use 500mL Liq/Liq Body 2. Add 20-25mL Hexane. 3. Add ~200mL DCM to Liq/Liq.
4. Add surr/spik. 5. Extract minimum 8 hrs. 6. KD (no drying column) to ~8mL at 80°.
7. Exchange (2 X with 10mL) to Hexane at 100°. 8. TurboVap. 9. Silica Clean-up Opt-Any Color=REQ (All or none).
10. TurboVap (if Silica Clean). 11. Vial in DCM. A. Archive Y / N

Analytical Resources, Incorporated
Analytical Chemists and Consultants

Organic Extractions Benchsheet

(8270) PNA-Water
Liquid/Liquid (3520C) (SOP # 3311S)

Preparation Test PNA # 3
ARI Job No(s)_____

In-House (1.0ppb)
Batch set up by:_____

Bottle #	Extraction Requirements	Verify Client ID	Volume Extracted	Disassemble Liq/Liq	KD **Hex X**	Turbo Vap 1 2 3	**(Opt)** Silica Gel Clean **(1:1)** Y / N	Turbo Vap 1 2 3	Final Effective Volume	Volume to Lab	Comments
	MBW	Date	500mL						0.5mL	0.5mL	
	SBW		↓						↓	↓	
	SBW Dup.		↓						↓	↓	
Analyst/Date:											

Standard	Standard ID	Volume	Expiration Date	Analyst	Witness
BAN Surrogate	**A**	**125µL**			
8270 PNA Spike	**20**	**125µL**			
Extraction Time:		**Liq/Liq Start:**		**Liq/Liq Stop:**	

SPECIAL INSTRUCTIONS: 1. Add ~200mL DCM to Liq/Liq. 2. Extract minimum 8 hrs.

3. KD (no drying column) to 8mL at 80°. 4. Exchange (2 X with 10mL) to Hexane at 100°. 5. TurboVap.

6. Silica Clean-up (All or none) Y / N. 7. TurboVap (if Silica Clean). 8. Vial in DCM. A. Archive Y / N

Analytical Resources, Incorporated
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Organic Extractions Benchsheet

(8270)1,4 Dioxane –Water Liquid/Liquid (3520C) (SOP # 3311S)

Preparation Test BAN # 3
ARI Job No(s)_____

In-House (2.0ppb)
Batch set up by: _____

Bottle #	Extraction Requirements	Verify Client ID	Volume Extracted	Disassemble Liq/Liq	KD	TurboVap 1 2 3	Final Effective Volume	Volume to Lab	Comments
	MBW	Date	500mL				1mL	1mL	
	SBW		↓				↓	↓	
	SBW Dup		↓				↓	↓	
Analyst/Date:									

Standard	Standard ID	Volume	Expiration Date	Analyst	Witness
1,4 Dioxane Surrogate	**G**	**250µL**			
1,4 Dioxane Spike	**16**	**250µL**			
Extraction Time:		**Liq/Liq Start:**		**Liq/Liq Stop:**	

SPECIAL INSTRUCTIONS: 1. Add ~200mL DCM to Liq/Liq. 2. Add 500mL sample + 500mL DI H2O. 3. Add surr/spk. 4. Adjust Acid (pH <2) using 1:1 Sulfuric Acid. (1/4 pipet for blanks & 1/2 pipet for samples). Stir to mix. Verify pH! Let sit 10 minutes. Verify pH again. 5. Extract minimum 8 hrs. 6. KD to 8mL at 80°. 7. TurboVap (do not concentrate less than 1mL). 8. Vial in DCM.

A. Archive Y / N

Analytical Resources, Incorporated
Analytical Chemists and Consultants

Organic Extractions Benchsheet

8270 BAN –Water
Liquid/Liquid (3520C) (SOP # 3311S)

Preparation Test BAN # 2
ARI Job No(s)_____

In-House (1.0-10.0ppb)
Batch set up by: _____

Bottle #	Extraction Requirements	Verify Client ID	Volume Extracted	Disassemble Liq/Liq	KD Or DryVap	TurboVap 1 2 3	Final Effective Volume	Volume to Lab	Comments
	MBW	Date	500mL				0.5mL	0.5mL	
	SBW		↓				↓	↓	
	SBW Dup		↓				↓	↓	
Analyst/Date:									

Standard	Standard ID	Volume	Expiration Date	Analyst	Witness
Surrogate	**A**	**125µL**			
Full List Spike (Freezer)	**7**	**125µL**			
Base Spike	**12**	**125µL**			
Acid Spike	**10**	**125µL**			
Extraction Time:		**Liq/Liq Start:**		**Liq/Liq Stop:**	

SPECIAL INSTRUCTIONS: 1. Add ~200mL DCM to Liq/Liq. 2. Add 500mL sample + 500mL DI H2O. 3. Add surr/spk. 4. Adjust Acid (pH <2) using 1:1 Sulfuric Acid. (1/4 pipet for blanks & 1/2 pipet for samples). Stir to mix. Verify pH! Let sit 10 minutes. Verify pH again. 5. Extract minimum 8 hrs. 6. DryVap to 0.5mL or KD (NO drying column) to 8mL at 80°. 7. (If KD)=TurboVap to 0.5mL. 8. Vial in DCM.

A. Archive Y / N

Analytical Resources, Incorporated
Analytical Chemists and Consultants

Organic Extractions Benchsheet

SIM PNA-Water
Separatory Funnel (3510C) (SOP # 3311S)
Low Level (0.01ppb)

Preparation Test SIM PNA # 6
ARI Job No(s)_____

Batch set up by:_____

Bottle #	Extraction Requirements	Verify Client ID	Volume Extracted	KD **Exchange to Hexane (X2)**	Turbo Vap 1 2 3	**(REQ)** Silica Gel Clean **(1:1)** Y / N	Turbo Vap 1 2 3	Final Effective Volume	Volume to Lab	Comments
	MBW	Date	500mL					0.5mL	0.5mL	
	SBW		↓					↓	↓	
	SBW Dup.		↓					↓	↓	
Analyst/Date:										

Standard	Standard ID	Volume	Expiration Date	Analyst	Witness
Surrogate	**I**	**100µL**			
Spike	**18**	**100µL**			
Extraction Time:					

SPECIAL INSTRUCTIONS: 1. Rinse all glassware with Low Level DCM. 2. Extract 3X with 30mL Low Level DCM.

3. KD (no drying column) to ~8mL at 80°. 4. Exchange (2 X with 10mL) to Low Level Hexane at 100°. 5. TurboVap.

6. Silica Clean-up=REQUIRED. 6. TurboVap. 7. Vial in Low Level DCM. 8. Post screen extracts with any color.
A. Archive Y / N

Analytical Resources, Incorporated
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Organic Extractions Benchsheet

SIM PNA-Water
Separatory Funnel (3510C) (SOP # 3311S)

Preparation Test SIM PNA # 2 In House (0.1ppb)

ARI Job No(s)_____ Batch set up by:_____

Bottle #	Extraction Requirements	Verify Client ID	Volume Extracted	KD **Hex X**	TurboVap 1 2 3	**(Opt)** Silica Gel Clean **(1:1)** Y / N	TurboVap 1 2 3	Final Effective Volume	Volume to Lab	Comments
	MBW	Date	500mL					0.5mL	0.5mL	
	SBW		↓					↓	↓	
	SBW Dup.		↓					↓	↓	
Analyst/Date:										

Standard	Standard ID	Volume	Expiration Date	Analyst	Witness
Surrogate	**B**	**100µL**			
Spike	**15**	**100µL**			
Extraction Time:					

SPECIAL INSTRUCTIONS: 1. Extract 3X with 30mL DCM. 2. KD (no drying column) to ~8mL at 80°.

3. Exchange (2 X with 10mL) to Hexane at 100°. 4. TurboVap. 5. Silica Clean-up Opt-Any Color=REQ (All or none).

6. TurboVap (if Silica Clean). 7. Vial in DCM. A. Archive Y / N

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Organic Extractions Benchsheet

(8270) PNA-Water
Separatory Funnel (3510C) (SOP # 3311S)

Preparation Test PNA # 2
ARI Job No(s)_____

In-House (1.0ppb)
Batch set up by:_____

Bottle #	Extraction Requirements	Verify Client ID	Volume Extracted	KD **Hex X**	TurboVap 1 2 3	**(Opt)** Silica Gel Clean **(1:1)** Y / N	TurboVap 1 2 3	Final Effective Volume	Volume to Lab	Comments
	MBW	Date	500mL					0.5mL	0.5mL	
	SBW		↓					↓	↓	
	SBW Dup.		↓					↓	↓	
Analyst/Date:										

Standard	Standard ID	Volume	Expiration Date	Analyst	Witness
BAN Surrogate	**A**	**125µL**			
8270 PNA Spike	**20**	**125µL**			
Extraction Time:					

SPECIAL INSTRUCTIONS: 1. Extract 3X with 30mL DCM. 2. KD (no drying column) to 8mL at 80°.

3. Exchange (2 X with 10mL) to Hexane at 100°. 4. TurboVap. 5. Silica Clean-up (<u>All or none</u>) Y / N.

6. TurboVap (if Silica Clean). 7. Vial in DCM. A. Archive Y / N

Analytical Resources, Incorporated
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Organic Extractions Benchsheet

8270 BAN –TCLP
Separatory Funnel (3510C) (SOP # 3311S)

Preparation Test BAN # 4
ARI Job No(s)_____

TCLP (10-50ppb)
Batch set up by: _____

Bottle #	Extraction Requirements	Verify Client ID	Volume Extracted	KD	TurboVap 1 2 3	Final Effective Volume	Volume to Lab	Comments
	MBW	Date	100mL			1mL	1mL	**(Blanks provided by Metals Prep).**
	SBW		↓			↓	↓	
	SBW Dup		↓			↓	↓	
Analyst/Date:								

Standard	Standard ID	Volume	Expiration Date	Analyst	Witness
Surrogate	**A**	**250µL**			
Full List Spike (Freezer)	**7**	**250µL**			
Base Spike	**12**	**250µL**			
Acid Spike	**10**	**250µL**			
Extraction Time:					

SPECIAL INSTRUCTIONS: 1. Use MB provided by Metals Prep for MB/SB. 2. Add surr/spk.
3. Adjust Acid (pH <2) using 1:1 Sulfuric Acid. (1/4 pipet for blanks & 1/2 pipet for samples). Verify pH! 4. Extract 1X 60mL DCM, Plus 2X 30mL DCM. 5. Adjust Basic (pH >12) using 1 pipet 10 N Sodium Hydroxide solution. Verify pH!
6. Extract 1X 60mL DCM, Plus 2X 30mL DCM. 7. Pour Acid Fraction first into KD then Basic Fraction into Acid.
8. KD to 8mL at 80. 9. TurboVap (do not concentrate less than 1mL). 10. Vial in DCM.
A. Archive Y / N

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Organic Extractions Benchsheet

8270 BAN –Water
Separatory Funnel (3510C) (SOP # 3311S)

Preparation Test BAN # 1
ARI Job No(s)_____

In-House (1.0-5.0ppb)
Batch set up by: _____

Bottle #	Extraction Requirements	Verify Client ID	Volume Extracted	KD	TurboVap 1 2 3	Final Effective Volume	Volume to Lab	Comments
	MBW	Date	500mL			0.5mL	0.5mL	
	SBW		↓			↓	↓	
	SBW Dup		↓			↓	↓	
Analyst/Date:								

Standard	Standard ID	Volume	Expiration Date	Analyst	Witness
Surrogate	**A**	**125µL**			
Full List Spike (Freezer)	**7**	**125µL**			
Base Spike	**12**	**125µL**			
Acid Spike	**10**	**125µL**			
Extraction Time:					

SPECIAL INSTRUCTIONS: 1. Add surr/spk. 2. Adjust Acid (pH <2) using 1:1 Sulfuric Acid. (1/4 pipet for blanks & 1/2 pipet for samples). Verify pH! 3. Extract 1X 60mL DCM, Plus 2X 30mL DCM. 4. Adjust Basic (pH >12) using 1 pipet 10 N Sodium Hydroxide solution. Verify pH! 5. Extract 1X 60mL DCM, Plus 2X 30mL DCM.
5. Pour Acid Fraction first into KD then Basic Fraction into Acid. 6. KD to 8mL at 80°. 7. TurboVap to 0.5mL.
8. Vial in DCM.

A. Archive Y / N

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Organic Extractions Benchsheet

AK102/103 –Water
Separatory Funnel (3510C) (SOP # 3311S)

Preparation Test AK102/103 # 1
ARI Job No(s)_____

In-House (0.25-0.50 ppm)
Batch set up by:_____

Bottle #	Extraction Requirements	Verify Client ID	Volume Extracted	DryVap Only	Silica Gel Clean (1:1) Y / N	Turbo Vap 1 2 3	Final Effective Volume	Volume to Lab	Comments
	MBW	Date	500mL		1mL		1mL	1mL	
	SBW		↓		↓		↓	↓	
	SBW Dup.		↓		↓		↓	↓	
Analyst/Date:									

Standard	Standard ID	Volume	Expiration Date	Analyst	Witness
Surrogate	**O**	**100µL**			
Spike	**19**	**200µL**			
Extraction Time:					

SPECIAL INSTRUCTIONS: 1. Check pH 2 (samples should be preserved with HCL).

2. If pH is not <2=add 1 pipet of 1:1 HCL and check pH again. (Note on Analyst Notes). 3. Add Surr/Spk.

4. Extract 3X with 30mL DCM. 5. Initial concentration by DryVap Only. 6. Silica Gel Clean-up? Y / N.

7. TurboVap is Silica-gel Cleaned 8. Vial in DCM.

A. Archive Y / N

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Organic Extractions Benchsheet

NWTPHD / NWHCID –Water
Separatory Funnel (3510C) (SOP # 3311S)

Preparation Test TPHD/HCID # 1
ARI Job No(s)_____

In-House (0.25-0.50ppm)
Batch set up by:_____

Bottle #	Extraction Requirements	Verify Client ID	Volume Extracted	DryVap Or KD	Turbo Vap 1 2 3	Acid/Silica Clean (1:1) Y / N	Final Effective Volume	Volume to Lab	Comments
	MBW	Date	500mL			1mL	1mL	1mL	
	SBW		↓			↓	↓	↓	
	SBW Dup.		↓			↓	↓	↓	
Analyst/Date:									

Standard	Standard ID	Volume	Expiration Date	Analyst	Witness
Surrogate	**O**	**100µL**			
Spike	**11**	**100µL**			
Extraction Time:					

SPECIAL INSTRUCTIONS: 1. Add Surr/Spk. 2. Acidify with 1 pipet of 1:1 Sulfuric Acid. 3. Check pH.

4. Extract 2X with 30mL DCM. 5. DryVap or KD at 80°. 6. TurboVap if KD. 7. Acid/Silica Clean-ups? Y / N.

8. Vial in DCM. **A. Archive Y / N**

Analytical Resources, Incorporated
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Organic Extractions Benchsheet

EPH – Water
Separatory Funnel (3510C) (SOP # 3311S)

Preparation Test EPH # 2
ARI Job No(s)_____

In-House (40ppb)
Batch set up by:_____

Bottle #	Extraction Requirements	Verify Client ID	Volume Extracted	KD	Turbo Vap 1 2 3	Fractionate Aromatic/Aliphatic	Turbo Vap 1 2 3	Final Effective Volume		Volume to Lab		Comments
								AR	AL	AR	AL	
	MBW	Date	500mL					1mL	1mL	1mL	1mL	
	SBW		↓					↓	↓	↓	↓	
	SBWDup		↓					↓	↓	↓	↓	
Analyst/Date:												

Standard	Standard ID	Volume	Expiration Date	Analyst	Witness
Surrogate	**M**	**100µL**			
Spike	**5**	**100µL**			
Extraction Time:					

SPECIAL INSTRUCTIONS: 1. Add surr/spk. 2. Acidify all with 1:1 Sulfuric Acid. 3. Extract 3X with 30mL DCM. 4. KD (*no drying column*) to 1mL at 80°C. 5. Exchange with 30mL 90:10 Pentane/Hexane. 6. KD to 1mL at 100°C. 7. Let Cool: After cooling: volume should be 5mL. 8. TurboVap to 2mL 9. Fractionate Aromatic/Aliphatic-collect each fractions extract into (2) Turbo Tubes. 10. TurboVap to 1mL. 11. Vial both fractions in DCM.
A. Archive Y / N

Analytical Resources, Incorporated
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Organic Extractions Benchsheet

Tributyl Tin – Water
Separatory Funnel (3510C) (SOP # 3311S)

Preparation Test TBT # 1

In-House (0.20-0.30ppb)

ARI Job No(s)______________________

Batch set up by: ________

ottle #	Extraction Requirements	Date	Volume Extracted	KD **Exchange To Hexane (X 2)**	Turbo Vap 1 2 3	**HexMgBr Addition**	**(REQ)** Derivitized **(1:1)**	**(REQ)** Alumina Clean **(1:1)**	Turbo Vap 1 2 3	Final Effective Volume	Volume to Lab	Comments
	MB		100mL				4mL	2mL		0.5mL	0.5mL	
	SB		↓				↓	↓		↓	↓	
	SB Dup.		↓				↓	↓		↓	↓	
alyst/Date:												

Standard	Standard ID	Volume	Expiration Date	Analyst	Witness
Surrogate	L	100µL			
Spike	8	100µL			
Extraction Time:					

SPECIAL INSTRUCTIONS: 1. Rinse all glassware with 0.02% Tropolone. 2. Pre-wash blanks with 30mL DCM (2min shake) (Discard DCM). 3. Add Surr/Spk. 4. Acidify with 1:1 HCL. 5. Extract 1 X with 30mL 0.02% Tropolone (4 min shake-SHAKE VIGOROUSLY). Plus 2 X 30mL DCM. 6. KD rinsed with 0.02% Tropolone (NO Drying Column) at 80°. 7. Exchange (2 X with 10mL) to Hexane at 100°. 8. *TurboVap to 3mL-Transfer* with Hexane to 40mL VOA vial.. 9. Derivitize=1 1/2 pipet HexMgBr (Mix by hand). Let sit 45min (mix every 10 min). Add (1) pipet conc. HCL. Vortex. Draw off/discard HCL. Add 5mL DI H2O. Vortex. Draw off/discard H20. Add 5mL DI H2O a second time. Vortex. Draw off/discard H2O. 10. Add sodium sulfate-Let sit 15min. 11. Transfer to Turbo Tube and concentrate to 2mL for Alumina Clean-up. 12. 5gram 0% Alumina Clean-up required using medium column. 13. TurboVap. 14. Vial in Hexane. A. Archive Y / N

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Organic Extractions Benchsheet

Tributyl Tin – Pore Water
Separatory Funnel (3510C) (SOP # 3311S)

Preparation Test TBT # 2

ARI Job No(s)________________________

Pore Water (0.0075-0.01ppb)
Batch set up by: ________

ottle #	Extraction Requirements	Date	Volume Extracted	KD **Exchange To Hexane (X 2)**	Turbo Vap 1 2 3	**HexMgBr Addition**	**(REQ)** Derivitized **(1:1)**	**(REQ)** Alumina Clean **(1:1)**	Turbo Vap 1 2 3	Final Effective Volume	Volume to Lab	Comments
	MBW		100mL				4mL	2mL		0.5mL	0.5mL	**Blanks= Sea H20**
	SBW		↓				↓	↓		↓	↓	↓
	SBWDup.		↓				↓	↓		↓	↓	↓
alyst/Date:												

Standard	Standard ID	Volume	Expiration Date	Analyst	Witness
Surrogate	J	100μL			
Spike	9	100μL			
Extraction Time:					

SPECIAL INSTRUCTIONS:

