Remedial Investigation
Quality Assurance Project Plan
Version: Final

Gould Island Site (D01RI033800)
Narragansett Bay
Jamestown, Rhode Island

Prepared for:
U.S. Army Corps of Engineers
New England District
696 Virginia Road
Concord, MA 01742

US Army Corps of Engineers®

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Contract W912WJ-16-D-007
Task Order W912WJ-17-F-0039

Prepared by:
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LIST OF ACRONYMS

ABB – ABB Environmental Services
Alion – Alion Science and Technology
AOC – area of concern
APP – Accident Prevention Plan
ARA – Absolute Resource Associates
AST – aboveground storage tank
BFB – bromofluorobenzene
bgs – below ground surface
CCB – continuing calibration blank
CCV – continual calibration verification
CDAP – Chemical Data Acquisition Plan
CENAE – Corps of Engineers New England District
CERCLA – Comprehensive Environmental Response, Compensation, and Liability Act
CG – Certified Geologist
COPC – contaminant of potential concern
Credere – Credere Associates, LLC
CVAA – cold vapor atomic absorption
°C – degrees Celsius
DERP – Defense Environmental Restoration Program
DO – dissolved oxygen
DoD – Department of Defense
DoN – Department of the Navy
DFTPP – decafluorotriphenylphosphine
DI - deionized
DQO – data quality objective
DU – decision unit
DUA – data usability assessment
EB – equipment blank
ECD – electron capture detector
EDD – electronic data deliverable
ELAP – Environmental Laboratory Accreditation Program
EPA – U.S. Environmental Protection Agency
EPH – extractable petroleum hydrocarbons
ERA – Ecological Risk Assessment
FB – field blank
FD – field duplicate
FID – flame ionization detector
FS – Feasibility Study
FSP – Field Sampling Plan
FUDS – Formerly Used Defense Sites
GC – gas chromatograph/chromatography
GIS – Geographic Information System
GPR – ground penetrating radar
GPS – global positioning system
GWO – groundwater objective
HCl – hydrochloric acid

HDPE – high density polyethylene
HHRA – Human Health Risk Assessment
HNO_3 – nitric acid
HPLC – high performance liquid chromatography
HRG – Hager-Richter Geosciences
HRS – Hazard Ranking System
HTRW – Hazardous, Toxic, and Radioactive Waste
IAS – Initial Assessment Study
ICAL – initial calibration
ICP – inductively coupled plasma
ICS – interference check sample (ICSA = solution A, ICSAB = solution AB)
ICV – initial calibration verification
IS – internal standard
ISC – instrument sensitivity check
ISM – Incremental Sampling Methodology
LC – liquid chromatography
LCS – laboratory control sample
LCSD – laboratory control sample duplicate
LDC – Laboratory Data Consultants
LHA – Lifetime Health Advisory
LOD – limit of detection
LOQ – limit of quantification
LSP – Licensed Site Professional
MADEP – Massachusetts Department of Environmental Protection
MC – munitions constituent
MCL – Maximum Contaminant Level
MCP – Massachusetts Contingency Plan
µg/l – microgram per liter
MEC – munitions and explosives of concern
mg/kg – milligrams per kilogram
mg/L – milligrams per liter
mL - milliliter
MMRP – Military Munitions Response Program
MS – mass spectrometry
MS/MSD – matrix spike/matrix spike duplicate
NCP - National Oil and Hazardous Substances Pollution Contingency Plan
NDAI – No Department of Defense Action Indicated
NE – not established
NED – New England District
NETC – Naval Education and Training Center
NFG – National Functional Guidelines
$(NH_4)_2SO_4$ – ammonia sulfate
OEW – ordnance and explosive waste
ORP – oxidation reduction potential
oz - ounce
PAH – polyaromatic hydrocarbon
PAL – Project Action Limit
PAOI – potential area of interest
PCB – polychlorinated biphenyls

PE – Professional Engineer
PEC – probable effect concentration
PG – Professional Geologist
PID – photoionization detector
PQL – project quantitation limit
PP – priority pollutant
PWS – Performance Work Statement
QAPP – Quality Assurance Project Plan
QC – quality control
QSM – Quality System Manual
%R – percent recovery
RAB – Restoration Advisory Board
RCRA – Resources Conservation and Recovery Act
RI – Remedial Investigation
RIDEM – Rhode Island Department of Environmental Management
RL – reporting limit
RPD – relative percent difference
RRT – relative retention time
RSD – relative standard deviation
RSL – regional screening levels
RT – retention time
RTC – response to comments
SEDD – Staged Electronic Data Deliverable
SHM – Safety and Health Manager
SIM – selective ion monitoring
SM – standard methods
SOP – standard operating procedure
SOW – scope of work
SSHO – Site Safety and Health Officer
SSHP – Site Safety and Health Plan
SSWP – Site-Specific Work Plan
SVOC – semi-volatile organic compound
SWEC – Stone & Webster Environmental Corporation
SWETS – Stone & Webster Environmental Technology Services
TAS – Test America Sacramento
TB – trip blank
TCRA – Time Critical Removal Action
TEC – threshold effect concentration
THQ – target hazard quotient
TIC – tentatively identified compound
TPH – total petroleum hydrocarbons
TSA – technical systems audit
UFP – Uniform Federal Policy
USACE – U.S. Army Corps of Engineers
UST – underground storage tank
UXO – unexploded ordnance
UV – ultra violet
VOA – volatile organic analysis
VOC – volatile organic compound

VPH – volatile petroleum hydrocarbons
YSI – Yellow Springs Instruments

INTRODUCTION

Credere Associates, LLC (Credere) was retained by the U.S. Army Corps of Engineers (USACE) New England District (CENAE) to prepare this Quality Assurance Project Plan (QAPP) and associated Field Sampling Plan (FSP), which is included as **Appendix A**, for the Remedial Investigation (RI) of the Gould Island Site in Narragansett Bay located in Jamestown, Newport County, Rhode Island (Site). A Site Location Plan is provided as **Figure 1**, and a Detailed Site Plan is provided as **Figure 2**.