1. Rinse all glassware with 0.02% Tropolone.
2. Pre-wash "Sea Water" blanks with 30mL DCM (2min shake) (Discard DCM). 3. Add Surr/Spk. 4. Acidify with 1:1 HCL.
5. Extract 1 X with 30mL 0.02% Tropolone (4 min shake-SHAKE VIGOROUSLY)!. Plus 2 X 30mL DCM.
6. KD rinsed with 0.02% Tropolone (NO Drying Column) at 80°. 7. Exchange (2 X with 10mL) to Hexane at 100°.
8. TurboVap to 3mL-Transfer with Hexane to 40mL VOA vial.. 9. Derivitize=1 1/2 pipet HexMgBr (Mix by hand). Let sit 45min (mix every 10 min). Add (1) pipet conc. HCL. Vortex. Draw off/discard HCL. Add 5mL DI H2O. Vortex. Draw off/discard H20. Add 5mL DI H2O a second time. Vortex. Draw off/discard H2O. 10. Add sodium sulfate-Let sit 15min.
11. Transfer to Turbo Tube and concentrate to 2mL for Alumina Clean-up 12. 5g 0% Alumina Clean-up Required.
13. TurboVap. 14. Vial in Hexane. A. Archive Y / N

Analytical Resources, Incorporated
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Organic Extractions Benchsheet

Herbicide – TCLP
Separatory Funnel (3510C) (SOP # 3311S)

Preparation Test Herb # 4 TCLP (1.25-1250ppb)

ARI Job No(s)_____ Batch set up by:_____

Bottle #	Extraction Requirements	Verify Client ID	Volume Extracted	Hydrolysis **(DIH20+KOH) Ph 12** **Mix Well Let Sit For 1 Hour**	Cleanup + Back Ext	KD **Add 40mL Hexane**	Final Effective Volume	Volume to Lab	Derivitize	Comments
	MBW	Date	100mL				50mL	10mL		**(TCLP Blanks provided by Metals Prep).**
	SBW		↓				↓	↓		↓
	SBW Dup		↓				↓	↓		↓
Analyst/Date:										

Standard	Standard ID	Volume	Expiration Date	Analyst	Witness
Surrogate	**E**	**100µL**			
Spike	**22**	**100µL**			
Extraction Time:					

SPECIAL INSTRUCTIONS: : 1. Measure 100mL sample and pour into Sep. Funnel. 2. Add surr/spk. 3. IF No Bottle Rinse=Add 30mL DI H20+1 pipet 10N Sodium Hydroxide to Sep. Funnel and Mix well. 4. Check pH 12. 5. Hydrolysis: Let sit @ pH 12 for one hour. 6. Cleanup: 1X 60 mL DCM to waste. 7. Acidify to pH 2 with 1:1 sulfuric acid. 8. Check pH 2. 9. Back Extract: Extract 1 X 60mL 7:3 DCM/Ether. 10. Extract 2 X 60mL 7:3 DCM/Hexane. 11. Add 10g Acidified (Herb) sodium sulfate in collection flasks. Let sit for 1 hour. 12. Transfer to KD (NO drying column) and rinse E-flask with 40mL Hexane (Add to KD). 13. KD at 100° to approx 5mL. 14. Vial at10mL using Hexane in Herb Tubes. 15. GC Analyst to Derivitize.

A. Archive Y / N

Analytical Resources, Incorporated
Analytical Chemists and Consultants

Organic Extractions Benchsheet

Herbicide – Low Level Water
Separatory Funnel (3510C) (SOP # 3311S)

Preparation Test Herb # 3
ARI Job No(s)_____

Low Level (0.05-50ppb)
Batch set up by:_____

Bottle #	Extraction Requirements	Verify Client ID	Volume Extracted	Hyrolysis **(DIH20+NaOH) Ph 12** **Mix Well Let Sit For 1 Hour**	Cleanup + Back Ext	KD **Add 40mL Hexane**	Final Effective Volume	Volume to Lab	Derivitize	Comments
	MBW	Date	500mL				10mL	10mL		
	SBW		↓				↓	↓		
	SBW Dup		↓				↓	↓		
Analyst/Date:										

Standard	Standard ID	Volume	Expiration Date	Analyst	Witness
Surrogate	**E**	**20µL**			
Spike	**22**	**20µL**			
Extraction Time:					

SPECIAL INSTRUCTIONS: 1. Measure 500mL sample and pour into Sep. Funnel. 2. Add surr/spk. 3. Bottle Rinse=Add 30mL DI H20+1 pipet 10N Sodium Hydroxide to bottle. Add rinsate to Sep. Funnel and Mix well. 4. Check pH 12. 5. Hydrolysis: Let sit @ pH 12 for one hour. 6. Cleanup: 1X 60 mL DCM to waste. 7. Acidify to pH 2 with 1:1 sulfuric acid. 8. Check pH 2. 9. Back Extract: Extract 1 X 60mL 7:3 DCM/Ether. 10. Extract 2 X 60mL 7:3 DCM/Hexane. 11. Add 10g Acidified (Herb) sodium sulfate in collection flasks. Let sit for 1 hour. 12. Transfer to KD (NO drying column) and rinse E-flask with 40mL Hexane (Add to KD). 13. KD at 100° to approx 5mL. 14. Vial at10mL using Hexane in Herb Tubes. 15. GC Analyst to Derivitize.

A. Archive Y / N

Analytical Resources, Incorporated
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Organic Extractions Benchsheet

Herbicide – Water
Separatory Funnel (3510C) (SOP # 3311S)

Preparation Test Herb # 1
ARI Job No(s)_____

In-House (0.25-250ppb)
Batch set up by:_____

Bottle #	Extraction Requirements	Verify Client ID	Volume Extracted	Hydrolysis **(DIH20+NaOH) Ph 12** **Mix Well Let Sit For 1 Hour**	Cleanup + Back Ext	KD **Add 40mL Hexane**	Final Effective Volume	Volume to Lab	Derivitize	Comments
	MBW	Date	500mL				50mL	10mL		
	SBW		↓				↓	↓		
	SBW Dup		↓				↓	↓		
Analyst/Date:										

Standard	Standard ID	Volume	Expiration Date	Analyst	Witness
Surrogate	**E**	**100µL**			
Spike	**22**	**100µL**			
Extraction Time:					

SPECIAL INSTRUCTIONS: 1. Measure 500mL sample and pour into Sep. Funnel. 2. Add surr/spk. 3. Bottle Rinse=Add 30mL DI H20+1 pipet 10N Sodium Hydroxide to bottle. Add rinsate to Sep. Funnel and Mix well. 4. Check pH 12. 5. Hydrolysis: Let sit @ pH 12 for one hour. 6. Cleanup: 1X 60 mL DCM to waste. 7. Acidify to pH 2 with 1:1 sulfuric acid. 8. Check pH 2. 9. Back Extract: Extract 1 X 60mL 7:3 DCM/Ether. 10. Extract 2 X 60mL 7:3 DCM/Hexane. 11. Add 10g Acidified (Herb) sodium sulfate in collection flasks. Let sit for 1 hour. 12. Transfer to KD (NO drying column) and rinse E-flask with 40mL Hexane (Add to KD). 13. KD at 100° to approx 5mL. 14. Vial at10mL using Hexane in Herb Tubes. 15. GC Analyst to Derivitize.

A. Archive Y / N

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Organic Extractions Benchsheet

8041 PCP – Low Level Water
Separatory Funnel (3510C) (SOP # 3311S)

Preparation Test PCP # 2

ARI Job No(s)_____

Low Level (0.05ppb)
Batch set up by:_____

Bottle #	Extraction Requirements	Verify Client ID	Volume Extracted	KD **Exchange To Hexane (X 2)**	Turbo Vap 1 2 3	Final Effective Volume	Volume to Lab	Derivitize	Comments
	MB	Date	500mL			10mL	1-2mL		
	SB		↓			↓	↓		
	SB Dup.		↓			↓	↓		
Analyst/Date:									

Standard	Standard ID	Volume	Expiration Date	Analyst	Witness
Surrogate	**F**	**20µL**			
Spike	**6**	**20µL**			
Extraction Time:					

SPECIAL INSTRUCTIONS: 1. Add surr/spike. 2. Acidify all with 1:1 Sulfuric Acid 3. Extract 3X with 30mL DCM.

4. KD (NO Drying Column) at 80° to 5mL. 5. Exchange (2 X with 20mL) Hexane at 100°. 6. Turbo Vap to 1-2mL.

7. Pipet using Hexane into Herb Tubes. 8. GC Analyst to Derivitize. A. Archive Y / N

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Organic Extractions Benchsheet

8041 PCP – Water
Separatory Funnel (3510C) (SOP # 3311S)

Preparation Test PCP # 1

In-House (0.25ppb)

ARI Job No(s)_____

Batch set up by:_____

Bottle #	Extraction Requirements	Verify Client ID	Volume Extracted	KD **Exchange To Hexane (X 2)**	Turbo Vap 1 2 3	Final Effective Volume	Volume to Lab	Derivitize	Comments
	MB	Date	500mL			50mL	1-2mL		
	SB		↓			↓	↓		
	SB Dup.		↓			↓	↓		
Analyst/Date:									

Standard	Standard ID	Volume	Expiration Date	Analyst	Witness
Surrogate	**F**	**100µL**			
Spike	**6**	**100µL**			
Extraction Time:					

SPECIAL INSTRUCTIONS: 1. Add surr/spike. 2. Acidify all with 1:1 Sulfuric Acid 3. Extract 3X with 30mL DCM.

4. KD (NO Drying Column) at 80° to 5mL. 5. Exchange (2 X with 20mL) Hexane at 100°. 6. Turbo Vap to 1-2mL

7. Pipet using Hexane into Herb Tubes. 8. GC Analyst to Derivitize. A. Archive Y / N

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Organic Extractions Benchsheet

SIM OP Pest–Water

Separatory Funnel (3510C) (SOP # 3311S)

In-House (0.20-1.0ppb)

Preparation Test SIM OP Pest # 2

ARI Job No(s)_____

Batch set up by:_____

Bottle #	Extraction Requirements	Verify Client ID	Volume Extracted	pH 5-9 Y/N	DryVap Or KD	Turbo Vap 1 2 3	Final Effective Volume	Volume to Lab	Comments
	MBW	Date	500mL				1mL	1mL	
	SBW		↓				↓	↓	
	SBW Dup.		↓				↓	↓	
Analyst/Date:									

Standard	Standard ID	Volume	Expiration Date	Analyst	Witness
Surrogate	**H**	**100µL**			
Spike	**26**	**100µL**			
Extraction Time:					

SPECIAL INSTRUCTIONS: 1. Verify pH is 5-9 2. Adjust pH (if necessary=Analyst Notes) 3. Add Surr/Spk.

4. Extract 3X with 30mL DCM. 5. DryVap or KD (No drying column) at 80°. 6. TurboVap if KD. 8. Vial in DCM.

A. Archive Y / N

Analytical Resources, Incorporated
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Organic Extractions Benchsheet

PCB – TCLP
Separatory Funnel (3510C) (SOP # 3311S)

TCLP (10ppb)

Preparation Test PCB # 4
ARI Job No(s)_____

Batch set up by: _____

Bottle #	ARI Sample I.D.	Verify Client ID	Volume Extracted	PH 5-9 Y / N	KD **Exchange To Hexane (X 2)**	Turbo Vap 1 2 3 **Hex X**	**(opt)** Acid Clean Y / N	**(opt)** Silica Gel Clean **(1:10)** Y / N	Turbo Vap 1 2 3	Final Effective Volume	Volume to Lab	Comments
	MBW	Date	100mL				10mL	1mL		10mL	1mL	**(TCLPBlanks provided by Metals Prep).**
	SBW		↓				↓	↓		↓	↓	
	SBW Dup.		↓				↓	↓		↓	↓	
Analyst/Date:												

Standard	Standard ID	Volume	Expiration Date	Analyst	Witness
Surrogate	**N**	**200µL**			
Spike	**1**	**250µL**			
Extraction Time:					

SPECIAL INSTRUCTIONS: 1. Verify pH 2. Adjust pH (if necessary=Analyst Notes). 3. Add Surr/Spike.

4. Extract 3X with 30mL DCM. 5. KD (NO Drying Column) at 80°. 6. Exchange (2 X with 20mL) Hexane at 100°.

7. TurboVap to 4mL=10mL Hexane Exchange 8. TurboVap to 10mL. 9. Clean-ups? 10. TurboVap if Silica Cleaned .

11. Vial in Hexane.

A. Archive Y / N

Analytical Resources, Incorporated
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Organic Extractions Benchsheet

PCB – Water
Separatory Funnel (3510C) (SOP # 3311S)
Preparation Test PCB # 3

Low Level (0.01ppb)

ARI Job No(s)_____ Batch set up by: _____

Bottle #	ARI Sample I.D.	Verify Client ID	Volume Extracted	PH 5-9 Y / N	KD	Turbo Vap 1 2 3	(REQ) Acid Clean (1:1) Y	(REQ) Sulfur Clean 1 2 3 (1:1) Y	(opt) Silica Gel Clean (1:1) Y / N	Turbo Vap 1 2 3	Final Effective Volume	Volume to Lab	Comments
	MBW	Date	1000mL				4mL	4mL	1mL		0.5mL	0.5mL	
	SBW		↓				↓	↓	↓		↓	↓	
	SBW Dup.		↓				↓	↓	↓		↓	↓	
Analyst/Date:													

Standard	Standard ID	Volume	Expiration Date	Analyst	Witness
Surrogate	**D**	**100µL**			
Spike	**13**	**25µL**			
Extraction Time:					

SPECIAL INSTRUCTIONS: 1. Rinse all glassware with Low Level Hexane. 2. Verify pH.

3. Adjust pH (if necessary=Analyst Notes). 4. Add Surr/Spike. 5. Extract 3X with 60mL Low Level Hexane.

6. KD (NO Drying Column) at 100°. 7. TurboVap. 8. Clean-ups=Transfer Rinse. 9. TurboVap.

10. Vial with Low Level Hexane. A. Archive Y / N

Analytical Resources, Incorporated
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Organic Extractions Benchsheet

PCB – Water
Separatory Funnel (3510C) (SOP # 3311S)

MTCA (0.1ppb)

Preparation Test PCB # 2
ARI Job No(s)_____

Batch set up by_____

Bottle #	ARI Sample I.D.	Verify Client ID	Volume Extracted	PH 5-9 Y / N	KD **Exchange to Hexane (X 2)**	Turbo Vap 1 2 3 **Hex X**	**(opt)** Acid Clean **(1:1)** Y / N	**(REQ)** Sulfur Clean **(1:1)** 1 2 3 Y / N	**(opt)** Silica Gel Clean **(1:1)** Y / N	Turbo Vap 1 2 3	Final Effective Volume	Volume to Lab	Comments
	MBW	Date	500mL				4mL	4mL	1mL		1mL	1mL	
	SBW		↓				↓	↓	↓		↓	↓	
	SBW Dup.		↓				↓	↓	↓		↓	↓	
Analyst/Date:													

Standard	Standard ID	Volume	Expiration Date	Analyst	Witness
Surrogate	**D**	**200µL**			
Spike	**1**	**25µL**			
Extraction Time:					

SPECIAL INSTRUCTIONS: 1. Verify pH 2. Adjust pH (if necessary=Analyst Notes). 3. Add Surr/Spike.

4. Extract 3X with 30mL Low Level DCM. 5. KD (NO Drying Column) at 80°.

6. Exchange (2 X with 20mL) Low Level Hexane at 100°. 7. TurboVap to 4mL=10mL Low Level Hexane Exchange.

8. TurboVap. 9. Clean-ups?=Transfer Rinse. 10. TurboVap. 11. Vial with Low Level Hexane.
A. Archive Y / N

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Organic Extractions Benchsheet

PCB – Water
Separatory Funnel (3510C) (SOP # 3311S)

Preparation Test PCB # 1
ARI Job No(s)_____

In-House (1ppb)
Batch set up by:_____

Bottle #	ARI Sample I.D.	Verify Client ID	Volume Extracted	PH 5-9 Y / N	KD **Exchange To Hexane (X 2)**	Turbo Vap 1 2 3 **Hex X**	**(REQ)** Acid Clean Y / N	**(opt)** Sulfur Clean 1 2 3 Y / N	**(opt)** Silica Gel Clean (1:5) Y / N	Turbo Vap 1 2 3	Final Effective Volume	Volume to Lab	Comments
	MBW	Date	500mL				5mL	5mL	1mL		5mL	1mL	
	SBW		↓				↓	↓	↓		↓	↓	
	SBW Dup.		↓				↓	↓	↓		↓	↓	
Analyst/Date:													

Standard	Standard ID	Volume	Expiration Date	Analyst	Witness
Surrogate	**N**	**100µL**			
Spike	**1**	**125µL**			
Extraction Time:					

SPECIAL INSTRUCTIONS: 1. Verify pH 2. Adjust pH (if necessary=Analyst Notes). 3. Add Surr/Spike.

4. Extract 3X with 30mL DCM. 5. KD (NO Drying Column) at 80°. 6. <u>Exchange (2 X with 20mL) Hexane at 100°</u>.

7. <u>TurboVap to 4mL=10mL Hexane Exchange</u>. 8. TurboVap 9. Clean-ups? 10. TurboVap (if Silica Clean).

11. Vial with Hexane. **A. Archive Y / N**

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Organic Extractions Benchsheet

Pest – TCLP
Separatory Funnel (3510C) (SOP # 3311S)

TCLP (0.50-1.0ppb)

Preparation Test Pest # 2

ARI Job No(s)_____

Batch set up by:_____

Bottle #	ARI Sample I.D.	Verify Client ID	Volume Extracted	PH 5-9 Y / N	KD **Hexane Exchange (X 2)**	Turbo Vap 1 2 3 **(Hex X)**	**(REQ)** Silica Gel Clean **(1:10)** Y / N	Turbo Vap 1 2 3	Final Effective Volume	Volume to Lab	Comment
	MBW	Date	100mL				1mL		10mL	1mL	**(Blanks provided by Metals Prep)**
	SBW		↓				↓		↓	↓	
Analyst/Date:											

Standard	Standard ID	Volume	Expiration Date	Analyst	Witness
Surrogate	**N**	**200µL**			
Spike	**3**	**100µL**			
Extraction Time:					

SPECIAL INSTRUCTIONS: 1. Verify pH 2. Adjust pH (if necessary=Analyst Notes). 3. Add Surr/Spike.

4. Extract 3X with 30mL DCM. 5. KD (NO Drying Column) at 80°. 6. Exchange (2 X with 20mL) Hexane at 100°.

7. TurboVap to 4mL=10mL Hexane Exchange. 8. TurboVap. 9. SPE Clean-up Required. 10. TurboVap.

11. Vial with Hexane. **A. Archive Y / N**

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Organic Extractions Benchsheet

Pest – Water
Separatory Funnel (3510C) (SOP # 3311S)

MTCA (0.005-0.01ppb)

Preparation Test Pest # 3

ARI Job No(s)_____

Batch set up by:_____

Bottle #	ARI Sample I.D.	Verify Client ID	Volume Extracted	PH 5-9 Y / N	KD Hex X (2)	Turbo Vap 1 2 3 Hex X	(REQ) Sulfur Clean 3.5mL+0.5mL Ethyl Acetate (1:1) Transfer Rinse 1 2 3	(REQ) Silica Gel Clean (1:1) Y	Turbo Vap 1 2 3	Final Effective Volume	Volume to Lab	Comment
	MBW	Date	1000mL				4mL	1mL		1mL	1mL	
	SBW		↓				↓	↓		↓	↓	
	SBW Dup.		↓				↓	↓		↓	↓	
Analyst/Date:												

Standard	Standard ID	Volume	Expiration Date	Analyst	Witness
Diluted Surrogate	**D**	**200µL**			
Diluted Spike	**4**	**100µL**			
Extraction Time:					

SPECIAL INSTRUCTIONS: 1. Rinse all glassware with Low Level DCM. 2. Verify pH

3. Adjust pH (if necessary=Analyst Notes). 4. Extract 3X with 60mL Low Level DCM.

5. KD (NO Drying Column) at 80°. 6. Exchange (2 X with 20mL) Low Level Hexane at 100°. 7. TurboVap to 4mL=10mL

Low Level Hexane Exchange. 8. TurboVap. 9. Clean-ups Required=(1:1)Transfer Rinse. 10. TurboVap.

11. Vial in Low Level Hexane. A. Archive Y / N

Analytical Resources, Incorporated
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Organic Extractions Benchsheet

Pest – Water
Separatory Funnel (3510C) (SOP # 3311S)

In-House (0.05-0.1ppb)

Preparation Test Pest # 1

ARI Job No(s)_____

Batch set up by:_____

Bottle #	ARI Sample I.D.	Verify Client ID	Volume Extracted	PH 5-9 Y / N	KD **Hex X (2)**	Turbo Vap 1 2 3 **Hex X**	**(Opt)** Sulfur Clean **4.5mL+0.5mL** Ethyl Acetate 1 2 3 Y / N	**(Opt)** Silica Gel Clean **(1:5)** Y / N	Turbo Vap 1 2 3	Final Effective Volume	Volume to Lab	Comment
	MBW	Date	500mL				5mL	1mL		5mL	1mL	
	SBW		↓				↓	↓		↓	↓	
	SBW Dup.		↓				↓	↓		↓	↓	
Analyst/Date:												

Standard	Standard ID	Volume	Expiration Date	Analyst	Witness
Surrogate	**N**	**100µL**			
Spike	**3**	**50µL**			
Extraction Time:					

SPECIAL INSTRUCTIONS: 1. Verify pH 2. Adjust pH (if necessary=Analyst Notes). 3. Add Surr/Spike.

4. Extract 3X with 30mL DCM. 5. KD (NO Drying Column) at 80°. 6. <u>Exchange (2 X with 20mL) Hexane at 100°.</u>

7. <u>TurboVap to 4mL=10mL Hexane Exchange.</u> 8. TurboVap. 9. Clean-ups? 10. TurboVap (if Silica Clean).

11. Vial with Hexane. A. Archive Y / N

Analytical Resources, Incorporated
Analytical Chemists and Consultants

Organic Extractions Benchsheet

SIM PNA/Phenol-Water
Liquid/Liquid (3520C) (SOP # 3311S)

Preparation Test SIM PNA/Phenol # 7
ARI Job No(s)_____

In-House (0.1ppb/0.5ppb)
Batch set up by:_____

Bottle #	Extraction Requirements	Verify Client ID	Volume Extracted	Disassemble Liq/Liq	KD or DryVap	Turbo Vap 1 2 3	Final Effective Volume	Volume to Lab	Derivitize	Comments
	MBW	Date	500mL				0.5mL	0.5mL		
	SBW		↓				↓	↓		
	SBW Dup.		↓				↓	↓		
Analyst/Date:										

Standard	Standard ID	Volume	Expiration Date	Analyst	Witness
Surrogate	**B**	**100µL**			
Spike	**15**	**100µL**			
Extraction Time:		**Liq/Liq Start:**		**Liq/Liq Stop:**	

SPECIAL INSTRUCTIONS: 1. Use Blue Labels 2. Add ~200mL DCM to Liq/Liq. 3. Add 500mL sample+500mL DI H2O 4. Add surr/spik.

5. Adjust Acid (pH<2) using 1:1 Sulfuric Acid. (1/4 pipet for blanks & 1/2 pipet for samples) Stir to mix. Verify pH! Let sit 10 minutes. Verify pH again.

6. Extract minimum 8 hrs. 7. DryVap to 0.5mL or KD (no drying column) to ~8mL at 80°. 8. (If KD)=TurboVap to 0.5mL

9. Vial in DCM. 10. GC to Derivitize A. Archive Y / N

Appendix 20.5

Forms

Analytical Resources, Incorporated
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Organic Extractions Laboratory Analyst Notes

ARI Job No.: _____ Client ID: _____

Parameter: Client Project:

Note problems, concerns, corrective actions	Analyst/Date
Screens: Soil/Sediment/Solid/Other:	
☐ **No Anomalies (standard soil/sediment)**	
☐ **Wet sediment/sludge=**	
☐ **Standing Water Decanted=**	
☐ **Standing Water Homogenized (Shared samples)=**	
☐ **Clay (Difficult to homogenize/Mixed with Kitchen Aid)=**	
☐ **Rocks/Organics=**	
☐ **Oily, obvious fuel/sulfur odors=**	
☐ **Other (Details)=**	
Aqueous:	
☐ **No Anomalies**	
☐ **Turbid/Color=**	
☐ **Particulates=**	
☐ **Emulsions=**	
☐ **Other (Details)=**	
☐ **Other Notes/Comments=**	

Standard Operating Procedure

Analysis of Butyltin Species
Using GC-MS
Selective Ion Monitoring (SIM)

SOP 802S
Revision 002

Revision Date: 2/2/10
Effective Date: 2/2/10

Prepared By:

_____

Approvals:

_____
Laboratory / Section Manager

_____
Quality Assurance

Analytical Resources, Incorporated
Analytical Chemists and Consultants

Annual Review

SOP Number:	802S
Title:	Analysis of Butyltin Species Using GC-MS Selective Ion Monitoring (SIM)

The ARI employee named below certifies that this SOP is accurate, complete and requires no revisions

Name	Reviewer's Signature	Date

GC-MS Analysis – Selective Ion Monitoring (SIM)

1. Scope and Application

1.1. This SOP describes the use of gas chromatography/mass spectrometry (GC/MS) using selective ion monitoring (SIM) to determine the concentration of selected butyltin species (BTS) in extracts prepared from various types of solid waste matrices, soils, tissues, sediments and waters. Some text has been taken directly from the analytical methods referenced in Section 19

1.2. Procedures for the several suites of analytes are described, including:

1.2.1. Butyltin compounds in water and sediment

1.2.2. Butyltin compounds in porewater

1.3. Instrument operating conditions vary, depending on the nature of the sample extract and the targeted reporting limit.

1.4. Routine reporting limits (RL) for each analysis are listed in the RL Table below. RLs will be proportionately higher for sample extracts that require dilution to avoid saturation of the detector and/or display chromatographic interference.

1.5. This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatograph/mass spectrometers and skilled in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method.

RL Table			
Analyte	**Water (µg/L) 100mL/0.5mL**	**Pore Water (µg/L) 100mL/0.5mL**	**Soil (µg/Kg) 5g/0.5mL**
Tributyltin Ion	0.2	0.008	3.9
Dibutyltin Ion	0.3	0.012	5.8
Butyltin Ion	0.2	0.008	4.1

2. Summary of Procedure

2.1. A measured amount of sample, normally 100 mL for water and porewater samples and 5 grams for solid samples) is extracted by a matrix appropriate technique. Extraction techniques are separatory funnel extraction for aqueous samples and sonication or microwave for solid samples. Section 1.1 lists other sample matrices and extraction methods amenable to analysis using this method. The extracted sample may be subjected to an appropriate cleanup and concentrated as described in ARI's sample preparation SOPs. Routine final volumes are 0.5 mL for aqueous and solid samples and 1 mL for tissue samples.

2.2. Extracts are derivatized with a Grignard Reagent to create hexylated derivatives of the tin species, which are analyzed as such on the GC-MS. Hexylation is used to increase instrument response and to lower the activity of the tin species.

2.3. Sample extracts are analyzed using GC-MS with the MS in the SIM mode. Prior to analysis the GC/MS system is tuned and calibrated as described in Section 10. Target analytes are identified by comparing their chromatographic retention times and mass spectra with known standards. Detected analytes are quantified by comparing their mass spectral response with that of standards of known concentrations relative to an internal standard.

2.4. Final concentrations of tin analytes are reported as tin ions; the concentrations generated by the GC-MS are converted from the hexylated species using the Table in Appendix 20.4.

3. Definitions

3.1. Analytical Batch- a 12 hour time period initiated by the injection time of the tune standard, not limited by the number of samples injected.

3.2. DDT Breakdown Factor- the ratio of the sum of the areas of the DDT breakdown products (DDD and DDE) divided by the summated areas of DDT and the DDT breakdown products. The factor is used to measure injector inertness.

3.3. DFTTP (decafluorotriphenylphosphine)- this is used to verify the tune.

3.4. Extracted Ion Current Profile (EICP) - plot of the abundance of a specific ion as a function of time.

3.5. Initial Calibration Verification Standard (ICVS)- are not available for this analysis.

3.6. Laboratory Control Sample is a volume of clean reference matrix (reagent water for water samples, or sodium sulfate for soil/sediment samples) that is carried through the entire analytical procedure. The LCS is spiked with all surrogates and target analytes required by the method.

3.7. Laboratory Information Management System (LIMS)

3.8. Limit of detection (LOD)

3.9. Method Blank: a volume of clean reference matrix (reagent water for water samples, or sodium sulfate for soil/sediment samples) that is carried through the entire analytical procedure. The volume or weight of the reference matrix must be approximately equal to the volume or weight of samples associated with the blank. The purpose of a method blank is to determine the levels of contamination associated with the processing and analysis of samples.

3.10. Matrix Spike Duplicates (MSD) - are replicate aliquots of the same sample taken through the entire analytical procedure Matrix Spike Analysis

3.11. Peak Tailing Factor- A measure of the relative activity / inertness of the chromatographic system as defined in Appendix 20.7.

3.12. Reagent Water- typically deionized water; however, for pore water extractions the reagent water is a combination of sea salt and deionized water used to mimic estuary and marine conditions.

3.13. Reconstructed Ion Current (RIC) - plot of the total instrument response versus time.

3.14. Relative Retention Time (RRT) - the elution time of an analyte relative to the elution time of its associated internal standard.

3.15. Sample Delivery Group- an extraction batch

3.16. Scan Descriptor- the summary of masses and scan time per mass the mass spectrometer uses through the chromatographic run.

3.17. Selected Ion Monitoring (SIM) - a mode of data acquisition wherein the mass spectrometer is programmed to scan for a limited number of specific masses, increasing the analytical sensitivity and specificity.

3.18. Standard Reference Material (SRM)- a standardized environmental reference material.

3.19. Surrogate Standards- are compounds chemically similar to target analytes that are added to a sample prior to the extraction process. The recovery of surrogates is used to evaluate extraction efficiency. Surrogates are halogenated, isotope labeled or chemically similar compounds not expected to be detected in environmental samples that are added to every sample analyzed.

4. Interferences

4.1. Extraction Interferences: Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation and/or cleanup of the samples and take corrective action to eliminate the problem. Most commonly this will involve contamination with the phthalate esters.

4.2. Method interferences are reduced by washing all glassware with hot soapy water and then rinsing with warm tap water, acetone, and methylene chloride followed by baking the glassware at 450 degrees centigrade overnight.

4.3. High purity reagents must be used to minimize interference problems.

4.4. Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, or the detector shows saturation due to analytes present in a sample, the following samples should be scrutinized for cross contamination.

4.5. Matrix interferences may be removed by the use of sample clean-ups depending on the list of target analytes, these may include (but are not limited to) the use of solid phase extraction clean-up, absorption chromatography or gel permeation chromatography. See the appropriate extractions SOPs for clean-up applicability to matrices or methods.

4.6. If detector saturation occurs, the analyst should follow the sample with instrument blanks until the system is decontaminated.

5. Safety

5.1. The toxicity and carcinogenicity of each reagent used in this method is not precisely defined. However, all compounds and solutions should be treated as health hazards, and exposure of these chemicals to skin and clothing should be minimized to the lowest possible level by whatever means available.

5.2. Contact with all chemicals should be minimized by the use of nitrile gloves, safety glasses, and laboratory coats.

5.3. Standard solutions should be handled in the fume hoods to avoid exposure to fumes.

5.4. ARI maintains a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDS) is available to all personnel involved in the chemical analysis. Consult with MSDS sheets for all chemicals handled. MSDS information is also available at www.msdshazcom.com

6. Equipment and Supplies

6.1. Gas chromatograph/mass spectrometer system

6.1.1. Gas chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection and all required accessories, including syringes, analytical columns, autosampler, and gases. The capillary column should be directly coupled to the source of the mass spectrometer.

6.1.2. Fused-silica capillary column - 30 m x 0.32 mm ID (or 0.25 mm ID) 0.25 -0.5 µm film thickness coated with cross bonded 5% diphenyl/95% dimethyl polysiloxane stationary phase (Phenomonex ZB-5msi or equivalent).

6.1.3. Mass spectrometer - Capable of scanning from 35 to 500 amu in 1 second or less The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets all of the criteria in Appendix 20.3 when 1 µL of the GC/MS tuning standard is injected onto the GC (5-25 ng of DFTPP).

6.1.4. GC/MS interface – ARI uses a capillary column directly fitted into the mass spectrometer source.

6.2. Data system - A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on magnetic media of all mass spectra obtained throughout the duration of the chromatographic program.

6.2.1. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits.

6.2.2. A mass spectral library utilizing a specific BTS scan descriptor must be created to provide library search information for automated compound identification and data reduction. The mass spectral library should be created using the midpoint of the initial calibration. The identical BTS scan descriptor must be employed for the initial calibration and all subsequent analyses in order for such comparisons to remain valid. Otherwise, a new mass spectral library must be created.

6.2.3. The data will be securely stored for at least seven years from date of acquisition.

6.3. Syringe - 10 µL, 25 µL, 50 µL, 100 µL, 500 µL, & 1000 µL

6.4. Volumetric flasks, Class A - 10 mL to 1000 mL.

6.5. Amber bottles - glass with Teflon-lined screw caps.

6.6. Balance - Analytical, 0.0001 g

6.7. Autosampler vials- amber 2 ml autosampler vials with crimp caps.

7. Reagents and Standards

7.1. Organic Solvents

7.1.1. High purity, reagent grade solvents and chemicals are used in all analyses.

7.1.2. The laboratory stocks acetone, hexane, methylene chloride, methanol and other appropriate solvents for preparing standards.

7.2. Stock Standards (1000 - 10,000 µg/L) may be purchased as certified solutions traceable to NBS standards or prepared by ARI staff from neat compounds.

7.2.1. Stock standard solutions are stored at 0 to 6℃ in amber glass containers with Teflon lined screw-caps. The solutions must be protected from light.

7.2.2. Stock standard solutions must be checked periodically and immediately prior to use for signs of degradation, evaporation, or precipitation.

7.2.3. Stock standard solutions must be replaced after 1 year or sooner if necessary.

7.3. Internal Standard Stock Solutions are prepared by dissolving 0.04 g of each compound with a small volume of methylene chloride or other appropriate solvent to a 4 mL final volume. The resulting solution will contain each standard at a concentration of 10,000 ng/µL. Most of the compounds are also soluble in small volumes of methanol, acetone, or hexane.

7.4. Surrogate Stock and Matrix Spike Standards are prepared stocks by dissolving 0.04g of each compound with methylene chloride to a 4 mL final volume. The resulting solutions will contain the surrogate standards that are 10,000 µg/mL.

7.5. Spiking solutions that include all target analytes are prepared from stock solutions.

7.6. Working standards are prepared from stock standards.

7.6.1. Working standards expire either on the date of expiration of the stock solutions they are prepared from, or on the expiration date provided by manufacturer's. They must be replaced upon expiration.

7.6.2. Working standard solutions are stored at 0 to 6℃ in amber glass containers with Teflon lined screw-caps. The solutions must be protected from light.

7.6.3. Working standards must be check frequently for signs of concentration or degradation.

7.7. Internal Standard Working Solutions are prepared from stock solutions such that the final concentration of the internal standards are 2 and 0.2 µg/mL for water and solids and 0.2 and 0.02 µg/mL for pore water.

7.8. A Surrogate Standard calibration standard is prepared by diluting the surrogate stock in hexane such that the concentration of the surrogates are 50 µg/mL for Tripropyltin and 100 µg/mL for Tripentyltin. This solution is used to calibrate the instrument to determine the concentration of surrogate standards in the final extract.

7.9. A Surrogate Spiking Standard is prepared by diluting the surrogate stock in methylene chloride-1% Tropolone such that the final concentration of the surrogates is 0.5 µg/mL (0.025 for pore water). This solution is prepared in methylene chloride/tropolone and used to spike all extracted samples and QC with surrogates. The GC-MS analyst prepares the spiking solution for use by the Organic Extractions Laboratory.

7.10. Target analyte spiking standards are used to fortify laboratory control and matrix spike samples (LCS, LCSD, MS, and MSD).

7.10.1. Spiking standards that contain all target analytes are prepared by diluting the matrix spiking stock solutions in methylene chloride-1% Tropolone such that the final concentration of each compound is 0.5 µg/mL. (0.025 and 0.05 for pore water analyses).

7.11. Working solutions used to prepare calibration curves are prepared from stock standards. Working calibration solutions are prepared by diluting these standards in hexane such that the concentration of each target analyte is 50 µg/mL (0.25 ug/mL for pore water).

7.12. A GC/MS tuning standard containing 250 µg/mL each of DFTPP, p,p'-DDT, Pentachlorophenol, and Benzidine is purchased as a certified solution. This solution is diluted to a 25-5 µg/mL standard used to tune the instrument.

8. Sample Collection, Preservation, Shipment and Storage

8.1. Extracts are delivered to Refrigerator 17 in the instrument laboratory by extractions technicians. The extracts are signed into the GC laboratory Extract Refrigerator Log when delivered.

8.2. Analysts in the instrument lab assume custody of the sample extracts and then move them into Refrigerator 15.

8.3. Extracts must be stored at 0 to 6℃ protected from light.

8.4. Extracts must be analyzed within 40 days of the first day of extraction.

8.5. Water samples must be extracted within 7 days of sampling date and solids within 14 days of sampling date.

9. Quality Control

9.1. Procedures include negative and positive controls, verification of detection limits, calibration and method selectivity.

9.2. Negative Control

9.2.1. Method blanks (MB) are used to assess the preparation batch for possible contamination resulting from the sample preparation process. Extraction and analysis are performed as follows:

9.2.2. One MB is analyzed with every preparation batch of twenty or fewer samples.

9.2.3. The MB is analyzed once for each preparation batch unless MB data for each instrument is mandated by project-specific requirements and a full data package is required.

9.2.4. When post preparation contamination is suspected, the method blank will be reanalyzed in a manner that will verify the source of contamination.

9.2.5. An aqueous MB is prepared using 0.5 or 1.0 Liters of Type 1 water from ARI's centralized water purification system, pore water method blanks use sea salt reagent water. Method blanks for solid samples (soil and sediment), are prepared using 5 g purified sodium sulfate.

9.2.6. Method blanks must be processed in exactly the same manner as other samples in a preparation batch.

9.3. Positive Controls

9.3.1. LCS analyses aid in the evaluation of the total analytical process including all preparation and analytical procedures.

9.3.1.1. Recovery of compounds added to the LCS are compared to established statistical criteria calculated from historic analytical data.

9.3.1.2. An LCS sample is analyzed with every batch of twenty of fewer samples.

9.3.1.3. When there is not sufficient sample to analyze an MS and/or MSD, an LCSD may be analyzed, at the client's discretion, and compared to the LCS to evaluate method precision.

9.3.2. Matrix Spike (MS) and Matrix Spike Duplicate (MSD) analyses indicate the effect of the sample matrix on the precision and accuracy of the results using the selected method. MS and MSD results should not be used to reject batch data.