This QAPP has been prepared in accordance with the Uniform Federal Policy (UFP) for Quality Assurance Project Plans (Department of Defense [DoD] *et al*, 2012) and generally with the U.S. Environmental Protection Agency (EPA) Requirements for Quality Assurance Project Plans (EPA, 2001). This QAPP and FSP were also developed in accordance with the Performance Work Statement (PWS) prepared by CENAE and dated June 13, 2017 (CENAE, 2017). This work will be completed as a task order (Task Order No. W912WJ-17-F-0039) under the Hazardous, Toxic, and Radioactive Waste (HTRW) contract between the USACE and Credere (Contract No. W912WJ-16-D-007). The work required under the PWS falls under the Defense Environmental Restoration Program (DERP) Formerly Used Defense Sites (FUDS), which complies with the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) and the National Oil and Hazardous Substances Pollution Contingency Plan (NCP).

The scope of work includes assessment of potential source areas across the FUDS eligible portion of the island identified in the Final Technical Memorandum (Credere, 2017). The potential source areas were identified based on review of prior environmental reports, munitions response reports, and historical information. The Final Technical Memorandum's objective was to provide a concise description of prior work and preliminary data gap analysis to support a Project Coordination Meeting with CENAE and Rhode Island Department of Environmental Management (RIDEM) to concur on Areas of Concern (AOCs) for the RI and risk assessments needed for site closure. Based on this memorandum, the Project Coordination Meeting on August 30, 2017, and subsequent discussion with CENAE; 28 New England District (NED) Sites of the 48 potential source areas originally identified require further investigation. Referenced NED Site Nos. from an Engineering Evaluation of Contamination report from 1997 (SWETS, 1997) were used throughout this QAPP for consistency. The following NED Site Nos. require further investigation:

- NED Site No. 1 Coal storage north
- NED Site No. 4 Pump house
- NED Site No. 8 Incinerator #49
- NED Site No. 9 Magazine ignitor storage #37
- NED Site No. 10 Disposal Area #14
- NED Site No. 11 Quonset huts/maintenance shops
- NED Site No. 12 Maintenance shop/garage/fire station #39
- NED Site No. 13 Electric substation/transformer pen #43
- NED Site No. 16 Barracks
- NED Site No. 19 Torch pot storage #51
- NED Site No. 21 Bunker #11

- NED Site No. 22 Bunker #12
- NED Site No. 23 Coal storage south
- NED Site No. 24 Cable terminal building #16
- NED Site No. 27 Two gas pits
- NED Site No. 28 Ordnance test facility/hangar
- NED Site No. 30 Pyrotechnic storage
- NED Site No. 31 Gas pit
- NED Site No. 32 Drum storage area
- NED Site No. 33 Paint and oil storage #47
- NED Site No. 35 Boiler house #29
- NED Site No. 37 Miscellaneous storage #28
- NED Site No. 38 Torpedo storage #10
- NED Site No. 39 Well house #81
- NED Site No. 42 5,000-gallon aviation fuel tank/ordnance test facility, gasoline outlet #30
- NED Site No. 43 Boiler house and ordnance test facility paint shed #30
- NED Site No. 44 Empty drums at south end of concrete pad
- NED Site No. 48 Debris stockpile (added by Credere)

The proposed Site investigation data will be used to support a Human Health and Ecological Risk Assessment (HHRA/ERA) to be completed as part of a RI.

WORKSHEETS #1 & 2 – TITLE AND APPROVAL PAGE

PROJECT IDENTIFYING INFORMATION:
Gould Island Site
Narragansett Bay, Jamestown, Rhode Island
D01RI033800

LEAD ORGANIZATION:
CENAE
696 Virginia Road
Concord, Massachusetts 01742
(978) 318-8238

CONTRACTOR:
Credere
776 Main Street, Westbrook, Maine 04092
(207) 828-1272
Contract No.: W912WJ-16-D-007
Task Order No.: W912WJ-16-F-0039

Federal Regulatory Agency:

16-April-2018
Erik Patton Date
USACE Project Manager

16-April-2018
Yixian Zhang Date
USACE Quality Manager

State Regulatory Agency:

Nicholas Noons Date
RIDEM Waste Management – Site Remediation

Other Stakeholders:

Rip Patten 4-16-2018
Rip Patten Date
Credere Associates, LLC Program Manager

Theresa Patten 4-16-2018
Theresa Patten Date
Credere Associates, LLC Quality Control (QC) Manager

A list of relevant plans and reports for prior investigations is included in the **Reference** Section of this document.

WORKSHEETS #3 & 5 – PROJECT ORGANIZATION AND QAPP DISTRIBUTION

A Project Organization Flow Chart is included as **Figure 3**. QAPP recipients who are required to be notified of changes are indicated with an *.

The following stakeholders will be issued a copy of this QAPP according to the below table:

Stakeholder	Number of Copies	Address
CENAE*	One (1) hard copy	CENAE-EDG Attn: Erik Patton 696 Virginia Road Concord, MA 01742-2751
RIDEM Office of Waste Management*	One (1) hard copy	Office of Waste Management Attn: Nicholas Noons, Project Manager 235 Promenade Street Providence, RI 02908
RIDEM Department of Fish & Wildlife	One (1) hard copy	Department of Fish & Wildlife Attn: Tanner Steeves 235 Promenade Street Providence, RI 02908
Restoration Advisory Board (RAB)	Electronic (CD)	Jamestown Philomenian Library Gould Island File 26 North Road Jamestown, RI 02835
Credere	Electronic	Project file
Absolute Resource Associates (ARA)*	Electronic	aarond@absoluteresourceassociates.com
Lab Data Consultants (LDC)*	Electronic	Via LDC Advantage

WORKSHEET #4, 7 & 8 – PERSONNEL QUALIFICATIONS AND SIGN-OFF SHEET

The following table summarizes project personnel, their roles, and qualifications.