9.3.2.1. Each preparatory batch of twenty or less samples of a similar sample matrix should include both MS and MSD analyses

9.3.2.2. MS samples are spiked with a all the target analytes.

9.3.2.3. MS and MSD results are evaluated against statistical control limits calculated from ARI's historical data. Project or client specific control limits may substitute for ARI's calculated values. Failure to meet these specific control limits will require corrective action even if in house limits are achieved

9.3.2.4. QA/QC samples (rinsate blank, trip blank, SRM, etc.) submitted by a client must not be used to prepare MS samples.

9.3.2.5. When ARI's client designates a specific sample for MS/MSD analysis, that sample must be used. When there is insufficient sample to perform MS/MSD analyses ARI will immediately inform the client. The client will approve one of the following options:

9.3.2.5.1. MS/MSD analyses are deleted from the sample batch.

9.3.2.5.2. An alternate sample is designated for MS/MSD analyses

9.3.2.5.3. LCS/LCSD analyses are used to assess method precision.

9.3.2.6. The client's rationale and decision must be documented on the "Analytical Notes" form and reported in the case narrative.

9.3.2.7. Dilution of MS/MSD extracts to get either spiked compounds or native analytes on scale is not required.

9.3.3. Duplicates may be analyzed as required by a specific client request and/or QA protocol. Duplicate analyses are used to assess the precision of analytical results in a given sample matrix. The results of an Duplicate analysis are normally expressed as an RPD (relative percent difference) value.

9.3.4. Surrogates Standards (SS) are added to all samples prior to extraction to provide a measure of method efficiency (recovery) for each sample matrix.

9.3.4.1. SS are chosen to represent the various target analytes.

9.3.5. The reporting limits for analytes in this procedure are defined by the lowest concentration calibration standard. MDL studies, as described in ARI SOP 1018S, are conducted to

determine an LOD(MDL). An analyte concentration between the one half the RL and the LOD (MDL) may be reported but must J - flagged to indicate the higher analytical uncertainty associated with these values.

9.3.6. Method selectivity is evaluated using the following checks:

9.3.6.1. Prior to any analysis each mass spectrometer must be hardware-tuned to meet the criteria in Appendix 20.3.

9.3.6.1.1. Inject 5-25 ng/uL solution of DFTPP, p,p'-DDT, benzidine and pentachlorophenol.

9.3.6.1.1.1. Evaluate the ion abundance of DFTPP using one of the following scan scenarios:

9.3.6.1.1.1.1. use the mean of the apex and the preceding and following scans

9.3.6.1.1.1.2. use the average across the entire peak.

9.3.6.1.1.2. Background correction should be compliant with method specifications and employed only for the purpose of correcting for instrument background ions. Do not subtract part of the DFTPP peak.

9.3.6.1.1.3. Degradation of DDT to DDE and DDD should not exceed 20%. DDT percent breakdown is calculated by dividing the sum of the DDD and DDE areas by the sum of the areas of DDT, DDE and DDD and then multiplying this result by 100.

9.3.6.1.1.4. Benzidine and pentachlorophenol should be present at their normal responses, with no visible peak tailing. Peak tailing factors are calculated using the formula in Appendix 20.7. Tailing factors should be <2.0 for benzidine and pentachlorophenol. See Section 16.2 for procedures on how to bring the instrument into compliance for tuning, DDT breakdown, or peak tailing factors. Non-compliances must be documented on the "Analyst's Notes" form and reported in the case narrative.

9.3.6.1.2. Section 16 includes procedures to bring the instrument into compliance for tuning, DDT breakdown, or peak tailing factors.

9.3.6.2. The relative retention time (RRT) of each target analyte must vary be less than .06 RRT units in the calibration standards. Late-eluting target analytes usually have much better agreement. The RRT is calculated by dividing the retention time of the target analyte/surrogate by the retention time of its assigned internal standard.

9.3.6.3. The internal standards selected should permit most of the components of interest in a chromatogram to have retention times of 0.80-1.20 relative to one of the internal

standards. Use the base peak ion from the specific internal standard as the primary ion for quantitation (see Appendix 20.2).

9.3.6.4. Internal Standard Response (EICP) area and retention time data are to be evaluated during and/or immediately after the analysis. The response for each of the internal standards must be within the inclusive range of -50.0 percent and +100.0 percent of the response of the internal standards in the midpoint of the most recent initial calibration. The retention time shift for each of the internal standards must be within ± 0.50 minutes (30 seconds) between the sample and the most recent continuing calibration analysis.

9.3.6.5. Statistical Control- Internal quality control limits for analyte spike and surrogate recoveries and relative percent difference for matrix spike and matrix spike duplicates are statistically generated on a periodic basis. These quality control limits are provided to bench chemists, managers, and QA staff as tools for assessing data quality in real-time at the point of data generation. Practical considerations relating to their dynamic nature require their presentation in a document separate from this SOP. All analysts using this SOP must use it in conjunction with Control Limit documentation in order to assess data quality and any possible need for corrective actions. Current control limits may be found in the ARI LQAP.

9.3.6.6. Initial Demonstration of Proficiency- Each analyst must demonstrate initial proficiency with each sample preparation and determinative method combination performed, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat these procedures whenever new staff is trained or significant changes in instrumentation or procedure are made. See Methods 8000 and 3500 for information on how to accomplish this demonstration.

10. Calibration and Standardization

10.1. Calibration standards - A minimum of five calibration standards are used. The lowest concentration calibration standard defines the reporting limit and the other concentrations should correspond to the range of concentrations found in real samples but must not exceed the working range of the GC/MS system. The lowest calibration point of each individual target analyte becomes the reporting limit for that target. All target analytes quantitated below the lowest calibration point for any target analyte must be qualified with a “J” flag to show the quantitation is below the working range of the curve. Calibration standards are prepared from the working calibration solutions at concentration levels listed in Appendix 20.5. The calibration standards are made with a final volume of 0.5 ml in an amber vial with a crimp cap. 10 µl of the internal standard solution is added prior to analysis. Each standard should contain each analyte for detection by

this method (e.g. some or all of the compounds listed in Appendix 20.5 may be included). However, no target analyte may be quantitated without first being calibrated. All standards should be stored at 0 to 6℃ and should be freshly prepare d once a year, or sooner if check standards indicate a problem. The daily calibration standard should be prepared weekly with the expiration date and standard identifications on the label and stored at 0 to 6℃. If the analyst suspects degradation the standard should be replaced.

10.2. If the DFTPP tuning and peak tailing factors meet acceptance criteria, the initial calibration may proceed by injecting the calibration standards prepared as described in Section 10.1 into the analytical column.

10.3. Calibration Requirements

10.3.1. The average RRF should be calculated for each compound. The percent relative standard deviation (% RSD = 100 [SD/average RRF]) should also be calculated for each compound. The % RSD should be less than 20% for each compound, in which case the average response factor will be used for quantitation as it is constant over the calibration range. Target analytes that exceed 20% RSD will utilize an alternative calibration option. If the analyst determines that there is no detector or chromatographic peak saturation and that the chromatographic system is functioning properly, then the analyst will proceed as follows:

10.3.1.1. A linear fit calibration that does not include the origin and generates a coefficient of determination (R2) that is greater than or equal to 0.99 is acceptable. ARI will attempt to force calibration curve through zero. Forcing the curve through zero is not the same as including the origin as a point in the calibration. When the curve is forced through zero, the intercept is set to 0 before the regression is calculated, thereby setting the bias to favor the low end of the calibration range by “pivoting” the function around the origin to find the best fit and resulting in one less degree of freedom.

10.3.1.2. Next the analyst may attempt a quadratic non-linear calibration which may be used with target analytes that have six calibration points only. An additional initial calibration point may be considered for target analytes that require non-linear calibration if only five calibration points are available.

10.3.1.3. Individual targets that are unable to meet the above requirements must be documented in the case narrative. The initial calibration may still be valid if approved on an individual basis by the Laboratory Supervisor and/or the Laboratory Manager.

10.3.1.4. See EPA Method 8000C Section 11 for reference to linear fit and non-linear (quadratic) calibration. Detailed in Section 10.3.1.1

10.4. Using data from the mid-point of the initial calibration, update the reference mass spectrum library used to identify all target analytes.

10.5. Daily GC/MS calibration verification - Performed at the beginning of each 12-hour analytical shift.

10.5.1. Prior to analysis of samples, the GC/MS tuning standard must be analyzed using a full scan of 35-500 amu. A 5-25 ng/µL injection of DFTPP must result in a mass spectrum for DFTPP which meets the specified criteria (Appendix 20.3). These criteria must be demonstrated during each 12 hour shift. The 12 hour shift begins from when the DFTPP was injected and does not include time of elution. The DFTPP check should also measure injector port inertness through the evaluation of DDT breakdown and the calculation of peak tailing factors for pentachlorophenol and benzidine.

10.5.2. Analysis of a continuing calibration verification standard (CCV) at mid-concentration (0.5 µg/mL or 25 ng/µL for pore water), containing each compound of interest, including all required surrogates, must be performed once every 12 hours before sample analysis, using the introduction technique used for the initial calibration. The results from the CCV analysis must meet the acceptance criteria detailed below in Sections 10.5.2.1 through 10.5.2.3.

10.5.2.1. Percent Difference Criteria- After the tune check has been performed; check the validity of the CCV by calculating the %D for each analyte.

$\% \text{ Difference} = \frac{RRF_i - RRF_c}{RRF_i} * (100)$
Where:
RRFi = Average relative response factor from initial calibration.
RRFc = Relative response factor from current verification check standard.

10.5.2.2. The percent difference for each analyte must be less than 20%. If the percent difference criterion is not met corrective action must be taken. See Section 16.3 or procedures for dealing with CCV failure. This criterion must be met before sample analysis begins. All corrective actions must be recorded on an “Analyst Notes” form.

10.5.2.3. The internal standard responses and retention times in the CCV must be evaluated immediately after or during data acquisition. Relative retention times should remain constant. If the retention time for any internal standard changes by more than 0.5 minutes (30 seconds) from the CCV or the response of any internal standard (EICP area) changes by a factor of two (-50% to +100%) from that in the mid-point standard level (0.5 ng/µL) of the most recent initial calibration, then the system should be inspected for

malfunctions and corrections made as required. When corrections are made, corrective action is required and may include reanalysis of samples analyzed while the system was malfunctioning-.

11. Procedure

11.1. Recommended GC/MS operating conditions are as follows (samples must be run using the same instrument conditions as the initial calibration).

Quantitation Mass range:	Selected Ions
Tuning Mass Range	35-500 amu
Scan time:	1 sec/scan
Initial temperature:	100℃, hold for 1 minutes
Temperature program:	100℃ at 25℃ /min to 320℃
Final temperature:	320℃, hold for 0.2 minutes
Injector temperature:	250℃
Transfer line temperature:	300℃
Source temperature:	According to manufacturer's specifications (170-180℃ for 5972 MSD,230℃ for 5973 & 5975)
Injector:	Grob-type, splitless/split
Sample volume:	1-3 µL
Carrier gas:	Helium at 30 cm/sec.

11.2. Sample extracts are analyzed with the GC-MS in the SIM mode.

11.3. Verify that the GC-MS method in use contains the proper scan descriptors for the target analytes. Scan descriptors are provided in Appendices 20.4

11.4. Tune the GC-MS instrument as described in Section 10.

11.5. Calibrate the instrument as described in Section 10.

11.6. Spike each sample extract to be analyzed with internal standard solution using 2 µL of the internal standard solution for every 0.1 mL of extract. (example: spike 10 µL of internal standard stock into a 0.5 ml sample) This results in a concentration of 2.0 and 0.2 ng/µL (0.2 and 0.02 for pore water) of each internal standard

11.7. Prior to sample analysis a CCVS must be analyzed, and this CCV must meet the criteria found in Section 10.5. If time remains in the 12 hour QC period begun with the initial calibration, the midpoint calibration standard from the initial calibration may be used as the CCV provided it meets the requirements found in Section 10.5.

11.8. Using the analyte RRT from the CCV, adjust the beginning of each scan group as necessary.

11.9. Enter all information required for the analytical batch in the instrument logbook.

11.10. Load the auto sampler and enter the sample sequence on the Chemstation data system.

11.11. Begin GC/MS data acquisition

11.12. When the acquisition is complete, export the data files from the GC/MS data system to the Target data system for data reduction.

11.13. Process the data using the appropriate Target method.

11.14. Confirm that the computer has correctly identified all target analytes and properly performed all chromatographic integrations.

11.15. Perform necessary manual integrations following the guidelines in Appendix 20.1.

11.16. The analyst must sign and date all hardcopy data, especially that documenting manual integrations.

11.17. Export the quantitative numbers into ARI's LIMS where final reports will be generated.

11.18. Submit a hardcopy job file to review. The file will contain:

11.18.1. Copies of all tune files

11.18.2. Copies of all CCVs

11.18.3. Raw data for all the samples and associated QC analyses

11.18.4. The LIMS report(s)

11.18.5. An "Analyst's Notes" form that includes all deviations from normal routine procedures and any other noteworthy observations or events concerning sample analysis

11.19. All extracts should be stored in Refrigerator 15 in amber vials with Teflon lined screw caps at 0 to 6℃.

12. Data Analysis and Calculations

12.1. Qualitative Analysis

12.1.1. Target analytes are identified by comparing characteristic ions of the sample mass spectrum with that of a known reference standard. Reference mass spectra are generated by the laboratory using routine calibration data obtained using conditions identical to the sample analyses. Characteristic ions are either the three ions of greatest intensity, or any ions over 30% intensity relative to the base ion. Two criteria must be satisfied to verify identification:

12.1.1.1. The target analyte in the sample must elute within ±0.06 RRT units of the RRT of the compounds in a calibration standard analyzed in the same 12 hour QC period.

12.1.1.1.1. When co-elution components prohibit accurate assignment of an RRT using total ion chromatogram, the RRT may be assigned by using extracted, ion-current profiles for ions unique to the component of interest.

12.1.1.2. The mass spectrum of the target analyte must correspond to that of the standard component.

12.1.1.2.1. Intensities of all characteristic ions must maximize in the same or an adjacent scan. A chromatographic peak selected by a data system based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time meets this criterion.

12.1.1.2.2. All ions present in the standard mass spectra at a relative intensity greater than 10% (the most abundant ion in the spectrum is equal to 100% intensity) must be present in the sample spectrum.

12.1.1.2.3. The relative intensities of ions specified in Section 12.1 in the standard and sample spectra must be within ± 30%. (Example: an ion with an abundance of 50% in the standard spectrum must have a corresponding ion between 20 and 80% in the sample spectrum.) Compounds with spectra not meeting this criterion may be considered acceptable and flagged with an M by an analyst or data (favors false positive results).

12.1.1.3. Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes co-elute (i.e., only one chromatographic peak is apparent), the identification criteria can be met, but each analyte spectrum will contain extraneous ions contributed by the co-eluting compound.

12.2. Quantitative analysis

12.2.1. Confirmed target analytes are quantified based on the integrated abundance from the EICP of the primary characteristic ion.

12.2.2. Quantitation will take place using the internal standard technique. The internal standard used shall be the one nearest the retention time of a given analyte.

12.2.3. When secondary ion quantitation is necessary due to interference a short quantitation report list is generated. This quantitation contains the integrated areas of the affected compounds, based on the secondary ion(s) for that compound, and of the relevant internal standards. Identical reports must be generated for the sample with interference and for the

relevant initial calibration. The report for the initial calibration is used to generate a relative response factor for the affected compound based on its secondary ion. This relative response factor is then used in the calculations for that compound in the affected sample. All quantitations using the primary characteristic ion use the average relative response factor from the initial calibration. The short quantitation report may be hand calculated by the analyst as long as it is signed and dated by the analyst.

12.2.4. Calculate the concentration of each identified analyte in the sample as follows:

Concentration (µg/L) = $\frac{(Ax)(Is)(Vt)}{(Ais)(RRF)(Vo)(Vi)}$
where:
A_X = Area of characteristic ion for compound being measured
Is = Amount of internal standard injected (ng).
Vt = Volume of total extract, taking into account dilutions (i.e. for a 1 to 10 dilution of a 1 ml extract Vt = 10,000 µL)
A_{IS} = Area of characteristic ion for the internal standard
RRF = Relative response factor for compound being measured
Vo = Volume of water extracted (ml)
Vi = Volume of extract injected (µL)

12.2.5. Sediment/Soil/Sludge (on a dry weight basis) and Waste (normally on a wet weight basis)

Concentration (µg/kg) =	$\frac{(Ax)(Is)(Vt)}{(Ais)(RRF)(Vi)(Ws)(D)}$
where:	
A_X = Area of characteristic ion for compound being measured	
Is = Amount of internal standard injected (ng).	
Vt = Volume of total extract, taking into account dilutions (i.e. for a 1 to 10 dilution of a 1 ml extract Vt = 10,000 µL)	
A_{IS} = Area of characteristic ion for the internal standard	
RRF = Relative response factor for compound being measured	
Vi = Volume of extract injected (µL)	
Ws = Weight of sample extracted or diluted in grams	
D = % Dry weight of sample = 1.0 on an as received basis	

13. Method Performance

13.1. The QA department measures method performance using a combination of annual method detection limit (MDL) studies, performance evaluation samples, and the monitoring of surrogate and spike recoveries.

13.1.1. Detection limits- detection limits for all analytes quantitated using this SOP are set using the low point of the initial calibration curve and validated via method detection limit studies.

13.1.2. MDL studies are performed each year for each analyte by each preparatory and analytical method (for example, analytes quantitated for in water samples will have MDL studies performed for both the liquid/liquid extraction and sep funnel extraction, and the associated mass spectrometry detection methods.)

13.1.3. MDL and reporting limit (RL) values may be found for each analyte in the ARI LQAP

13.2. Laboratory precision and bias measurements are performed by monitoring surrogate and spike recoveries in samples and quality control samples.

13.3. Method performance must be re-evaluated every time there is a change in instrument type, personnel or method. Method performance will be demonstrated using the Demonstration of Capability (DOC) procedure described in ARI SOP 1017S. A certification statement and all raw data from the DOC will be forwarded to QA for approval and archived. Each DOC must be documented in the instrument run logbook.

13.4. Control limits are calculated by monitoring these recoveries. These control limits are disseminated to the bench chemists and LIMS administrator for use in monitoring method performance in real time. As these limits are updated regularly, their dynamic nature prevents their inclusion in this SOP. However, they may be found in the ARI LQAP.

14. Pollution Prevention

14.1. All syringe rinsing must be performed over charcoal to minimize the exposure of the environment to solvent or extract. The charcoal containers must be covered when not in use to prevent fugitive vapors.

14.2. All autosampler vials stored in the refrigerators when not being used.

14.3. All GC split vents will be connected to a charcoal exhaust filter or to an exhaust vent.

14.4. All MS vacuum pumps will have a charcoal exhaust filter or to an exhaust vent.

15. Data Assessment and Acceptance Criteria for QC Measure

15.1. Requirements relating to initial and continuing calibration are detailed in Section 10 of this document.

15.2. Method Blanks must be free of target analytes with a concentration greater than 1/2 their reporting limit or corrective action is required.

15.3. Internal Standard EICP areas following the continuing calibration standard must meet the technical acceptance criteria listed in SOP Section 9.3.6.4.

15.4. Surrogate Recoveries for all sample, MB, LCS, MS, MSD, MD, & SRM analyses must have acceptable surrogate recoveries based on ARI's current control charts.

15.4.1. These requirements do not apply to subsequent dilutions of samples where a prior analysis of the diluted sample extract shows acceptable surrogate recovery.

15.4.2. Certain methods or clients may specify project specific surrogate recovery acceptance windows.

15.4.3. When mandated by contract-specific requirements, corrective actions must be performed in response to failure to meet project specific surrogate acceptance criteria, even when in house criteria are met.