Name	Role/Title	Organization	Qualifications	Signature/ Date
Rip Patten, PE, LSP	Program Manager, Vice President, Engineer	Credere	Bachelors of Science in Environmental Engineering, Professional Engineer (PE), 23 years' experience	See Worksheet #1 & 2
Theresa Patten, PE	QC Manager, President, Engineer	Credere	Bachelors of Science and Masters in Civil Engineering, Professional Engineer (PE), 19 years' experience	
Sean McNamara, Ph.D.	Project Manager, Project Engineer	Credere	Bachelors of Science in Environmental Engineering, Masters in Civil Engineering, Doctorate in Civil/Environmental Engineering, 8 years' experience	NA
Allison Drouin, PG, CG	Technical Lead, Geologist Alternate Field QC Manager, Alternate Site Safety and Health Officer (SSHO)	Credere	Bachelors of Science in Geology/Environmental Studies, Professional/Certified Geologist (PG/CG), 8 years' experience	NA
Judd Newcomb, PG, CG	Alternate Field QC Manager, Alternate SSHO, Geologist	Credere	Bachelors of Science in Geology, Professional/Certified Geologist (PG/CG), 17 years' experience	NA
Rick Vandenberg, CG, PG	Alternate Field QC Manager, Alternate SSHO, Hydrologist	Credere	Bachelors of Arts in Geology/Chemistry, Professional/Certified Geologist (PG/CG), 29 years' experience	NA
Sean Gannon	Alternate SSHO, Geologist	Credere	Bachelors of Science in Geology/Environmental Studies, 2 years' experience	NA
Stacy Towne	Alternate SSHO, Field Alternate, Technician	Credere	Bachelors of Arts in Environmental Science and Policy, Masters in Geographic Information Systems (GIS), 4 years' experience	NA
Matthew Kennedy	Field QC Manager, SSHO	Credere	Bachelors of Science in Environmental Science, 3 years' experience	NA
Mark Willis	Safety and Health Manager (SHM), Industrial Hygienist	Credere	Bachelors or Science in Environmental Safety and Health, 8 years' experience	NA
Cynthia Fuller	Lead for Risk Assessment	Sovereign Consulting, Inc. (Sovereign)	Bachelors of Science in Pharmacy and Allied Health Professions, Masters of Public Health in Environmental and Occupational Health Sciences; 25+years' experience	NA
Aaron Dewees	Laboratory QC Officer, Chemist	ARA	Bachelors of Science in General Biology, 18 years' experience	NA
Shauna McKellar	Data Validator, Chemist	LDC	Bachelors of Science in Environmental Toxicology, 11 years' experience	NA

NA – no approval need.

WORKSHEET #6 – COMMUNICATION PATHWAYS

The following table summarizes specific issues that will trigger the need to communicate with other project personnel or stakeholders.

Communication Driver	Organization	Name	Contact Information	Procedures
Regulatory agency interface	USACE	Erik Patton	(978) 318-8051	Credere will direct correspondence for RIDEM to USACE as needed.
Field progress reports	Credere	Sean McNamara	(603) 817-5775	For this phase of the project Credere will provide an email work summary report at the completion of field activities, unless additional progress updates due to delays or unforeseen circumstances are required.
Stop work due to safety issue	Credere	Matthew Kennedy	(978) 578-6801	Field QC managers/personnel will communicate with the SHM for stop work issues
Suspected unexploded ordnance (UXO)	Credere	Matthew Kennedy	(978) 578-6801	Field QC manager/personnel communicate any suspected UXO to the Project Manager immediately after evacuating the area. The Project Manager will immediately notify USACE to coordinate next steps.
Public interface	USACE	Erik Patton	(978) 318-8051	Credere will direct public comments/questions during implementation of the project to USACE
QAPP changes prior to and during project execution	Credere	Theresa Patten	(207) 730-1053	QAPP changes prior to or during execution will be communicated between the indicated parties.
		Allison Drouin	(207) 749-1141	
		Sean McNamara	(603) 817-5775	
	USACE	Erik Patton	(978) 318-8051	
Field corrective action	Credere	Sean McNamara	(603) 817-5775	The Field QC Manager will coordinate with the Project Manager for field corrective action. The Project Manager will contact USACE if significant issues impacting the program arise.
		Matthew Kennedy	(978) 578-6801	

Communication Driver	Organization	Name	Contact Information	Procedures
Sample receipt variances	Credere	Allison Drouin	(207) 749-1141	The laboratory will communicate with the Technical Lead to rectify sample receipt variances.
Laboratory quality control variances				Laboratory quality control variances and corrective actions identified by the lab or validator will be reported to Allison Drouin. Ms. Drouin will communicate issues to USACE if the data quality objectives will be compromised.
Analytical corrective action				
Data verification issues				LDC will notify Allison Drouin to communicate verification and corrective actions to ARA. LDC may also communicate directly to the lab with permission from Credere.
Data review corrective actions				

WORKSHEET #9 – PROJECT PLANNING SESSION SUMMARY

The following summarizes the project planning sessions completed to support preparation of this QAPP.

Date of Planning Session: August 30, 2017
Location: RIDEM, 235 Promenade Street, Providence, Rhode Island
Purpose: Project team meeting to consider basic scope, review background, and logistics

Name	Organization	Title/Role	Email/Phone
Rip Patten	Credere	Engineer/Program Manager	rpatten@crederellc.com (207) 730-1039
Sean McNamara	Credere	Project Manager	smcnamara@crederellc.com (603) 817-5775
Allison Drouin	Credere	Geologist/Technical Lead	adrouin@crederellc.com (207) 749-1141
Deborah Acone	USACE	Project Manager (acting)	Deborah.e.acone@usace.army.mil (978) 318-8130
Paul Young	USACE	Project Geologist	Paul.j.young@usace.army.mil (978) 318-8597
Michael Narcisi	USACE	Environmental Reviewer	Michael.j.narcisi@usace.army.mil (978) 318-8454
Richard Gottlieb	RIDEM	DoD Program Manager	Richard.gottlieb@dem.ri.gov (401) 222-2797 x7138
Nicholas Noons	RIDEM	Project Manager	Nicholas.noons@dem.ri.gov (401) 222-2797 x7517
Shawn Lowry	RIDEM	Assistant Project Manager	Shawn.lowry@dem.ri.gov (401) 222-2797 x7142
Tanner Steeves	RIDEM	Division of Fish & Wildlife representative	Tanner.steeves@dem.ri.gov (401) 789-0281

Notes/Comments: The Site background was presented by Allison Drouin based on a thorough review of the prior reports, as was presented in the Draft Technical Memorandum (Credere, 2017). The general scope of work (SOW) was presented including contaminants of potential concern (COPC), media to be assessed, and methods for assessment. It was agreed that the previously removed underground storage tanks (USTs) would not be further assessed as they were the responsibility of the Navy and, therefore, not eligible for investigation under the FUDS program. Of greatest importance was the discussion of logistics and schedule with regard to natural resources and nesting birds. RIDEM Fish & Wildlife policy dictates island access is restricted from April 1 to August 15 for nesting. It was agreed that Credere may access the island after the April 1st date to perform low-disturbance sampling, if needed (e.g., hand tool sampling). RIDEM Fish & wildlife also indicated they would have no restrictions on the clearing for access to NED sites as long as it occurred outside the restricted time period.

Consensus decisions made: Based on the discussions of the meeting, it was decided to pursue Site clearing in the fall of 2017 and to target field work for late-February and March 2018. No concerns were expressed by RIDEM regarding the direction of scope of work for the Site at the time.

Action items:

Action	Responsible Party	Due Date
Continue to revise the Technical Memorandum	Sean McNamara	September 13, 2017
Begin drafting QAPP	Allison Drouin	September 29, 2017

Date of Planning Session: September 21, 2017
Location: Credere Office, 776 Main Street, Westbrook, Maine
Purpose: Internally review Credere's planned SOW included herein

Name	Organization	Title/Role	Email/Phone
Rip Patten	Credere	Engineer/Program Manager	rpatten@crederellc.com (207) 730-1039
Sean McNamara	Credere	Project Manager	smcnamara@crederellc.com (603) 817-5775
Allison Drouin	Credere	Geologist/Technical Lead	adrouin@crederellc.com (207) 749-1141
Judd Newcomb	Credere	Geologist/Field QC Manager Alternate	jnewcomb@crederellc.com (207) 232-5387
Sean Gannon	Credere	Geologist/Field Personnel	sgannon@crederellc.com (860) 733-2510

Notes/Comments: Discussions focused on refining the SOW outline in this QAPP. Specific discussions focused on Incremental Sampling Methodology (ISM) decision units (DUs), ensuring sufficient data was collected for use in multiple risk assessment methods, and the need for discreet samples in addition to the ISM samples.

Consensus decisions made: Scope outlined herein.

Action items:

Action	Responsible Party	Due Date
QAPP Draft with Field Sampling Plan submittal	Credere	September 29, 2017

Date of Planning Session: September 27, 2017
Location: Credere Office, 776 Main Street, Westbrook, Maine teleconference
Purpose: Review Credere's scope of work with Risk Assessment team

Name	Organization	Title/Role	Email/Phone
Rip Patten	Credere	Engineer/Program Manager	rpatten@crederellc.com (207) 730-1039
Sean McNamara	Credere	Project Manager	smcnamara@crederellc.com (603) 817-5775
Allison Drouin	Credere	Geologist/Technical Lead	adrouin@crederellc.com (207) 749-1141
Cynthia Fuller	Sovereign	Risk Assessor	cfuller@sovcon.com (401) 323-9571
Rachel Leary	Sovereign	Engineer/Project Manager	rleary@sovcon.com (413) 540-0650

Notes/Comments: Credere presented the draft SOW to Sovereign and Ms. Fuller had reviewed the scope for consistency with her general experience in Rhode Island and for her risk assessment needs. Primary topics of discussion included the following:

1. Undesirable nature of ISM sampling for use in a risk assessment. Cynthia recommended discrete sampling in addition to the ISM.
2. Sediment depth intervals for use in an ecological risk assessment should be approximately 10 to 15 cm as opposed to 0-2 feet. Credere agreed to add the shallower samples and reduce the number of 0-2 foot samples. The 0-2 foot samples were still desirable to attempt to delineate historical (i.e., deeper) contamination and the possible extent of tar in Disposal Area 14 (NED Site 10).
3. Adding hexavalent chromium to the COPC list given the previous documentation of chromium at the Site.
4. Expanding the metals list to be more inclusive of metals of concern when doing ecological risk assessment.
5. Use of RIDEM standards versus the need to use CERCLA criteria.
6. Improvement of the usability of petroleum data in the risk assessment by analyzing for extractable petroleum hydrocarbons (EPH)/volatile petroleum hydrocarbons (VPH) instead of total petroleum hydrocarbons (TPH).
7. Addition of samples for arsenic from locations better positioned to represent background concentrations than the previous background samples collected.
8. Addition of pesticide soil analyses at the Disposal Area #14 AOC.

Consensus decisions made: Credere relied on the risk assessment expertise and made adjustment to the scope accordingly. All agreed the use of CERCLA criteria for COPCs, with the exception of petroleum, which falls under RIDEM standards.