15.4.4. Surrogate acceptance criteria are both matrix and concentration level specific (e.g. water vs. pore water). When analyzing matrices or concentration levels for which no acceptance criteria are available, the closest approximation of available acceptance criteria may be provided as estimates for advisory purposes only.

15.5. Laboratory Control Samples (LCS)

15.5.1. The LCS recovery values should fall within the specified recovery acceptance limits. If an LCSD is performed then relative percent difference (RPD) acceptance limits may also apply, if available.

15.5.2. LCS recovery acceptance windows are ideally determined statistically from method and matrix-specific laboratory data updated on a periodic basis. Project or method specific limits may supersede laboratory acceptance criteria.

15.6. Matrix Spike/Matrix Spike Duplicates (MS/MSD)

15.6.1. Matrix Spike/Matrix Spike Duplicate recovery values should fall within the specified recovery acceptance limits. If a MSD is performed then relative percent difference (RPD) acceptance limits may also apply, if available. MS/MSD recoveries should not be used to reject batch data.

15.6.2. MS/MSD recovery and RPD acceptance windows are ideally determined statistically from method and matrix-specific laboratory data updated on a periodic basis. Certain methods or clients may require project specific MS/MSD recovery and RPD acceptance windows.

15.7. Holding Times

15.7.1. Samples must be extracted within holding times (seven days for water samples and fourteen days for solid samples).

15.7.2. Extracts must be analyzed within the extract holding time (forty days from the initial date of extraction.)

15.7.3. In the event that re-extraction due to an out of control event requires that samples be re-extracted after their extraction holding time has elapsed (seven days for water and fourteen days for tissues/solids) the analyst should analyze and report both extraction sets, whenever practical, distinguishing between the initial extraction and re-extraction on all deliverables. This will document that the samples were originally extracted within holding times and may allow for comparisons that will determine whether any of the more active analytes were lost in the interval between extractions.

15.7.4. If any extracts are analyzed after the 40 day extract holding time has elapsed, the analyst shall document this in the analytical notes accompanying the data so that it may be included in the case narrative.

16. Corrective Actions for Out of Control of Unacceptable Data

16.1. Mass Spectrometer Tuning

16.1.1. When the MS does not produce an acceptable mass spectrum with 5-25 µg/mL of DFTPP, re-inject the DFTPP. If the spectrum again fails to meet the criteria found in Appendix 20.3, the MS may need to be re-tuned.

16.1.2. If the re-acquired mass spectrum still fails to meet the criteria found in Appendix 20.3, the MS may need maintenance. The MS should be vented, and maintenance may include: replacing the filaments, cleaning the MS source, cleaning the MS quadrapoles, or replacing the electron multiplier.

16.2. Peak tailing factors and DDT breakdown

16.2.1. If the peak tailing factors or the DDT breakdown exceed the limits found in Section 9.3.6.1.1.4, the chromatographic system may need maintenance. Inspect the system for leaks. Should none be found, perform maintenance on the chromatographic system. This maintenance includes, but is not limited to: replacing the inlet liner and liner packing, clean the inlet liner, cleaning or replacing the inlet seal, cleaning or replacing the inlet body, replacing the split line, cleaning the split arm, clipping a length from the front of the column, or replacing the column. Any maintenance is to be documented in the GC/MS run logbook for that instrument and/or the maintenance logbook.

16.3. Initial Calibration/CCV failure- should RSD or %D failure be evident in either the initial calibration or the CCV, perform the maintenance found in Section 16.1.2. If the failure is deemed to be the result of an improperly prepared calibration standard, re-prepare the standard and reanalyze it. Reanalyzing or replacing a single standard must NOT be confused with the practice

of discarding individual calibration results for specific target compounds in order to pick and choose a set of results that will meet the RSD or correlation criteria for the linear model. The practice of discarding individual calibration results is addressed as a fourth alternative option, and is very specific as to how a set of results are chosen to be discarded. If a standard is reanalyzed, or a new standard is analyzed, then ALL of the results from the original analysis of the standard in question must be discarded. Further, the practice of running additional standards at other concentrations and then picking only those results that meet the calibration acceptance criteria is EXPRESSLY PROHIBITED, since the analyst has generated data that demonstrate that the linear model does not apply to all of the data.

16.4. Internal Standards-

16.4.1. If the internal standards fail to meet their acceptance criteria, check calculations, the internal standard compound spiking solutions, and the instrument operation. If the calculations were incorrect, correct the calculations and verify that the internal standard response met their acceptance criteria.

16.4.2. If the internal standard compound spiking solution was improperly prepared, concentrated, or degraded, re-prepare solutions and re-extract/reanalyze the samples.

16.4.3. If the instrument malfunctioned, correct the instrument problem and reanalyze the sample extract. This correction will most likely involve instrument maintenance similar to the maintenance described in Section 16.1.2. If the instrument malfunction affected the calibration, recalibrate the instrument before reanalyzing the sample extract.

16.4.4. If the above actions do not correct the problem, then the problem may be due to a sample "matrix effect". To determine if there is a matrix effect, take the following corrective action steps.

16.4.4.1. Reanalyze the sample extract. EXCEPTION: if the internal standard compounds responses and/or retention times in a sample used for a matrix spike and/or matrix spike duplicate were outside the acceptance criteria, then it should be reanalyzed only if the internal standard responses and/or retention times were within the acceptance criteria for the associated matrix spike (MS only) or for both the matrix spike and matrix spike duplicate analysis (MS/MSD). These corrective actions are also to be applied in the case of un-spiked duplicates of a given sample which is outside acceptance criteria.

16.4.4.2. If the corrective actions listed above do not prove matrix effect, then the problem with the initial analysis is deemed to be within the laboratory's control. Submit either both sets of data or only the data from the reanalysis with the internal standard responses and retention times within acceptance limits.

16.4.4.3. If the corrective actions listed above prove matrix effect, then submit data from both analyses, distinguishing between the initial analysis and reanalysis on all deliverables.

16.4.4.4. If internal standard acceptance criteria are not met in an extract judged by the GC/MS analyst to be overly concentrated in background (e.g. hydrocarbons) and therefore damaging to the analytical system, the extract may be diluted prior to the reanalysis performed to prove matrix effect.

16.4.4.5. If a set of sample extracts from the same sample delivery group and/or samples collected from the same sample location are judged to be similar based on examination of the total ion chromatogram, it may be necessary only to reanalyze one extract or a subset of the extracts to prove matrix interference. Prior consultation with the Laboratory Manager, Project Manager and the client is necessary for this option to be used.

16.5. Surrogates

16.5.1. If the surrogate compounds fail to meet their recovery acceptance criteria, first reanalyze the extract.

16.5.2. If the surrogate recoveries do not meet their recovery acceptance criteria after reanalysis, check calculations, sample preparation logs, the surrogate compound spiking and calibration solutions and the instrument operation. If the calculations were incorrect, correct the calculations and verify that the surrogate compound recoveries meet their acceptance criteria. If the sample preparation logs indicate that the incorrect amount of surrogate compound spiking solution was added either recalculate surrogate recoveries based on the actual amount of surrogate compound spiking solution added, or re-extract/reanalyze the sample, adding the correct amount of surrogate spiking solution. If the surrogate compound spiking solution and/or surrogate calibration solution was improperly prepared, concentrated, or degraded, re-prepare solutions and re-extract/reanalyze samples.

16.5.3. If the analytical instrument malfunctioned, correct the instrument problem and reanalyze the sample extract. This correction will mostly involve maintenance similar to the maintenance discussed in Section 16.1.2. Verify that the surrogate recoveries meet their acceptance criteria. If the instrument malfunction affected the calibration, recalibrate the instrument before reanalyzing the sample extract.

16.5.4. If the above actions do not correct the problem, then the problem may be due to a sample matrix effect. To determine if there was matrix effect, take the following corrective action steps.

16.5.4.1. Re-extract and reanalyze the sample. EXCEPTION: If surrogate compound recoveries in a sample used for a matrix spike and/or matrix spike duplicate were considered unacceptable, then it should be re-extracted/reanalyzed only if the surrogate compound recoveries were within the acceptance criteria for the associated matrix spike (MS only) or for both the matrix spike and matrix spike duplicate analysis (MS/MSD). These corrective actions are also to be applied in the case of un-spiked duplicates of a given sample which is outside acceptance criteria.

16.5.4.2. If the surrogate compound recoveries meet acceptance criteria in the re-extracted/reanalyzed sample, then the problem with the initial analysis is deemed to be within the laboratory's control. Therefore, submit data only from the re-extraction/ reanalysis if the re-extraction was performed within holding time, otherwise report both sets of data.

16.5.4.3. If the surrogate compound recoveries fail to meet the acceptance criteria in the re-extracted/reanalyzed sample, then submit data from both analyses, distinguishing between the initial analysis and the re-extraction/reanalysis on all deliverables.

16.6. Method Blanks- Corrective action for a method blank which fails acceptance criteria may involve re-extraction and reanalysis of all associated samples and/or "B" flagging of the associated sample data. Each occurrence will be evaluated on an individual basis upon consultation with the Project Manager, the client, the Laboratory Supervisor, and the Laboratory Manager.

16.7. Laboratory Control Samples

16.7.1. If the LCS compounds fail to meet their recovery acceptance criteria, first reanalyze the extract.

16.7.2. If the LCS recoveries do not meet their recovery acceptance criteria after reanalysis, check calculations, sample preparation logs, the LCS compound spiking and calibration solutions and the instrument operation. If the calculations were incorrect, correct the calculations and verify that the LCS compound recoveries and RPDs (if applicable) meet their acceptance criteria. If the sample preparation logs indicate that the incorrect amount of LCS compound spiking solution was added either recalculate LCS recoveries based on the actual amount of LCS compound spiking solution added or re-extract/reanalyze the samples, adding the correct amount of LCS spiking solution. If the LCS compound spiking solution and/or LCS calibration solution was improperly prepared, concentrated, or degraded, re-prepare solutions and re-extract/reanalyze samples.

16.7.3. If the analytical instrument malfunctioned, correct the instrument problem and reanalyze the sample extract. Verify that the LCS recoveries meet their acceptance criteria.

If the instrument malfunction affected the calibration, recalibrate the instrument before reanalyzing the sample extract.

16.7.4. If the LCS compounds still fail to meet their acceptance criteria, re-extract and reanalyze the LCS and all associated samples and QC samples if deemed appropriate (i.e. after consideration of all batch QC data) or mandated by contract-specific requirements. Any decision to forgo re-extraction/reanalysis based on failure to meet LCS acceptance criteria will require approval of the Project Manager and the Lab Manager, at a minimum.

16.8. Matrix Spike/Matrix Spike Duplicates

16.8.1. If the MS/MSD compounds fail to meet their advisory acceptance criteria, it may be necessary to reanalyze the extracts; however, MS/MSD recoveries should not be used to reject batch data.

16.8.2. If this fails to bring the recoveries or RPDs into compliance, check calculations, sample preparation logs, the MS/MSD compound spiking and calibration solutions and the instrument operation. If the calculations were incorrect, correct the calculations and verify that the MS/MSD compound recoveries and RPD's (if applicable) meet their advisory acceptance criteria. If the sample preparation logs indicate that the incorrect amount of MS/MSD compound spiking solution was added either recalculate MS/MSD recoveries based on the actual amount of MS/MSD compound spiking solution added or re-extract/reanalyze the samples, adding the correct amount of MS/MSD spiking solution. If the MS/MSD compound spiking solution and/or MS/MSD calibration solution was improperly prepared, concentrated, or degraded, re-prepare solutions and re-extract/reanalyze samples.

16.8.3. If the analytical instrument malfunctioned, correct the instrument problem and reanalyze the sample extract. Verify that the MS/MSD recoveries meet their advisory acceptance criteria. If the instrument malfunction affected the calibration, recalibrate the instrument before reanalyzing the sample extract.

16.8.4. If the MS/MSD compounds still fail to meet their advisory acceptance criteria, an assessment of batch QC data is necessary in order to determine the possibility of a matrix effect. If the RPDs are within acceptance limits (particularly for non-native MS/MSD analytes) and all of the batch QC elements which are not affected by the sample matrix are in control (e.g. method blank, LCS, calibration checks) and there is no evidence of improper spiking levels, the poor recovery may be attributed to matrix effects. Additional consideration also needs to be given to the ratio of spike to native analyte, as well as sample heterogeneity, as these factors may have an adverse effect on both spike recovery and RPD

values. If matrix effect is determined to be the cause, the associated data must be flagged, but re-preparation and reanalysis is not required. Examination of spike recoveries in the matrix spike duplicate is also pertinent to assessing matrix effects.

16.8.5. If any of the batch QA elements which are not affected by the sample matrix are out of control, or there is evidence that spiking may have been improperly performed, the matrix spike and matrix spike duplicate must be re-extracted and reanalyzed. If insufficient sample is available or if the extraction holding time has elapsed, the laboratory must notify the client and flag the associated data and/or document the problem in the narrative.

16.9. Sample Dilution- When the calculated concentration of any analyte exceeds the working range of the curve, the analyte must be qualified with an “E” flag, and a dilution must be performed to reduce the analytes concentration to within the upper half calibration range ideally.

16.9.1. Additional internal standard must be added to the diluted extract to maintain the required 2.0 – 0.02 ng/µL of each internal standard in the extracted volume.

16.9.2. All dilutions should keep the response of the major constituents in the upper half of the linear range of the curve.

16.9.3. When ions from a target compound in the sample saturate the detector the analyst must:

16.9.3.1. Flag all affected analytes with an S flag.

16.9.3.2. Analyze an Instrument Blank consisting of clean solvent until the system has been decontaminated. It is advisable to reanalyze the sample run directly after the sample with the saturated analyte and compare the original and reanalysis values to look for carryover.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. See Section 16.1 for guidance on dealing with out-of-control tuning events.

17.2. See Section 16.2 for guidance on dealing with out-of-control events related to peak tailing factors or DDT breakdown.

17.3. See Section 16.3 for guidance on dealing with out-of-control initial and continuing calibration events.

17.4. See Section 16.4 for guidance on dealing with internal standard out-of-control events.

17.5. See Section 16.5 for guidance on dealing with surrogate out-of-control events.

17.6. See Section 16.6 for guidance on dealing with method blank related out-of-control events.

17.7. See Section 16.7 for guidance on dealing with laboratory control sample related out-of-control events.

17.8. See Section 16.8 for guidance on dealing with MS/MSD related out-of-control events.

17.9. See Section 16.9 for guidance on dealing with sample dilution related out-of-control events.

18. Waste Management

18.1. All extract vials must be disposed of by placing them in the blue hazardous waste drum in the station set aside for this purpose (Satellite Accumulation Station GC-2.) No vials may be thrown in the trash or receptacles not expressly designated for this purpose.

18.2. All solvents must be disposed of by pouring them out over charcoal. No solvent may be poured down the drain or disposed of in any other non-hygienic manner.

18.3. All spent charcoal must be disposed of by placing it in the charcoal disposal bin located in the extractions lab.

19. Method References

19.1. "Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Method 8270D, Test Methods for Evaluating Solid Waste (SW-846), Revision 4, February, 2007.

19.2. "Determinative Chromatographic Separations": Method 8000C, Test Methods For Evaluating Solid Waste (SW-846), Revision 3, March, 2003.

19.3. Department of Defense QSM, Version 4.1, April, 2009.

19.4. Krone *et al*, "A Method for Analysis of Butyltin Species and Measurement of Butyltins in Sediment and English Sole Livers from Puget Sound", *Marine Environmental Research*, **27** (1989) p 1-18

20. Appendices

20.1. Appendix 1: Manual Data Integration

20.2. Appendix 2: TBT Target Analytes, Quantitation Ions, Calibration Criteria and Associated Internal Standards

20.3. Appendix 3: DFTPP key ions and ion abundance criteria.

20.4. Appendix 4: TBT Ion Conversion Factor Table

20.5. Appendix 5: Calibration standard concentrations

20.6. Appendix 6: GC and GC/MS Semivolatile Organics Logbook (Example)

20.7. Appendix 7: Peak Tailing Factor Calculations

Appendix 20.1

Manual Integration

Manual Adjustment: Modern chromatographic instruments include computer software to identify a detector response as a chromatographic peak, characterize that peak and determine the relative height or area of the signal. The software utilizes parameters (threshold, slope, etc.) that are adjusted by the instrument operator to optimize the results. A single set of operator controlled settings that determine peak characteristics for an entire data file (or sets of data files) is defined as an "automated procedure." An automated procedure often characterizes chromatographic peaks incorrectly. ARI requires that trained analysts identify and resolve these errors using an alternative automated procedure or a "manual adjustment" of the data. Manual adjustment is defined as the process used by an analyst to adjust an individual peak or a subset of data in a chromatographic file.

The settings for the routine automated procedure normally used to process chromatographic data for this SOP are listed below for both the total (or reconstructed) ion chromatogram (RIC) used for the quantitation of TIC's and for the extracted ion current profile (EICP) used for the quantitation of target compound analytes and surrogates.

Parameter	RIC	EICP
Peak Detection - Start	0.200	0.200
Peak Detection - End	0.000	0.000
Bunch	1	1
Peak Smoothing	on	off
Area Cutoff	0 %	5 %
Maximum Peaks to Detect	200	100
Baseline Reset	3 - 5	5
Set Valley	100	100

Trained analysts may substitute one automated procedure for another in order to optimize peak characteristics. The use of an alternate automated procedure must be permanently documented using either a software generated log file or analyst notes.

Manual adjustment of chromatographic peak characteristics will be used to correct the results of an automated procedure that, in the trained analyst's opinion, are clearly incorrect and will result in erroneous peak identification, integration or quantification.

Manual adjustment will be implemented in a reasonable and consistent manner. The adjustment shall include only the area attributed to the specific target compound. The area adjusted shall not include baseline background noise. The area adjusted shall not extend past the points where the sides of the peak intersect with the baseline noise.

All manually adjusted data shall be clearly identified for approval in the data review process. A permanent record of all manual adjustments shall be maintained in both electronic and hardcopy versions of the data.

Manual adjustment of chromatographic data will not be used to falsify data for any purpose or as a substitute for corrective action on the chromatographic system. Falsification of data through the use of manual peak adjustment is unethical, unlawful and will result in the termination of the offending analyst.

An analyst who changes an integration must identifying the manual integration on the quantitation report then initial and date the report.

All manually integrated reported data will be flagged, and, when mandated by project-specific requirements, the final data report and associated documentation must provide justification for manual integrations.