Action items:

Action	Responsible Party	Due Date
QAPP Draft with Field Sampling Plan submittal	Credere	October 5, 2017

WORKSHEET #10 – CONCEPTUAL SITE MODEL

SITE DESCRIPTION

The 39.15-acre Site is the southern portion of Gould Island that has been assessed under the DERP and is eligible for funding as a FUDS. The Site is currently owned by the RIDEM Division of Fish & Wildlife and is designated a bird sanctuary. The island is currently inaccessible during bird nesting between April 1st and August 15th. The 16.9-acre remainder of the island to the north is still owned by the Navy. Additionally, within the FUDS Site boundary, four areas are retained by the Navy or Coast Guard, including the Keeper's House and lighthouse, water treatment plant #42 (NED Site No. 18), the water tank #31 (NED Site No. 47), the transformer vault #25A (NED Site No. 17), and the 100-foot x 100-foot area around the beacon at the southern end of the island. These areas are not FUDS eligible and are not considered part of the Site.

The Site is currently heavily overgrown with remnants of dilapidated buildings, relict foundations, and bunkers that were formerly used in the storage, testing, and repair of torpedoes, research, housing, and infrastructure (e.g., power, heating, waste disposal). The southernmost point of the island has a large concrete former aviation pad and two dilapidated seaplane ramps (Alion Science and Technology [Alion], 2009). The Site is reportedly accessible from these ramps with use of a ramp barge or shallow landing craft.

PHYSICAL SETTING

The Site is densely vegetated with young trees and abundant vines and shrub. Substantial vegetation clearing is required prior to implementation of the RI. Prior aerial photographs and surveyed topography indicate large portions of the western side of the island were built up with steeply sloping areas down to the historical roads.

Generally, overburden at the Site was previously described to be sand with varying amounts of silt and gravel. Overburden was reported to be thin along the westernmost side of the island, particularly in areas of bedrock outcropping and as thick as 10 feet elsewhere on the eastern side of the island. Investigation was limited in the central built up portion of the island; therefore, thickness in this area is not yet known. Groundwater recharge is considered minimal due to the island nature with most precipitation discharging to the bay. Overburden groundwater is present only in the thickest areas of overburden on the eastern side of the island and possibly in only perched lenses and in the bedrock in the western portion of the island (SWETS, 1997).

According to drilling reports from prior investigations at the Navy northern portion of the island, bedrock is relatively soft and was easily cored with diamond bit coring equipment and required screens for well construction to avoid cave-in. Water bearing fractures or specific zones were not encountered during drilling of bedrock monitoring wells at the Navy portion of the island; however, bedrock wells were installed with 30 feet of screen 30 feet into bedrock and yielded groundwater.

SITE HISTORY

Beginning in 1887, a lighthouse was constructed for Narragansett Bay on the eastern side of the island (RIHPHC, 1995), and the island was otherwise used privately for residential purposes. Between 1918 and 1920, the island was seized by the United States for use by the U.S. Navy and Marines. Initial construction included adaptation of the existing residence for marine barracks, air hangars, a water tower and infrastructure, a personnel pier at the northern end of the island, a concrete torpedo pier at the southeast point of the island, a torpedo storage building, two warhead bunkers (later converted to a firing range and storage), the south powerhouse, and a railroad network for moving heavy equipment throughout the island. Torpedoes were test fired from seaplanes staged at the southern end of the Site and from an off-island barge. Test torpedoes were unarmed and used to test guidance and launching techniques only.

In the 1940s, additional buildings were rapidly constructed to support World War II including additional hangars, a second southern seaplane ramp, torpedo overhaul building, power station, additional barracks, Quonset huts, degaussing buildings, and the firing pier in the northern offsite portion of the island. Warheads were transferred to Prudence Island in 1941 due to safe distance limitations, and a pistol range was constructed at former Bunker 11. By 1943, the firing pier was in operation and approximately 65,000 unarmed torpedoes were tested from the firing pier, barges, and submarines by the end of World War II.

In the 1950s, testing and production of torpedoes was outsourced to private contractors (Envirodyne, 1983), and in 1975 and 1989, parcels were transferred to the State of Rhode Island to form the current 39.15-acre Site. The Site has been generally abandoned since and is heavily overgrown with vegetation. Public access to the Site is by permit only, and the Site has been designated as a bird sanctuary.

ENVIRONMENTAL HISTORY

Initial Assessment Study, Envirodyne Engineers, Inc., March 1983 (Envirodyne, 1983)

In 1983, an Initial Assessment Study (IAS) was completed for the Naval Education and Training Center (NETC) in Newport, Rhode Island, which included assessment of 16 potentially contaminated sites including Gould Island. The assessment identified the Gould Island Disposal Area 14, Bunker 11, and the offsite electroplating shop (not part of this FUDS project) as requiring further action.

In Disposal Area 14 (NED Site No. 10), approximately 200 yards of the western shoreline of the island was used to dispose of domestic trash, metal scrap, wood, pipes, rusted drums, diesel oil tanks, concrete blocks, electroplating wastes, and degreasing wastes (solvents). Approximately 20 full drums were also noted at the time. Specific chemicals would have included muriatic acid, chromic acid, copper cyanide, sodium cyanide, sodium hydroxide, nickel sulfate, and anodex cleaner associated with electroplating operations. Reportedly, no wastes were transported back to Aquidneck Island from Gould Island, indicating all waste was disposed onsite. Due to the proximity to the bay and tidal action on the beach, these materials could have migrated and may have been transported out into the bay.

In Bunker 11 (NED Site No. 21), at least 10 unlabeled drums and other possible buried drums beneath the collapsed room were present. Surface drainage from this area discharged directly to the bay.

Based on the above, the following was recommended as part of a Confirmation Study:

- Collect four sediment samples plus two background samples to 3 feet below the bay bed in approximately 20 feet of water along Disposal Area 14 and analyze for polychlorinated biphenyls (PCBs) and metals (cadmium, chromium, copper, lead, mercury, nickel, and silver)
- Sample the contents of the drums in Disposal Area 14 in accordance with Resource Conservation and Recovery Act (RCRA)/RIDEM regulations for disposal
- Characterize the contents of the drums in Bunker 11 in accordance with RCRA/RIDEM regulations for disposal and properly dispose offsite

Investigation and Removal of Hazardous Waste on Gould Island, July 1983 (RIDEM, 1983a and 1983b)

In July 1983, RIDEM completed a Site inspection and identified numerous drums and other containers throughout the island for disposal (RIDEM, 1983a). On July 19, 1983, numerous drums of waste oil, solvents, paint sludge and caustics; 5-gallon containers of paint, grease, solvents, two diesel aboveground storage tanks (ASTs) and transformers were removed from the island (RIDEM, 1983b). Additionally, numerous acetylene, propane, and air cylinders were removed.

Confirmation Study Report, May 15, 1986 (Loureiro, 1986)

The Confirmation Study was conducted to assess the extent of previously identified impacts in Disposal Area 14 (NED Site No. 10) extending into sediment within the bay. Sediment and mussels were sampled for PCBs and metals. Results indicated elevated levels of metals – particularly lead, chromium, nickel, and copper – in near-shore sediments, and copper and lead in mussels above control sample results.

UST Removals,1989-1990

On February 13, 1989, the Department of the Navy (DoN) notified RIDEM of the planned removal of the following USTs from the Site (DoN, 1989a):

- Unknown volume tank NUSC 1 from Building 23 (gas pump house, NED Site No. 26)
- Unknown volume tank NUSC 2 from Building 23 (gas pump house, NED Site No. 26)
- 5,000-gallon steel tank from Building 39 (maintenance, garage, and fire station, NED Site No. 12)

The letter also included notification of removal for seven other USTs from the offsite Navy portion of Gould Island that are not discussed further. RIDEM responded with a letter dated March 1, 1989, indicating the need for a closure application, a sampling plan, and compliance

with the Division of Air and Hazardous Materials (RIDEM, 1989), which DoN provided on September 19, 1989 (DoN, 1989b).

According to a field investigation report prepared by RIDEM (RIDEM, 1990), the tanks were removed. RIDEM collected samples from three monitoring wells at that time; however, no results were available for review (it is not known if these wells were onsite or associated with the offsite USTs on the Navy parcel). Historical correspondence indicates the closure reports prepared in accordance with RIDEM requirements were completed; however, these reports were used during litigation in Philadelphia, PA, and were never returned to the permanent record file. These reports have not been located, but will be required to confirm the USTs were removed according to RIDEM requirements and that no residual contamination remained.

As these tanks were previously removed by the Navy, contamination associated with these tanks is not eligible for assessment under FUDS.

Chemical Data Acquisition Plan (ABB/SWEC, 1994), November 1994, and Engineering Evaluation of Contamination (SWETS, 1997), July 1997

In 1994, Stone and Webster Environmental Corporation (SWEC) reviewed available records for the Site with USACE, the NETC, and RIDEM, as well as historical aerial photographs and Site plans. Based on this review, SWEC identified three primary APCs in their Chemical Data Acquisition Plan (CDAP): Disposal Area 14, Bunker 11, and the offsite electroplating shop (located offsite on the Navy parcel and not considered part of the FUDS Site). The plan also identified 47 total onsite areas to be assessed as part of an Engineering Evaluation of Contamination.

In December 1994, SWEC and ABB Environmental Services (ABB) conducted a visual Site inspection, geophysical magnetometry survey, and surface/near surface soil sampling. The investigation identified a UST, 55-gallon drums and smaller chemical containers, transformers, remnant torpedo parts, compressed gas tanks, universal waste, residual coal, and general debris from former structures. Soil sampling results indicated COPC that were detected above EPA and/or RIDEM soil screening criteria (not applicable to FUDS sites, except in the case of certain UST related assessment) were polycyclic aromatic compounds (PAH), arsenic, lead, chromium, nickel, TPH, and PCBs.

Ordnance and Explosive Waste Archives Search Report, July 1995 (USACE, 1995)

In 1994, a historical records search and Site inspection was conducted under DERP to evaluate the Site for any ordnance and explosive waste (OEW). Sub-areas of the Site were classified into three categories with respect to OEW: confirmed, potential, and uncontaminated. The findings from the visual inspection and archives review concluded the Site is considered uncontaminated for OEW with the exception of Disposal Area 14 (NED Site No. 10), which was categorized as having the potential for OEW. Disposal Area 14 was the primary dump location on Gould Island while in operation. Ordnance most likely to be disposed in this area were ignitors, 1.1-inch rounds, and pyrotechnics. A torpedo propeller section and shaft were visible, though, as stated previously, none of the torpedoes on the island were armed for testing. The former Ordnance Test Facility/Hanger (NED Site No. 28), Hangar 5496-61 (NED Site No. 29), and Pyrotechnic Storage Building area (NED Site No. 30) are considered uncontaminated as they were washed

out to sea in 1985 during Hurricane Bob and there is no visual evidence to indicate OEW. Any remaining spent casings from the pistol range at Bunker 11 are not considered OEW, but may represent an environmental concern for metals contamination in Site soils.

Draft Baseline Risk Assessment Workplan, March 1998 (SWETS, 1998a)

In 1998, Stone & Webster Envrionmental Technology & Services (SWETS) completed a Draft Baseline Risk Assessment Workplan and Site Health and Safety Plan to support the Phase II Engineering Evaluation of Contamination for the Site. The document presents proposed data gap sampling locations for human health and ecological risk assessment. Additionally, a Site Health and Safety Plan (SWETS, 1998b) and an Environmental Compliance Summary for Field Activities (SWETS, 1998c) were completed. Correspondence from June 24, 1998, indicated a significant scope modification due to Site constraints would occur. Based on review of records, this scope modification ***did not occur and the Phase II Engineering Evaluation of Contamination was never completed.*** This prior workplan served as the baseline for the scope outlined herein, which was adapted to represent current industry standards and new information.