Appendix 20.2
SIM - BTS Scan Descriptors, Target Analytes, Quantitation Ions, Calibration Criteria and Associated Internal Standards

Scan Group	Start Time (min)	Ions Scanned	Internal Standard Associated Analyte [2,3]	CAS Number	Primary Ion	Min RRF	Max %RSD [1]	Max %D [1]
1	2.00	231, 233, 235, 287, 289, 291	(SS) Tripropyl tin		291	0.01	20	20
			Tetrabutyl tin		289	0.01	20	20
2	6.00	231, 233, 235, 315, 317, 319	Tributyl tin	1461-22-9 [4] 36643-28-4 [5]	319	0.01	20	20
3	6.60	315, 317, 319, 333, 343, 345, 347	(IS) Tetrapentyl tin		333	NA	NA	20
			Dibutyl tin	683-18-1 [4] 14488-53-0 [5]	347	0.01	20	20
			(SS) Tripentyl tin		347	0.01	20	20
4	7.30	287, 289, 291, 343, 345, 347	Butyl tin	118-46-3 [4] 78763-54-9 [5]	347	0.01	20	20
5	7.60	244	(IS) d_{14}-p-Terphenyl		244	NA	NA	20

1 – All compounds must meet 20% RSD
2 – Compounds followed by (IS) are internal standards
3 – Compounds followed by (SS) are surrogate standards
4 – CAS numbers for the hexyl derivatives
5 – CAS number for the BTS ion

Appendix: 20.3
DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	30-80 % of mass 198
68	< 2 % of mass 69
70	< 2 % of mass 69
127	25-75 % of mass 198
197	< 1 % of mass 198
198	Base peak, 100 % relative abundance
199	5-9 % of mass 198
275	10-30 % of mass 198
365	> 0.75 of mass 198
441	Present but less than mass 443
442	40-110 % of mass 198
443	15-24 % of mass 442

Appendix 20.4 TBT Ion Conversion Factor Table

Analyte	Conversion to Chloride	Conversion to Tin Ion	Conversion to Total Tin
Hexyltributyltin	0.8675	0.773	0.316
Hexyldibutyltin	0.7534	0.578	0.294
Hexylbutyltin	0.6542	0.408	0.275
Hexyltripropyltin	0.8508	NA	NA
Hexyltripentyltin	0.8809	NA	NA

Appendix 20.5 Calibration concentrations

	Pore Water (ng/mL)					
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
Butyl tin	4	10	20	50	100	200
Dibutyl tin	4	10	20	50	100	200
Tributyl tin	2	5	10	25	50	100
Tetrabutyl tin	2	5	10	25	50	100
Tripropyl tin	2	5	10	25	50	100
Tripentyl tin	4	10	20	50	100	200
Tetrapentyl tin						
d_{14}-p-Terphenyl						

	Water & Solid Matrix (µg/mL)					
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
Butyl tin	0.1	0.4	1	2	4	8
Dibutyl tin	0.1	0.4	1	2	4	8
Tributyl tin	0.05	0.2	0.5	1	2	4
Tetrabutyl tin	0.05	0.2	0.5	1	2	4
Tripropyl tin	0.05	0.2	0.5	1	2	4
Tripentyl tin	0.1	0.4	1	2	4	8
Tetrapentyl tin						
d_{14}-p-Terphenyl						

Appendix 20.6
GC and GC/MS Semivolatile Organics Logbook
(Example)

Analytical Resources Inc.: Organics Instrument Log
NT-2 Serial No.: 82321977

Date:_____ Analysis: _____ Analyst: _____
GC Program: _____ Column No: _____ Column Type:_____
Instrument Tune (.U or .CT.):_____ EM Voltage: _____
Calibration File:_____ Curve Date: _____

IS/SS	Ical/Ccal	LCS/ICV

Maintenance / Comments

Maintenance Verification (Identify ICal or CCal that demonstrates the instrument is in control):

Every line must contain information or be lined out. Make all entries legible. Start a new page for each QC period.

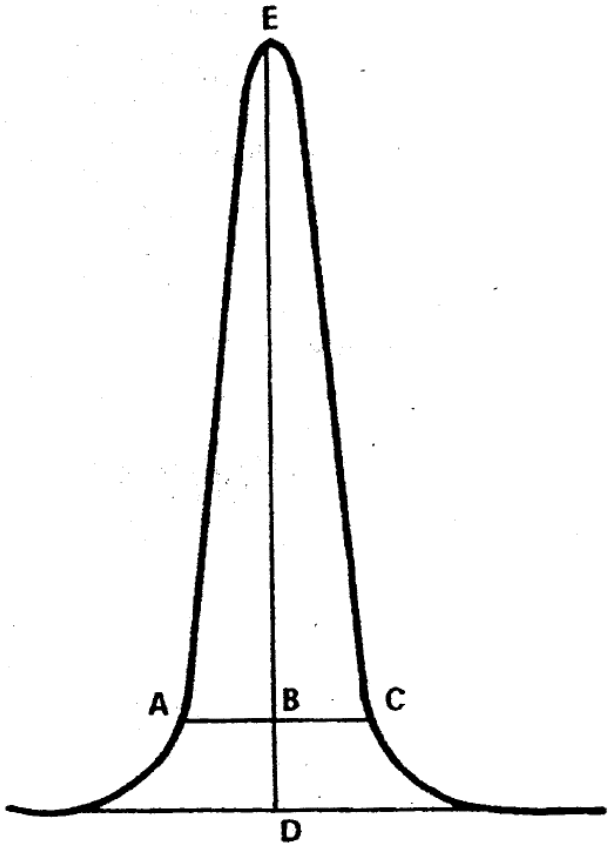
Form 7042F
Organic Instrument Log

NT-2 7/19/2006
Page 01849

Revision 001
1/16/06

Appendix 20.7
Peak Tailing Factor Calculations

$$RF = \frac{(A_s)\ (C_{is})}{(A_{is})\ (C_s)}$$



TAILING FACTOR= $\frac{BC}{AB}$

Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

Therefore: Tailing Factor = $\frac{12}{11}$ = 1.1

PERRY JOHNSON LABORATORY ACCREDITATION, INC.

Certificate of Accreditation

Perry Johnson Laboratory Accreditation, Inc., has assessed the Laboratory of:

Analytical Resources, Inc.
4611 South 134th Place, Suite 100
Tukwila, WA 98168

(Hereinafter called the Organization) and hereby declares that Organization has met the requirements of ISO/IEC 17025:2005 "General Requirements for the competence of Testing and Calibration Laboratories" and the DoD Quality Systems Manual for Environmental Laboratories Version 4.1 (4/22/2009) and is accredited in accordance with the**:**

United States Department of Defense Environmental Laboratory Accreditation Program (DoD-ELAP)

This accreditation demonstrates technical competence for the defined scope:

Environmental Testing (As detailed in the supplement)

Such testing shall only be offered at or from the address given above. This Accreditation is granted subject to the system rules governing the Accreditation referred to above, and the Organization hereby covenants with the Accreditation Body's duty to observe and comply with the said rules.

For PJLA:

The validity of this certificate is mandated through ongoing surveillance.

Tracy Szerszen
President/Operations Manager
Perry Johnson Laboratory
Accreditation, Inc. (PJLA)
755 Big Beaver, Suite 1325
Troy, Michigan 48084

Initial Accreditation Date:	Issue Date:	Expiration Date:
October 10, 2010	October 10, 2010	October 9, 2012
Accreditation No:	Certificate No:	Page No:
66169	L10-179	Page 1 of 17

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

Analytical Resources, Inc.
4611 South 134th Place, Suite 100
Tukwila, WA 98168

Accreditation is granted to this facility to perform the following testing:

Matrix	Standard / Method	Technology	Analyte
Aqueous	EPA 160.4	Gravimetric	Settleable Solids
Aqueous	EPA 180.1	Nephelometry	Turbidity
Aqueous	EPA 245.1	CVAA	Mercury
Aqueous	EPA 410.4	Spectrophotometric	COD
Aqueous	EPA 7470A	CVAA	Mercury
Aqueous	EPA 8260C	GC-MS	Diisopropyl ether
Aqueous	EPA 8260C	GC-MS	Ethanol
Aqueous	EPA 8260C	GC-MS	Ethyl-*tert*-butyl ether
Aqueous	EPA 8260C	GC-MS	*tert*-Amyl methyl ether
Aqueous	EPA 8260C	GC-MS	*tert*-Butanol
Aqueous	EPA 9060A	Combustion IR	Total Organic Carbon
Aqueous	SM 2130B-01	Nephelometry	Turbidity
Aqueous	SM 2320 B-97	Titration	Alkalinity
Aqueous	SM 2540-C 97	Gravimetric	TDS
Aqueous	SM 2540-D 97	Gravimetric	TSS
Aqueous	SM 2540-F 97	Gravimetric	Settleable Solids
Aqueous	SM 5220 D-97	Spectrophotometric	COD
Solids	EPA 3060A	Extraction	Hexavalent Chromium Preparation
Solids	EPA 7471B	CVAA	Mercury
Solids	Plumb 1981	Combustion IR	Total Organic Carbon
Aqueous/Solids	AK101	GC-FID	Gasoline (TPH-G, GRO)
Aqueous/Solids	AK102	GC-FID	Diesel (TPH-D, DRO)
Aqueous/Solids	AK103	GC-FID	Motor Oil (RRO)
Aqueous/Solids	EPA 1613B	HRGC-HRMS	1,2,3,4,6,7,8-HpCDD
Aqueous/Solids	EPA 1613B	HRGC-HRMS	1,2,3,4,6,7,8-HpCDD-13C
Aqueous/Solids	EPA 1613B	HRGC-HRMS	1,2,3,4,6,7,8-HpCDF
Aqueous/Solids	EPA 1613B	HRGC-HRMS	1,2,3,4,6,7,8-HpCDF-13C
Aqueous/Solids	EPA 1613B	HRGC-HRMS	1,2,3,4,7,8,9-HpCDF
Aqueous/Solids	EPA 1613B	HRGC-HRMS	1,2,3,4,7,8,9-HpCDF-13C
Aqueous/Solids	EPA 1613B	HRGC-HRMS	1,2,3,4,7,8-HxCDD
Aqueous/Solids	EPA 1613B	HRGC-HRMS	1,2,3,4,7,8-HxCDD-13C
Aqueous/Solids	EPA 1613B	HRGC-HRMS	1,2,3,4,7,8-HxCDF

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ISO/IEC 17025:2005 and DoD-ELAP

Analytical Resources, Inc.
4611 South 134th Place, Suite 100
Tukwila, WA 98168

Accreditation is granted to this facility to perform the following testing:

Matrix	Standard / Method	Technology	Analyte
Aqueous/Solids	EPA 1613B	HRGC-HRMS	1,2,3,4,7,8-HxCDF-13C
Aqueous/Solids	EPA 1613B	HRGC-HRMS	1,2,3,6,7,8-HxCDD
Aqueous/Solids	EPA 1613B	HRGC-HRMS	1,2,3,6,7,8-HxCDD-13C
Aqueous/Solids	EPA 1613B	HRGC-HRMS	1,2,3,6,7,8-HxCDF
Aqueous/Solids	EPA 1613B	HRGC-HRMS	1,2,3,6,7,8-HxCDF-13C
Aqueous/Solids	EPA 1613B	HRGC-HRMS	1,2,3,7,8,9-HxCDD
Aqueous/Solids	EPA 1613B	HRGC-HRMS	1,2,3,7,8,9-HxCDF
Aqueous/Solids	EPA 1613B	HRGC-HRMS	1,2,3,7,8,9-HxCDF-13C
Aqueous/Solids	EPA 1613B	HRGC-HRMS	1,2,3,7,8-PeCDD
Aqueous/Solids	EPA 1613B	HRGC-HRMS	1,2,3,7,8-PeCDD-13C
Aqueous/Solids	EPA 1613B	HRGC-HRMS	1,2,3,7,8-PeCDF
Aqueous/Solids	EPA 1613B	HRGC-HRMS	1,2,3,7,8-PeCDF-13C
Aqueous/Solids	EPA 1613B	HRGC-HRMS	2,3,4,6,7,8-HxCDF
Aqueous/Solids	EPA 1613B	HRGC-HRMS	2,3,4,6,7,8-HxCDF-13C
Aqueous/Solids	EPA 1613B	HRGC-HRMS	2,3,4,7,8-PeCDF
Aqueous/Solids	EPA 1613B	HRGC-HRMS	2,3,4,7,8-PeCDF-13C
Aqueous/Solids	EPA 1613B	HRGC-HRMS	2,3,7,8-TCDD
Aqueous/Solids	EPA 1613B	HRGC-HRMS	2,3,7,8-TCDD-13C
Aqueous/Solids	EPA 1613B	HRGC-HRMS	2,3,7,8-TCDD-37Cl4
Aqueous/Solids	EPA 1613B	HRGC-HRMS	2,3,7,8-TCDF
Aqueous/Solids	EPA 1613B	HRGC-HRMS	2,3,7,8-TCDF-13C
Aqueous/Solids	EPA 1613B	HRGC-HRMS	OCDD
Aqueous/Solids	EPA 1613B	HRGC-HRMS	OCDD-13C
Aqueous/Solids	EPA 1613B	HRGC-HRMS	OCDF
Aqueous/Solids	EPA 1613B	HRGC-HRMS	Total 2,3,7,8-TCDD Equiv
Aqueous/Solids	EPA 1613B	HRGC-HRMS	Total HpCDD
Aqueous/Solids	EPA 1613B	HRGC-HRMS	Total HpCDF
Aqueous/Solids	EPA 1613B	HRGC-HRMS	Total HXCDD
Aqueous/Solids	EPA 1613B	HRGC-HRMS	Total HxCDF
Aqueous/Solids	EPA 1613B	HRGC-HRMS	Total PeCDD
Aqueous/Solids	EPA 1613B	HRGC-HRMS	Total PeCDF
Aqueous/Solids	EPA 1613B	HRGC-HRMS	Total TCDD

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Matrix	Standard / Method	Technology	Analyte
Aqueous/Solids	EPA 1613B	HRGC-HRMS	Total TCDF
Aqueous/Solids	EPA 300.0	IC	Bromide
Aqueous/Solids	EPA 300.0	IC	Chloride
Aqueous/Solids	EPA 300.0	IC	Fluoride
Aqueous/Solids	EPA 300.0	IC	Nitrate
Aqueous/Solids	EPA 300.0	IC	Nitrite
Aqueous/Solids	EPA 300.0	IC	Phosphate
Aqueous/Solids	EPA 300.0	IC	Sulfate
Aqueous/Solids	EPA 6010C	ICP-OES	Aluminum
Aqueous/Solids	EPA 6010C	ICP-OES	Antimony
Aqueous/Solids	EPA 6010C	ICP-OES	Arsenic
Aqueous/Solids	EPA 6010C	ICP-OES	Barium
Aqueous/Solids	EPA 6010C	ICP-OES	Beryllium
Aqueous/Solids	EPA 6010C	ICP-OES	Boron
Aqueous/Solids	EPA 6010C	ICP-OES	Cadmium
Aqueous/Solids	EPA 6010C	ICP-OES	Calcium
Aqueous/Solids	EPA 6010C	ICP-OES	Chromium
Aqueous/Solids	EPA 6010C	ICP-OES	Cobalt
Aqueous/Solids	EPA 6010C	ICP-OES	Copper
Aqueous/Solids	EPA 6010C	ICP-OES	Iron
Aqueous/Solids	EPA 6010C	ICP-OES	Lead
Aqueous/Solids	EPA 6010C	ICP-OES	Magnesium
Aqueous/Solids	EPA 6010C	ICP-OES	Manganese
Aqueous/Solids	EPA 6010C	ICP-OES	Molybdenum
Aqueous/Solids	EPA 6010C	ICP-OES	Nickel
Aqueous/Solids	EPA 6010C	ICP-OES	Potassium
Aqueous/Solids	EPA 6010C	ICP-OES	Selenium
Aqueous/Solids	EPA 6010C	ICP-OES	Silicon
Aqueous/Solids	EPA 6010C	ICP-OES	Silver
Aqueous/Solids	EPA 6010C	ICP-OES	Sodium
Aqueous/Solids	EPA 6010C	ICP-OES	Strontium
Aqueous/Solids	EPA 6010C	ICP-OES	Thallium

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4611 South 134th Place, Suite 100
Tukwila, WA 98168

Accreditation is granted to this facility to perform the following testing:

Matrix	Standard / Method	Technology	Analyte
Aqueous/Solids	EPA 6010C	ICP-OES	Tin
Aqueous/Solids	EPA 6010C	ICP-OES	Titanium
Aqueous/Solids	EPA 6010C	ICP-OES	Vanadium
Aqueous/Solids	EPA 6010C	ICP-OES	Zinc
Aqueous/Solids	EPA 6020A	ICP-MS	Aluminum
Aqueous/Solids	EPA 6020A	ICP-MS	Antimony
Aqueous/Solids	EPA 6020A	ICP-MS	Arsenic
Aqueous/Solids	EPA 6020A	ICP-MS	Barium
Aqueous/Solids	EPA 6020A	ICP-MS	Beryllium
Aqueous/Solids	EPA 6020A	ICP-MS	Cadmium
Aqueous/Solids	EPA 6020A	ICP-MS	Calcium
Aqueous/Solids	EPA 6020A	ICP-MS	Chromium
Aqueous/Solids	EPA 6020A	ICP-MS	Cobalt
Aqueous/Solids	EPA 6020A	ICP-MS	Copper
Aqueous/Solids	EPA 6020A	ICP-MS	Iron
Aqueous/Solids	EPA 6020A	ICP-MS	Lead
Aqueous/Solids	EPA 6020A	ICP-MS	Magnesium
Aqueous/Solids	EPA 6020A	ICP-MS	Manganese
Aqueous/Solids	EPA 6020A	ICP-MS	Molybdenum
Aqueous/Solids	EPA 6020A	ICP-MS	Nickel
Aqueous/Solids	EPA 6020A	ICP-MS	Potassium
Aqueous/Solids	EPA 6020A	ICP-MS	Selenium
Aqueous/Solids	EPA 6020A	ICP-MS	Silver
Aqueous/Solids	EPA 6020A	ICP-MS	Sodium
Aqueous/Solids	EPA 6020A	ICP-MS	Thallium
Aqueous/Solids	EPA 6020A	ICP-MS	Thorium
Aqueous/Solids	EPA 6020A	ICP-MS	Titanium
Aqueous/Solids	EPA 6020A	ICP-MS	Uranium
Aqueous/Solids	EPA 6020A	ICP-MS	Vanadium
Aqueous/Solids	EPA 6020A	ICP-MS	Zinc
Aqueous/Solids	EPA 7196A	Colorimetric	Hexavalent Chromium
Aqueous/Solids	EPA 8015C	GC-FID	Diesel (TPH-D, DRO)

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Accreditation is granted to this facility to perform the following testing:

Matrix	Standard / Method	Technology	Analyte
Aqueous/Solids	EPA 8015C	GC-FID	Motor Oil (RRO)
Aqueous/Solids	EPA 8081B	GC-ECD	2,4'-DDD
Aqueous/Solids	EPA 8081B	GC-ECD	2,4'-DDE
Aqueous/Solids	EPA 8081B	GC-ECD	2,4'-DDT
Aqueous/Solids	EPA 8081B	GC-ECD	4,4'-DDD
Aqueous/Solids	EPA 8081B	GC-ECD	4,4'-DDE
Aqueous/Solids	EPA 8081B	GC-ECD	4,4'-DDT
Aqueous/Solids	EPA 8081B	GC-ECD	Aldrin
Aqueous/Solids	EPA 8081B	GC-ECD	Alpha-BHC
Aqueous/Solids	EPA 8081B	GC-ECD	Beta-BHC
Aqueous/Solids	EPA 8081B	GC-ECD	Chlordane-technical
Aqueous/Solids	EPA 8081B	GC-ECD	*cis*-Chlordane
Aqueous/Solids	EPA 8081B	GC-ECD	*cis*-Nonachlor
Aqueous/Solids	EPA 8081B	GC-ECD	Delta-BHC
Aqueous/Solids	EPA 8081B	GC-ECD	Dieldrin
Aqueous/Solids	EPA 8081B	GC-ECD	Endosulfan I
Aqueous/Solids	EPA 8081B	GC-ECD	Endosulfan II
Aqueous/Solids	EPA 8081B	GC-ECD	Endosulfan Sulfate
Aqueous/Solids	EPA 8081B	GC-ECD	Endrin
Aqueous/Solids	EPA 8081B	GC-ECD	Endrin Aldehyde
Aqueous/Solids	EPA 8081B	GC-ECD	Endrin Ketone
Aqueous/Solids	EPA 8081B	GC-ECD	Gamma-BHC (Lindane)
Aqueous/Solids	EPA 8081B	GC-ECD	Heptachlor
Aqueous/Solids	EPA 8081B	GC-ECD	Heptachlor Epoxide
Aqueous/Solids	EPA 8081B	GC-ECD	Hexachlorobenzene
Aqueous/Solids	EPA 8081B	GC-ECD	Hexachloroethane
Aqueous/Solids	EPA 8081B	GC-ECD	Hexchlorobutadiene
Aqueous/Solids	EPA 8081B	GC-ECD	Methoxychlor
Aqueous/Solids	EPA 8081B	GC-ECD	Mirex
Aqueous/Solids	EPA 8081B	GC-ECD	*oxy*-Chlordane
Aqueous/Solids	EPA 8081B	GC-ECD	Toxaphene