Final Site Inspection Report for Gould Island, August 2009 (Alion, 2009)

Alion completed a Site Inspection (SI) in accordance with their Site-Specific Work Plan (SSWP) Addendum (Alion, 2008) dated November 2008. The objective of the SI was restricted to assessment of munitions and explosives of concern (MEC) and munitions constituents (MC) under the Military Munitions Response Program (MMRP) and did not assess the need for response actions under FUDS, the need for a Time Critical Removal Action (TCRA), collection of data for a Hazard Ranking System (HRS) score, nor characterization of wastes for initiation of a RI/FS.

In December 2008, analog geophysics and visual observation were completed at the island in Disposal Area 14 (MRS-1; NED Site No. 10), Bunkers 11 and 12 (potential area of interest [PAOI]-1; NED Site Nos. 21 and 22, respectively), the torpedo storehouse (PAOI-2; NED Site No. 38), the north and south gun mounts (PAOI-3, NED Site No. 46), and the magazine ignitor storage area (PAOI-4; NED Site No. 9). No evidence of MEC or MC was observed in any of the PAOIs; therefore, no sampling was conducted. Specifically, Alion attempted to located a previously reported torpedo tail; however, the torpedo was not located during the 2009 inspection.

Several soil and sediment samples were collected from Disposal Area 14 (MRS-1) and analyzed for explosives. Nitroglycerin was detected in one soil sample. Additionally, subsurface material removed from the beach along Disposal Area 14 reportedly contained evidence tar or oil that hardened when exposed to the air.

Based on the low risk considering the absence of a source, stable environment, limited access to the island, and limited exposure frequency, Alion recommended closure of the MMRP project as the identified nitroglycerin would be assessed and addressed as part of the ongoing HTRW project.

DERP FUDS MMRP Closeout Letter of Non-Concurrence, RIDEM, September 17, 2014 (RIDEM, 2014)

In response to USACE's September 11, 2014, letter requesting a No Department of Defense Action Indicated (NDAI) for the Site, RIDEM issued a letter of non-concurrence and identified the following areas that remained to be assessed for munitions:

1. Extent of nitroglycerin in Disposal Area 14 (NED Site No. 10)
2. Location of previously observed, but recently missing, torpedo carcass
3. Bunkers 11 and 12 (NED Site No. 21 and 22, respectively)
4. Torpedo Storehouse (NED Site No. 38)
5. North and South Gun Mounts (NED Site No. 46)
6. Magazine Ignitor Storehouse (NED Site No. 9)

CONTAMINANTS OF POTENTIAL CONCERN

Based on the Site history and previous investigations, the following COPCs are identified for the Site for the indicated media and will be screened for during the investigation:

Soil	Groundwater	Sediment
VOCs SVOCs PP metals Hexavalent chromium EPH/VPH PCBs Explosives Dioxins/furans Pesticides Acids (via pH)	VOCs SVOCs PP metals Hexavalent chromium EPH/VPH PCBs Explosives Pesticides Acids (via pH)*	VOCs SVOCs PP metals Hexavalent chromium EPH/VPH PCBs Explosives Pesticides Acids (via pH)

VOCs – volatile organic compounds
PP – priority pollutant
*To be analyzed by field instrumentation during groundwater sampling.

NATURE AND EXTENT OF CONTAMINATION

As a limited number of samples were previously collected from each specific NED Site No., the extent of contamination at the Site is not known. The inferred source, specific COPCs for each media, expected extent based on currently available information, and current data gaps are summarized in a table in **Appendix B**. A visual depiction of the CSM is also included in **Appendix B**.

EXPOSURE PATHWAYS & POTENTIAL RECEPTORS

As the Site is to be assessed for the need for remedial actions under an unrestricted use scenario, potential receptors are conservatively assumed to be future residents and recreational users, as well as terrestrial and aquatic biota. Exposure pathways to these receptors given the current conceptual site model (CSM) summarized in **Appendix B** would be through direct contact or incidental uptake of contaminated soil, inhalation of VOCs, or active ingestion through drinking contaminated water.

WORKSHEET #11 – PROJECT/DATA QUALITY OBJECTIVES

The purpose of this QAPP is to provide guidance for generating data that are of the precision, accuracy, and completeness necessary for the intended end use of the data. QAPP approval will be obtained prior to initiating any field activities and documented by signatures on **Worksheets #1 & 2** Title and Approval Page.

STATEMENT OF PROBLEM

The problem to be addressed by the SOW proposed herein is insufficient data to support a HHRA/ERA to evaluate the need for remedial action at the Site. Limited prior sampling identified some areas of elevated COPCs and several other AOCs have not been assessed.

STATEMENT OF OBJECTIVES

The primary objective of this RI is to collect sufficient analytical data to support a HHRA/ERA and assess the need for remedial action. The following Site-specific objectives are established:

- Assess each NED Site requiring further investigation and collect sufficient soil, groundwater, and/or sediment data to confirm or dismiss the need for risk-based remedial actions

The physical boundaries of the area to be assessed are depicted on **Figures 4A through 4C**. The scope of work designed to meet these objectives is summarized in **Worksheet #17**.

The primary data quality objectives (DQOs) for all projects is to ensure a) measurements are representative of actual Site conditions and all data resulting from field, sampling, and analytical activities be comparable, reproducible, and generated in a scientifically valid and legally defensible manner; and b) judgments can be made against the applicable regulatory criteria with limited uncertainty.

The analytical sensitivities noted in **Worksheet #15** are those achievable by the respective analytical methods by the analytical laboratory (ARA and their subcontracted lab Test America of Sacramento [TAS]). The primary DQO will be for the reporting limits to be sufficiently lower than the regulatory criteria.

REQUIRED INPUTS

The following data are required to support the above objective:

- Field observations of physical conditions
- Geophysical investigation of unknown subsurface conditions including ground penetrating radar (GPR) and magnetometer data to supplement prior surveys
- Field screening data (e.g., photoionization detector [PID])
- Description of overburden soil types

- Observation of bedrock geology including rock type, quality, and the presence fractures and bedrock aquifers
- Soil, groundwater, and sediment analytical data
- Understanding of tidal fluctuation potential influence on Site hydrogeology

ANALYTICAL APPROACH

Analytical data will be collected to a degree of certainty such that results of the HHRA/ERA are sufficient to meet the DQOs of the RI. Samples are summarized per NED Site in **Worksheet #17** and are considered to represent conditions specific to that NED Site No. unless otherwise specified.