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Tukwila, WA 98168

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Matrix	Standard / Method	Technology	Analyte
Aqueous/Solids	EPA 8081B	GC-ECD	*trans*-Chlordane
Aqueous/Solids	EPA 8081B	GC-ECD	*trans*-Nonachlor
Aqueous/Solids	EPA 8082A	GC-ECD	Aroclor-1016 (PCB-1016)
Aqueous/Solids	EPA 8082A	GC-ECD	Aroclor-1221 (PCB-1221)
Aqueous/Solids	EPA 8082A	GC-ECD	Aroclor-1232 (PCB-1232)
Aqueous/Solids	EPA 8082A	GC-ECD	Aroclor-1242 (PCB-1242)
Aqueous/Solids	EPA 8082A	GC-ECD	Aroclor-1248 (PCB-1248)
Aqueous/Solids	EPA 8082A	GC-ECD	Aroclor-1254 (PCB-1254)
Aqueous/Solids	EPA 8082A	GC-ECD	Aroclor-1260 (PCB-1260)
Aqueous/Solids	EPA 8082A	GC-ECD	Aroclor-1262 (PCB-1262)
Aqueous/Solids	EPA 8082A	GC-ECD	Aroclor-1268 (PCB-1268)
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #101
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #105
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #110
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #118
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #128
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #132
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #138
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #141
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #149
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #151
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #153
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #156
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #158
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #170
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #174
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #177
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #18
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #180
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #183
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #187
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #194

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4611 South 134th Place, Suite 100
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Accreditation is granted to this facility to perform the following testing:

Matrix	Standard / Method	Technology	Analyte
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #195
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #201
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #203
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #28
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #31
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #33
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #44
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #49
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #52
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #56
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #60
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #66
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #70
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #74
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #8
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #87
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #95
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #97
Aqueous/Solids	EPA 8082A	GC-ECD	PCB #99
Aqueous/Solids	EPA 8151A	GC-ECD	Pentachlorophenol
Aqueous/Solids	EPA 8260C	GC-MS	1,1,1,2-Tetrachloroethane
Aqueous/Solids	EPA 8260C	GC-MS	1,1,1-Trichloroethane
Aqueous/Solids	EPA 8260C	GC-MS	1,1,2,2-Tetrachloroethane
Aqueous/Solids	EPA 8260C	GC-MS	1,1,2-Trichloro-1,2,2-Trifluoroethane
Aqueous/Solids	EPA 8260C	GC-MS	1,1,2-Trichloroethane
Aqueous/Solids	EPA 8260C	GC-MS	1,1-Dichloroethane
Aqueous/Solids	EPA 8260C	GC-MS	1,1-Dichloroethene
Aqueous/Solids	EPA 8260C	GC-MS	1,1-Dichloropropene
Aqueous/Solids	EPA 8260C	GC-MS	1,2,3-Trichlorobenzene
Aqueous/Solids	EPA 8260C	GC-MS	1,2,3-Trichloropropane
Aqueous/Solids	EPA 8260C	GC-MS	1,2,4-Trichlorobenzene
Aqueous/Solids	EPA 8260C	GC-MS	1,2,4-Trimethylbenzene

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Matrix	Standard / Method	Technology	Analyte
Aqueous/Solids	EPA 8260C	GC-MS	1,2-Dibromo 3-Chloropropane
Aqueous/Solids	EPA 8260C	GC-MS	1,2-Dibromoethane
Aqueous/Solids	EPA 8260C	GC-MS	1,2-Dichlorobenzene
Aqueous/Solids	EPA 8260C	GC-MS	1,2-Dichloroethane
Aqueous/Solids	EPA 8260C	GC-MS	1,2-Dichloropropane
Aqueous/Solids	EPA 8260C	GC-MS	1,3,5-Trimethyl Benzene
Aqueous/Solids	EPA 8260C	GC-MS	1,3-Dichlorobenzene
Aqueous/Solids	EPA 8260C	GC-MS	1,3-Dichloropropane
Aqueous/Solids	EPA 8260C	GC-MS	1,4-Dichlorobenzene
Aqueous/Solids	EPA 8260C	GC-MS	2,2-Dichloropropane
Aqueous/Solids	EPA 8260C	GC-MS	2-Butanone
Aqueous/Solids	EPA 8260C	GC-MS	2-Chloroethyl Vinyl Ether
Aqueous/Solids	EPA 8260C	GC-MS	2-Chlorotoluene
Aqueous/Solids	EPA 8260C	GC-MS	2-Hexanone
Aqueous/Solids	EPA 8260C	GC-MS	4-Chlorotoluene
Aqueous/Solids	EPA 8260C	GC-MS	4-Isopropyl Toluene
Aqueous/Solids	EPA 8260C	GC-MS	4-Methyl-2-Pentanone
Aqueous/Solids	EPA 8260C	GC-MS	Acetone
Aqueous/Solids	EPA 8260C	GC-MS	Acrolein
Aqueous/Solids	EPA 8260C	GC-MS	Acrylonitrile
Aqueous/Solids	EPA 8260C	GC-MS	Benzene
Aqueous/Solids	EPA 8260C	GC-MS	Bromobenzene
Aqueous/Solids	EPA 8260C	GC-MS	Bromochloromethane
Aqueous/Solids	EPA 8260C	GC-MS	Bromodichloromethane
Aqueous/Solids	EPA 8260C	GC-MS	Bromoethane
Aqueous/Solids	EPA 8260C	GC-MS	Bromoform
Aqueous/Solids	EPA 8260C	GC-MS	Bromomethane
Aqueous/Solids	EPA 8260C	GC-MS	Carbon Disulfide
Aqueous/Solids	EPA 8260C	GC-MS	Carbon Tetrachloride
Aqueous/Solids	EPA 8260C	GC-MS	Chlorobenzene
Aqueous/Solids	EPA 8260C	GC-MS	Chlorodibromomethane
Aqueous/Solids	EPA 8260C	GC-MS	Chloroethane

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Matrix	Standard / Method	Technology	Analyte
Aqueous/Solids	EPA 8260C	GC-MS	Chloroform
Aqueous/Solids	EPA 8260C	GC-MS	Chloromethane
Aqueous/Solids	EPA 8260C	GC-MS	Cis-1,2-Dichloroethene
Aqueous/Solids	EPA 8260C	GC-MS	cis-1,3-Dichloropropene
Aqueous/Solids	EPA 8260C	GC-MS	Dibromomethane
Aqueous/Solids	EPA 8260C	GC-MS	Dichlorodifluoromethane
Aqueous/Solids	EPA 8260C	GC-MS	Ethyl Benzene
Aqueous/Solids	EPA 8260C	GC-MS	Hexachloro-1,3-Butadiene
Aqueous/Solids	EPA 8260C	GC-MS	Iodomethane
Aqueous/Solids	EPA 8260C	GC-MS	Isopropyl Benzene
Aqueous/Solids	EPA 8260C	GC-MS	m,p-Xylene
Aqueous/Solids	EPA 8260C	GC-MS	Methylene Chloride
Aqueous/Solids	EPA 8260C	GC-MS	Methyl-t-butyl ether (MTBE)
Aqueous/Solids	EPA 8260C	GC-MS	Naphthalene
Aqueous/Solids	EPA 8260C	GC-MS	n-Butyl Benzene
Aqueous/Solids	EPA 8260C	GC-MS	n-Propyl Benzene
Aqueous/Solids	EPA 8260C	GC-MS	o-Xylene
Aqueous/Solids	EPA 8260C	GC-MS	s-Butylbenzene
Aqueous/Solids	EPA 8260C	GC-MS	Styrene
Aqueous/Solids	EPA 8260C	GC-MS	t-Butylbenzene
Aqueous/Solids	EPA 8260C	GC-MS	Tetrachloroethene
Aqueous/Solids	EPA 8260C	GC-MS	Toluene
Aqueous/Solids	EPA 8260C	GC-MS	trans-1,2-Dichloroethene
Aqueous/Solids	EPA 8260C	GC-MS	trans-1,3-Dichloropropene
Aqueous/Solids	EPA 8260C	GC-MS	trans-1,4-Dichloro-2-Butene
Aqueous/Solids	EPA 8260C	GC-MS	Trichloroethene
Aqueous/Solids	EPA 8260C	GC-MS	Trichlorofluoromethane
Aqueous/Solids	EPA 8260C	GC-MS	Vinyl Acetate
Aqueous/Solids	EPA 8260C	GC-MS	Vinyl Chloride
Aqueous/Solids	EPA 8270D	GC-MS	1,2,4,5-Tetrachlorobenzene
Aqueous/Solids	EPA 8270D	GC-MS	1,2,4-Trichlorobenzene
Aqueous/Solids	EPA 8270D	GC-MS	1,2-Dichlorobenzene

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Matrix	Standard / Method	Technology	Analyte
Aqueous/Solids	EPA 8270D	GC-MS	1,3-Dichlorobenzene
Aqueous/Solids	EPA 8270D	GC-MS	1,4-Dichlorobenzene
Aqueous/Solids	EPA 8270D	GC-MS	1,4-Dioxane
Aqueous/Solids	EPA 8270D	GC-MS	1-methylnaphthalene
Aqueous/Solids	EPA 8270D	GC-MS	2,2'-oxybis(1-Chloropropane)
Aqueous/Solids	EPA 8270D	GC-MS	2,3,4,6-Tetrachlorophenol
Aqueous/Solids	EPA 8270D	GC-MS	2,4,5-Trichlorophenol
Aqueous/Solids	EPA 8270D	GC-MS	2,4,6-Trichlorophenol
Aqueous/Solids	EPA 8270D	GC-MS	2,4-Dichlorophenol
Aqueous/Solids	EPA 8270D	GC-MS	2,4-Dimethylphenol
Aqueous/Solids	EPA 8270D	GC-MS	2,4-Dinitrophenol
Aqueous/Solids	EPA 8270D	GC-MS	2,4-Dinitrotoluene
Aqueous/Solids	EPA 8270D	GC-MS	2,6-Dinitrotoluene
Aqueous/Solids	EPA 8270D	GC-MS	2-Benzyl-4-Chlorophenol
Aqueous/Solids	EPA 8270D	GC-MS	2-Chloronaphthalene
Aqueous/Solids	EPA 8270D	GC-MS	2-Chlorophenol
Aqueous/Solids	EPA 8270D	GC-MS	2-Methylnaphthalene
Aqueous/Solids	EPA 8270D	GC-MS	2-Methylphenol
Aqueous/Solids	EPA 8270D	GC-MS	2-Nitroaniline
Aqueous/Solids	EPA 8270D	GC-MS	2-Nitrophenol
Aqueous/Solids	EPA 8270D	GC-MS	3,3'-Dichlorobenzidine
Aqueous/Solids	EPA 8270D	GC-MS	3,4,5-Trichloroguaiacol
Aqueous/Solids	EPA 8270D	GC-MS	3,4,6-Trichloroguaiacol
Aqueous/Solids	EPA 8270D	GC-MS	3,4-Dichloroguaiacol
Aqueous/Solids	EPA 8270D	GC-MS	3-Nitroaniline
Aqueous/Solids	EPA 8270D	GC-MS	4,5,6-Trichloroguaiacol
Aqueous/Solids	EPA 8270D	GC-MS	4,5-Dichloroguaiacol
Aqueous/Solids	EPA 8270D	GC-MS	4,6-Dichloroguaiacol
Aqueous/Solids	EPA 8270D	GC-MS	4,6-Dinitro-2-methylphenol
Aqueous/Solids	EPA 8270D	GC-MS	4-Bromophenyl-phenylether
Aqueous/Solids	EPA 8270D	GC-MS	4-Chloro-3-methylphenol
Aqueous/Solids	EPA 8270D	GC-MS	4-Chloroaniline

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Analytical Resources, Inc.
4611 South 134th Place, Suite 100
Tukwila, WA 98168

Accreditation is granted to this facility to perform the following testing:

Matrix	Standard / Method	Technology	Analyte
Aqueous/Solids	EPA 8270D	GC-MS	4-Chloroguaiacol
Aqueous/Solids	EPA 8270D	GC-MS	4-Chlorophenyl-phenylether
Aqueous/Solids	EPA 8270D	GC-MS	4-Methylphenol
Aqueous/Solids	EPA 8270D	GC-MS	4-Nitroaniline
Aqueous/Solids	EPA 8270D	GC-MS	4-Nitrophenol
Aqueous/Solids	EPA 8270D	GC-MS	Acenaphthene
Aqueous/Solids	EPA 8270D	GC-MS	Acenaphthylene
Aqueous/Solids	EPA 8270D	GC-MS	Acetophenone
Aqueous/Solids	EPA 8270D	GC-MS	alpha-Terpineol
Aqueous/Solids	EPA 8270D	GC-MS	Aniline
Aqueous/Solids	EPA 8270D	GC-MS	Anthracene
Aqueous/Solids	EPA 8270D	GC-MS	Azobenzene (1,2-DP-Hydrazine)
Aqueous/Solids	EPA 8270D	GC-MS	Benzidine
Aqueous/Solids	EPA 8270D	GC-MS	Benzo(a)anthracene
Aqueous/Solids	EPA 8270D	GC-MS	Benzo(a)pyrene
Aqueous/Solids	EPA 8270D	GC-MS	Benzo(b)fluoranthene
Aqueous/Solids	EPA 8270D	GC-MS	Benzo(g,h,i)perylene
Aqueous/Solids	EPA 8270D	GC-MS	Benzo(k)fluoranthene
Aqueous/Solids	EPA 8270D	GC-MS	Benzoic acid
Aqueous/Solids	EPA 8270D	GC-MS	Benzyl alcohol
Aqueous/Solids	EPA 8270D	GC-MS	Biphenyl
Aqueous/Solids	EPA 8270D	GC-MS	Bis(2-Chloroethoxy)methane
Aqueous/Solids	EPA 8270D	GC-MS	Bis(2-Chloroethyl)ether
Aqueous/Solids	EPA 8270D	GC-MS	bis(2-Ethylhexyl)phthalate
Aqueous/Solids	EPA 8270D	GC-MS	Butyl Diphenyl Phosphate
Aqueous/Solids	EPA 8270D	GC-MS	Butylatedhydroxytoluene (BHT)
Aqueous/Solids	EPA 8270D	GC-MS	Butylbenzylphthalate
Aqueous/Solids	EPA 8270D	GC-MS	Carbaryl
Aqueous/Solids	EPA 8270D	GC-MS	Carbazole
Aqueous/Solids	EPA 8270D	GC-MS	Chrysene
Aqueous/Solids	EPA 8270D	GC-MS	Dibenzo(a,h)anthracene
Aqueous/Solids	EPA 8270D	GC-MS	Dibenzofuran

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Aqueous/Solids	EPA 8270D	GC-MS	Dibutyl Phenyl Phosphate
Aqueous/Solids	EPA 8270D	GC-MS	Dibutyltin ion
Aqueous/Solids	EPA 8270D	GC-MS	Diethylphthalate
Aqueous/Solids	EPA 8270D	GC-MS	Dimethylphthalate
Aqueous/Solids	EPA 8270D	GC-MS	Di-n-butylphthalate
Aqueous/Solids	EPA 8270D	GC-MS	Di-n-octylphthalate
Aqueous/Solids	EPA 8270D	GC-MS	Diphenyl Oxide
Aqueous/Solids	EPA 8270D	GC-MS	Fluoranthene
Aqueous/Solids	EPA 8270D	GC-MS	Fluorene
Aqueous/Solids	EPA 8270D	GC-MS	Guaiacol
Aqueous/Solids	EPA 8270D	GC-MS	Hexachlorobenzene
Aqueous/Solids	EPA 8270D	GC-MS	Hexachlorobutadiene
Aqueous/Solids	EPA 8270D	GC-MS	Hexachlorocyclopentadiene
Aqueous/Solids	EPA 8270D	GC-MS	Hexachloroethane
Aqueous/Solids	EPA 8270D	GC-MS	Indeno(1,2,3-cd)pyrene
Aqueous/Solids	EPA 8270D	GC-MS	Isophorone
Aqueous/Solids	EPA 8270D	GC-MS	Monobutyltin ion
Aqueous/Solids	EPA 8270D	GC-MS	Naphthalene
Aqueous/Solids	EPA 8270D	GC-MS	n-Decane
Aqueous/Solids	EPA 8270D	GC-MS	Nitrobenzene
Aqueous/Solids	EPA 8270D	GC-MS	N-Nitrosodimethylamine
Aqueous/Solids	EPA 8270D	GC-MS	N-Nitroso-di-n-propylamine
Aqueous/Solids	EPA 8270D	GC-MS	N-Nitrosodiphenylamine
Aqueous/Solids	EPA 8270D	GC-MS	n-Octadecane
Aqueous/Solids	EPA 8270D	GC-MS	p-Benzoquinone
Aqueous/Solids	EPA 8270D	GC-MS	Pentachlorobenzene
Aqueous/Solids	EPA 8270D	GC-MS	Pentachlorophenol
Aqueous/Solids	EPA 8270D	GC-MS	Phenanthrene
Aqueous/Solids	EPA 8270D	GC-MS	Phenol
Aqueous/Solids	EPA 8270D	GC-MS	Pyrene
Aqueous/Solids	EPA 8270D	GC-MS	Pyridine
Aqueous/Solids	EPA 8270D	GC-MS	Retene

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Matrix	Standard / Method	Technology	Analyte
Aqueous/Solids	EPA 8270D	GC-MS	Tetrachloroguaiacol
Aqueous/Solids	EPA 8270D	GC-MS	Tributyl Phosphate
Aqueous/Solids	EPA 8270D	GC-MS	Tributyltin ion
Aqueous/Solids	EPA 8270D	GC-MS	Triphenyl Phosphate
Aqueous/Solids	EPA 8290A	HRGC-HRMS	1,2,3,4,6,7,8-HpCDD
Aqueous/Solids	EPA 8290A	HRGC-HRMS	1,2,3,4,6,7,8-HpCDD-13C
Aqueous/Solids	EPA 8290A	HRGC-HRMS	1,2,3,4,6,7,8-HpCDF
Aqueous/Solids	EPA 8290A	HRGC-HRMS	1,2,3,4,6,7,8-HpCDF-13C
Aqueous/Solids	EPA 8290A	HRGC-HRMS	1,2,3,4,7,8,9-HpCDF
Aqueous/Solids	EPA 8290A	HRGC-HRMS	1,2,3,4,7,8,9-HpCDF-13C
Aqueous/Solids	EPA 8290A	HRGC-HRMS	1,2,3,4,7,8-HxCDD
Aqueous/Solids	EPA 8290A	HRGC-HRMS	1,2,3,4,7,8-HxCDD-13C
Aqueous/Solids	EPA 8290A	HRGC-HRMS	1,2,3,4,7,8-HxCDF
Aqueous/Solids	EPA 8290A	HRGC-HRMS	1,2,3,4,7,8-HxCDF-13C
Aqueous/Solids	EPA 8290A	HRGC-HRMS	1,2,3,6,7,8-HxCDD
Aqueous/Solids	EPA 8290A	HRGC-HRMS	1,2,3,6,7,8-HxCDD-13C
Aqueous/Solids	EPA 8290A	HRGC-HRMS	1,2,3,6,7,8-HxCDF
Aqueous/Solids	EPA 8290A	HRGC-HRMS	1,2,3,6,7,8-HxCDF-13C
Aqueous/Solids	EPA 8290A	HRGC-HRMS	1,2,3,7,8,9-HxCDD
Aqueous/Solids	EPA 8290A	HRGC-HRMS	1,2,3,7,8,9-HxCDF
Aqueous/Solids	EPA 8290A	HRGC-HRMS	1,2,3,7,8,9-HxCDF-13C
Aqueous/Solids	EPA 8290A	HRGC-HRMS	1,2,3,7,8-PeCDD
Aqueous/Solids	EPA 8290A	HRGC-HRMS	1,2,3,7,8-PeCDD-13C
Aqueous/Solids	EPA 8290A	HRGC-HRMS	1,2,3,7,8-PeCDF
Aqueous/Solids	EPA 8290A	HRGC-HRMS	1,2,3,7,8-PeCDF-13C
Aqueous/Solids	EPA 8290A	HRGC-HRMS	2,3,4,6,7,8-HxCDF
Aqueous/Solids	EPA 8290A	HRGC-HRMS	2,3,4,6,7,8-HxCDF-13C
Aqueous/Solids	EPA 8290A	HRGC-HRMS	2,3,4,7,8-PeCDF
Aqueous/Solids	EPA 8290A	HRGC-HRMS	2,3,4,7,8-PeCDF-13C
Aqueous/Solids	EPA 8290A	HRGC-HRMS	2,3,7,8-TCDD
Aqueous/Solids	EPA 8290A	HRGC-HRMS	2,3,7,8-TCDD-13C
Aqueous/Solids	EPA 8290A	HRGC-HRMS	2,3,7,8-TCDD-37Cl4

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Matrix	Standard / Method	Technology	Analyte
Aqueous/Solids	EPA 8290A	HRGC-HRMS	2,3,7,8-TCDF
Aqueous/Solids	EPA 8290A	HRGC-HRMS	2,3,7,8-TCDF-13C
Aqueous/Solids	EPA 8290A	HRGC-HRMS	OCDD
Aqueous/Solids	EPA 8290A	HRGC-HRMS	OCDD-13C
Aqueous/Solids	EPA 8290A	HRGC-HRMS	OCDF
Aqueous/Solids	EPA 8290A	HRGC-HRMS	Total 2,3,7,8-TCDD Equiv
Aqueous/Solids	EPA 8290A	HRGC-HRMS	Total HpCDD
Aqueous/Solids	EPA 8290A	HRGC-HRMS	Total HpCDF
Aqueous/Solids	EPA 8290A	HRGC-HRMS	Total HXCDD
Aqueous/Solids	EPA 8290A	HRGC-HRMS	Total HxCDF
Aqueous/Solids	EPA 8290A	HRGC-HRMS	Total PeCDD
Aqueous/Solids	EPA 8290A	HRGC-HRMS	Total PeCDF
Aqueous/Solids	EPA 8290A	HRGC-HRMS	Total TCDD
Aqueous/Solids	EPA 8290A	HRGC-HRMS	Total TCDF
Aqueous/Solids	EPA 9010C	Distillation	Cyanide Preparation
Aqueous/Solids	EPA 9014	Colorimetric	Cyanide
Aqueous/Solids	EPA 9030B	Distillation	Sulfide Preparation
Aqueous/Solids	EPA 9034	Titration	Sulfide
Aqueous/Solids	EPA 9045D	Electrometric	pH
Aqueous/Solids	EPA 9056A	IC	Bromide
Aqueous/Solids	EPA 9056A	IC	Chloride
Aqueous/Solids	EPA 9056A	IC	Fluoride
Aqueous/Solids	EPA 9056A	IC	Nitrate
Aqueous/Solids	EPA 9056A	IC	Nitrite
Aqueous/Solids	EPA 9056A	IC	Phosphate
Aqueous/Solids	EPA 9056A	IC	Sulfate
Aqueous/Solids	NWTPH-D	GC-FID	Diesel (TPH-D, DRO)
Aqueous/Solids	NWTPH-Dext	GC-FID	Motor Oil (RRO)
Aqueous/Solids	NWTPH-G	GC-FID/PID	Gasoline (TPH-G, GRO)
Aqueous/Solids	SM 2540-B 97	Gravimetric	TS
Aqueous/Solids	SM 2540-E 97	Gravimetric	TVS
Aqueous/Solids	SM 2540-G 97	Gravimetric	Total / Fixed Solids

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Matrix	Standard / Method	Technology	Analyte
Aqueous/Solids	SM 4100-00	IC	Bromide
Aqueous/Solids	SM 4100-00	IC	Chloride
Aqueous/Solids	SM 4100-00	IC	Fluoride
Aqueous/Solids	SM 4100-00	IC	Nitrate
Aqueous/Solids	SM 4100-00	IC	Nitrite
Aqueous/Solids	SM 4100-00	IC	Phosphate
Aqueous/Solids	SM 4100-00	IC	Sulfate
Aqueous/Solids	SM 4500-CI G-97	Colorimetric	Chloride
Aqueous/Solids	SM 4500-CN E-99	Colorimetric	Cyanide
Aqueous/Solids	SM 4500-F C-97	ISE	Fluoride
Aqueous/Solids	SM 4500-NH3 D-97	Colorimetric	Ammonia
Aqueous/Solids	SM 4500-NO3 I-00	Colorimetric	Nitrate
Aqueous/Solids	SM 4500-NO3 I-00	Colorimetric	Nitrite
Aqueous/Solids	SM 4500-NO3 I-00	Colorimetric	Nitrite + Nitrate
Aqueous/Solids	SM 4500-Norg D-97	Colorimetric	Total Kjedahl Nitrogen
Aqueous/Solids	SM 4500-S2 D-00	Colorimetric	Sulfide
Aqueous/Solids	SM 4500-SO4 G-97	Colorimetric	Sulfate
Aqueous/Solids	WA-EPH	GC-FID	Extractable Petroleum Hydrocarbons
Aqueous/Solids	WA-VPH	GC-FID/PID	Volatile Petroleum Hydrocarbons

Matrix	Standard / Method	Technology	Analyte
Aqueous	EPA 3005A	Acid Digestion	Digestion Prep Method
Aqueous	EPA 3010A	Acid Digestion	Digestion Prep Method
Aqueous	EPA 3510C	Sep Funnel	Extraction Prep Method
Aqueous	EPA 3520C	Liq-Liq	Extraction Prep Method
Aqueous	EPA 3020A	Acid Digestion	Digestion Prep Method
Aqueous	EPA 7470A	Acid Digestion	Digestion Prep Method
Solids	EPA 3540C	Soxhlet	Extraction Prep Method
Solids	EPA 3546	Microwave	Extraction Prep Method
Solids	EPA 3550C	Ultrasonic	Extraction Prep Method
Solids	EPA 7471A	Acid Digestion	Digestion Prep Method

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Matrix	Standard / Method	Technology	Analyte
Aqueous/Solids	EPA 1311	Leaching	Synthetic Precipitation Leaching Procedure
Aqueous/Solids	EPA 1312	Leaching	Toxicity Characteristic Leaching Procedure
Aqueous/Solids	EPA 3050B	Acid Digestion	Digestion Prep Method
Aqueous/Solids	EPA 3580A	Waste Dilution	Extraction Prep Method
Aqueous/Solids	EPA 3611B	Alumina	Extraction Cleanup Method
Aqueous/Solids	EPA 3620C	Florisil	Extraction Cleanup Method
Aqueous/Solids	EPA 3630B	Silica Gel	Extraction Cleanup Method
Aqueous/Solids	EPA 3640A	GPC	Extraction Cleanup Method
Aqueous/Solids	EPA 3650A	Acid / Base	Extraction Cleanup Method
Aqueous/Solids	EPA 3660B	Sulfur Cleanup	Extraction Cleanup Method
Aqueous/Solids	EPA 3665A	Sulfuric Acid	Extraction Cleanup Method
Aqueous/Solids	EPA 5030B	Purge & Trap	Extraction Prep Method
Aqueous/Solids	EPA 5035A	Methanol Extraction	Extraction Prep Method

APPENDIX F

Detailed Field Sampling Procedures

APPENDIX F
PROJECT-SPECIFIC SAMPLE COLLECTION AND EVALUATION PROCEDURES

Surface water and sediment sampling at the Former Derecktor Shipyard will be performed according to the SOPs and project-specific sampling procedures described below. The SOPs and field documents referred to below are included in Appendices A and B of this SAP, respectively.

Quality control (QC) samples will be collected as part of the investigation, including field duplicates and rinsate blanks. Samples will also be assigned on the chain-of-custody (COC) form for laboratory QC analyses. Worksheet #20 summarizes the QC samples that are to be collected for each matrix.

The sample locations are presented on Figure 11-2. Worksheet #18 presents the analytical groups for samples that will be collected at each sample location. Worksheet #19 presents the analytical methods, sample container types, preservative requirements, and the maximum holding times for the associated analyses.

Samples will be identified in accordance with the sample location identification system presented in Worksheet #27. All laboratory analytical samples will be kept on ice in coolers and will be shipped with appropriate QC samples. Samples will be handled and delivered in accordance with the COC procedures detailed in Worksheet #27.

1.0 SEDIMENT SAMPLING PROCEDURES

Sediment cores will be advanced to a maximum depth of four feet at 117 grid locations within the Former Derecktor Shipyard for sampling. At ten additional geophysical sample stations sediment cores will be advance to a minimum of 1 foot for physical evaluation, however, repeated attempts and additional depths may be needed for adequate volume or to find target intervals for physical testing, including cohesion and radioisotope tests.

The majority of cores will be advanced using a barge mounted vibracore device. There are a total of 17 cores that need to be collected from beneath Pier 1 and Pier 2. Due to access issues (low clearance) it is unlikely that a barge mounted vibracore device will be used at these

locations. It is anticipated that a first attempt will be made to collect cores utilizing a small boat and a hammer drill connected to as many rods as are necessary to reach the sediment. Attempts will be made to obtain 4 ft of recovery. If the core barrel is unable to penetrate to a depth of at least 3 ft into the sediment using the hammer drill from a small boat the project team will consider alternative methods. One possibility would be to collect sediment from the pier platform by cutting holes in the deck at the sample locations. Once holes have been cut sediment could be collected using either a conventional drill rig, or a vibracore device from the pier deck.

If refusals or low recoveries are encountered, the sample location will be moved within the sample square and up to 4 attempts will be made to reach the desired depth or to obtain better recovery (see below). An estimated 345 grid surface and subsurface sediment samples will be collected for laboratory analysis of HMW PAHs, PCBs, lead and TBT. Select samples will also be analyzed for one or a combination of the following: asbestos, copper, zinc and TOC. Worksheet #18 presents sample location, interval and analysis for all sediment samples. An estimated ten samples will be collected for grain size analysis, at geophysical sample stations. Sediment coring will be performed by a subcontractor according to the procedures in SOP GH-1.3.

Sediment samples will be collected for laboratory analysis from the sediment cores in accordance with SOP SA-1.3. Samples for chemical analysis will be collected from the 0-1 foot, 1-2 foot, and 2-4 foot intervals at all grid locations if recovery permits. At geophysical stations samples will be collected from the 0-1 foot interval initially. Sediment sample log sheets will be completed for each sample that will include date and time of collection, a description of the material, analysis requested and any observations made by the field team such as visual evidence of contamination or any anomalies.

Sediment samples for cohesion testing will be collected from the maximum depth allowed by a gravity core device. Sediment samples for radioisotope analysis will be collected from intervals selected by the Project Geologist, based on presence of layering evident, estimated cohesion and other physical characteristics. The model for determining the sediment accumulation rate is presented below in Section 3.0.

<u>Sediment Sampling Procedures</u>

1. Record all required data on the sample log sheet. Include the sampling equipment, sampling personnel, date, time, depth of sample, recovery, depth of water to bottom, and sample analyses for that station.

2. Label appropriate sample jars with the sample location number, sampler's name, date, and analytical fractions.

3. Acquire the core tube from the drilling contractor. Divide the core into four equal portions vertically; isolate the top portion, the second portion, and then the bottom two portions together, to make up the three sample intervals.

4. Transfer each portion of sediment from the target interval of the acetate core tube to a decontaminated stainless-steel bowl using only decontaminated stainless steel trowels, and homogenize the sample once observations including sediment description, evidence of contamination and anomalies have been recorded on the sample log sheet.

5. Remove any large particles such as shell hash, gravel or artificial fill too large to be sent for analysis. Note the removal of material and types of material removed on the sample log sheet.

6. Homogenize the remaining material so that it is representative of the whole vertical sample interval. Fill the appropriate sample containers with the homogenized sediment.

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

8. Ensure that the samples are properly labeled with location, parameter, date, and samplers name. Place each container within a ziplock bag, and immediately place in coolers with ice. Complete the chain-of-custody in accordance with procedures described in Worksheet 27. Package and ship the sample coolers to the appropriate laboratory for overnight delivery.

9. Decontaminate the sampling equipment before reuse.

Care should be taken in handling all sediment samples to ensure that the exterior of the sample containers are clean and free of sediment before shipping to the laboratory.

1.1 ADDRESSING POOR RECOVERY

If refusal is encountered (identified by lack of penetration), the sample team will identify the intervals that were recovered and only those intervals will be sampled from that location. If cores are successfully taken to only a portion of the lower interval, the sample log sheet will be appropriately noted.

Accuracy of the sample depths is important to determine contaminant presence in the different sample depth intervals. However, there is always some compression of fine sediments during coring. To maximize the vertical accuracy of the samples, a target recovery of 75% of core barrel penetration at each location will first be sought (at least three feet of sediment recovered from a four foot core run). If this first target is not reached, the station will be cored three additional times. If any of the four cores provide more than 60% recovery of the total penetration (approximately 2.5 feet of recovery), the core with the highest recovery will be retained and sampled, assumed to represent the entire length of the core run with uniform compaction across the four foot vertical interval. The core will be divided into three portions to represent the intervals described above.

If there is no recovery (less than two inches) at a location, contact the project manager for permission to relocate to another position within the target area. In the event that poor recovery (<60%, but more than two inches) is encountered at a grid location after 4 attempts have been made, the following steps will be taken:

1) Drive one core to 0-1 foot, recover core. If no obstructions are present, assume that the entire core is representative of the 0-1 foot interval. If less than two inches of material is present in the core, assume there was an obstruction that was driven ahead of the drive point, and no sample is acquired. Contact the project manager to relocate to another position.

2) Shift position slightly and drive a second core tube to 2 feet. Recover the core. If the recovery is at least 150% of the previous core, assume uniform compaction and that the lower portion of the core tube contains material representative of the 1-2 foot interval, using the previous core recovery as a guideline for the surface interval. Discard the surface material from this core. If the recovery is less than 150% the previous core, assume no recovery from that interval as a result of obstruction.

3) Shift position again and drive a third core tube to 4 feet. Recover the core. If the recovery is at least 150% of the previous core driven to 2 feet, assume that the lower 50% of the core tube contains material representative of the 2-4 foot interval, and sample that material. If the recovery is less than 150% of the previous core, assume no recovery from that interval based on obstructions. Retain this core and move to step 4.

4) Remove all the contents and evaluate them for obstructions. Note lithology on the sample log sheet and notify the project manager of the poor recovery.

Samples will be labeled in accordance with Worksheet #27 of the SAP regardless of recovery. For example, if only 40 inches of sediment are recovered and samples are collected from the 0-10 inch, 10-20 inch and 20-40 inch intervals, samples will still be labeled with 0-12 inch, 12-24 inch and 24-48 inch nomenclature to identify the interval where they came from. Field log sheets will provide details regarding recovery and actual sample intervals.

2.0 SURFACE WATER SAMPLING PROCEDURE

Surface water samples will be collected at each geophysical location to aid in the analysis of the ADCP data. Water samples will be collected from near the sediment surface for TSS analysis so as to provide a baseline that will be used to provide a baseline of “backscatter” for interpretation of data collected by the ADCP recorders.

Surface water samples will be collected using a Kemmerer water sampler to ensure that only water from the desired depth is collected. Water will be sampled using the following procedure:

1. Record all required data on the sample log sheet. Include the sampling equipment, sampling personnel, date, time, depth of sample, recovery, depth of water to bottom, and sample analyses for that station.

2. Label appropriate sample jars with the sample location number, sampler's name, date, and analytical fractions.

3. Pre-measure rope on Kemmerer Sampler to ensure that the desired depth (approximately two feet above sediment – water interface) is reached when lowering the sampler into the water column.

4. Open up the Kemmerer Sampler and lower it down to the desired depth making sure to keep the messenger weight above the water surface. Once the sampler reaches the appropriate depth release the messenger weight and wait for the snap, indicating that the sampler is closed.

5. Retrieve the sampler and fill appropriate sample jar(s)

Samples will be placed on ice and proper chain of custody procedures, as described in WS #27, will be followed.

3.0 MODEL FOR DETERMINATION OF SEDIMENT ACCUMULATION RATE

Sediment accumulation (deposition rate) will be modeled based on measured activity of lead 210 radioisotopes. This section describes how the measured Pb-210 activity is used to create this estimated rate.

The Pb-210 (Po-210) activity reported by the laboratory will be a total value comprising "supported" (background) and "unsupported" (from atmospheric deposition) Pb-210 activity. Supported activity, which exhibits relatively constant rates of decay, is expected to be found at the lower segments of the cores. The model for determining the sedimentation rate is based on unsupported activity. Therefore, activity from the lower segments of the cores (supported) will be subtracted from the laboratory (total) Pb-210 activity to obtain the unsupported, or "excess", Pb-210 activity. In addition, the laboratory results will be converted from units of picocuries per gram (pCi/g) to disintegrations per minute per gram (dpm/g) by multiplying by a conversion factor of 2.22 dpm/pCi to be consistent with units in the model.

Initially it will be assumed that the flux of lead-210 to the sediment is constant, but that the sediment supply rate may vary over time. Consistent with these assumptions, a constant rate of supply (CRS) model (Appleby & Oldfield, 1978) will be applied. This model calculates the age of a given depth in a core by comparing the cumulative excess Po-210 activity below that core depth (dpm/cm^2) to the total excess Po-210 activity in the core (Flett, 2009).

The density of each sediment sample will be used to calculate mass sedimentation at selected depths within the core (Pizzolato and De Hon, 1995). Excess Pb-210 (Po-210) activity will then be plotted on a logarithmic scale (x-axis) against the mass sedimentation at the sample depth in the core from which the samples were collected (y-axis). A fitted line sloping from the top right corner of the plot to the bottom left would represent Pb-210 activity decreasing with greater depth (and greater age) of deposited sediment. If the top section of a core has a near-vertical plot (similar activity for different depths), it will be assumed that this represents an area of mixing of older and newer sediment, and this horizon will be omitted in calculating the sedimentation rates. Numerical integration of this Pb-210 activity profile will be performed to obtain the core activity values needed to calculate sediment layer age. The following formulas (Lubis, 2006) will be used to calculate age and sedimentation rate:

The cumulative residual excess Pb-210 activity, *A* beneath sediments of age t varies according to the formula:

$$A = A(o)e^{-kt}$$

where *A*(*o*) is the total residual excess Pb-210 activity in the sediment column and k is the Pb-210 radioactive decay constant:

$k = \ln(2)/T_{1/2}$, where $T_{1/2}$ is the half-life of Pb-210. (k = 0.03114/year)

A and *A*(*o*) are calculated by direct numerical integration of the Pb-210 profile. The age of sediments of depth x is then given by:

$$t = 1/k * \ln (A(o)/A)$$

The sedimentation rates can be shown to be given directly by the formula:

$$R=kA/C$$

where C is the excess Pb-210 activity at a given depth.