PERFORMANCE AND ACCEPTANCE CRITERIA

Performance and acceptance criteria are summarized throughout this QAPP for both field and laboratory activities. Generally, the project action limits (PALs) are summarized for each compound in **Worksheet #15**.

PLAN FOR OBTAINING DATA

Data will be collected for each specific NED Site based on the expected COPCs from historical use or previous investigations. The sample design includes a mix of discrete and ISM depending on the NED Site specific conditions, estimated degree of risk associated with the NED Site, and nature of the COPCs. A detailed plan for data collection is provided in **Worksheet #17**.

WORKSHEET #12 – MEASUREMENT PERFORMANCE CRITERIA

The following tables summarize the performance criteria for each media and method of proposed analyses.

SOIL, SEDIMENT & CONCRETE

Matrix: Soil/sediment
Analytical Group or Method: VOCs by EPA Method 8260C

Data Quality Indicator	QC Sample or Measurement Performance Activity	Performance Criteria
Precision	Field duplicate	RPD ≤50% when compound detected in both samples ≥LOQ
Analytical Precision	LCSD	RPD ≤20%
Accuracy/Bias	LCS	See worksheet #28
Matrix Interference	MS/MSD	See worksheet #28
Cross Contamination	Equipment blank	No detections >LOQ
	Trip blank	No detections>LOQ
Sensitivity	LOQ verification	Compare to worksheet #15
Completeness	See worksheet #34	See worksheet #34

RPD – relative percent difference
LCS – laboratory control sample
LCSD – laboratory control sample duplicate
MS/MSD – matrix spike/matrix spike duplicate
LOQ – limit of quantitation

Matrix: Sediment
Analytical Group or Method: PAHs by EPA Method 8270D with selective ion monitoring (SIM)

Data Quality Indicator	QC Sample or Measurement Performance Activity	Performance Criteria
Precision	Field duplicate	RPD ≤50% when compound detected in both samples ≥LOQ
Analytical Precision	LCSD	RPD ≤40%
Accuracy/Bias	LCS	See worksheet #28
Matrix Interference	MS/MSD	See worksheet #28
Cross Contamination	Equipment blank	No detections >LOQ
Sensitivity	LOQ verification	Compare to worksheet #15
Completeness	See worksheet #34	See worksheet #34

Matrix: Soil/sediment
Analytical Group or Method: SVOCs by EPA Method 8270D

Data Quality Indicator	QC Sample or Measurement Performance Activity	Performance Criteria
Precision	Field duplicate	RPD ≤50% when compound detected in both samples ≥LOQ
Analytical Precision	LCSD	RPD ≤40%
Accuracy/Bias	LCS	See worksheet #28
Matrix Interference	MS/MSD	See worksheet #28
Cross Contamination	Equipment blank	No detections >LOQ
Sensitivity	LOQ verification	Compare to worksheet #15
Completeness	See worksheet #34	See worksheet #34

Matrix: Soil/sediment
Analytical Group or Method: Metals by EPA Method 6020A

Data Quality Indicator	QC Sample or Measurement Performance Activity	Performance Criteria
Precision	Field duplicate	RPD ≤50% when metal detected in both samples ≥LOQ
Analytical Precision	LCSD	RPD ≤35%
Accuracy/Bias	LCS	See worksheet #28
Matrix Interference	MS/MSD	See worksheet #28
Cross Contamination	Equipment blank	No detections >LOQ
Sensitivity	LOQ verification	Compare to worksheet #15
Completeness	See worksheet #34	See worksheet #34

Matrix: Soil/sediment
Analytical Group or Method: Mercury by EPA Method 7471B

Data Quality Indicator	QC Sample or Measurement Performance Activity	Performance Criteria
Precision	Field duplicate	RPD ≤50% when metal detected in both samples ≥LOQ
Analytical Precision	LCSD	RPD ≤30%
Accuracy/Bias	LCS	See worksheet #28
Matrix Interference	MS/MSD	See worksheet #28
Cross Contamination	Equipment blank	No detections >LOQ
Sensitivity	LOQ verification	Compare to worksheet #15
Completeness	See worksheet #34	See worksheet #34

Matrix: Soil/sediment
Analytical Group or Method: Hexavalent chromium by EPA Method 7196A

Data Quality Indicator	QC Sample or Measurement Performance Activity	Performance Criteria
Precision	Field duplicate	RPD ≤50% when metal detected in both samples ≥LOQ
Analytical Precision	LCSD	RPD ≤20%
Accuracy/Bias	LCS	See worksheet #28
Matrix Interference	MS/MSD	See worksheet #28
Cross Contamination	Equipment blank	No detections >LOQ
Sensitivity	LOQ verification	Compare to worksheet #15
Completeness	See worksheet #34	See worksheet #34

Matrix: Soil/sediment
Analytical Group or Method: EPH by Massachusetts Department of Environmental Protection (MADEP) EPH-04-1.1

Data Quality Indicator	QC Sample or Measurement Performance Activity	Performance Criteria
Precision	Field duplicate	RPD ≤50% when TPH detected in both samples ≥LOQ
Analytical Precision	LCSD	RPD ≤25%
Accuracy/Bias	LCS	See worksheet #28
Matrix Interference	MS/MSD	See worksheet #28
Cross Contamination	Equipment blank	No detections >LOQ
Sensitivity	LOQ verification	Compare to worksheet #15
Completeness	See worksheet #34	See worksheet #34

Matrix: Soil/sediment
Analytical Group or Method: VPH by MADEP VPH-04-1.1

Data Quality Indicator	QC Sample or Measurement Performance Activity	Performance Criteria
Precision	Field duplicate	RPD ≤50% when TPH detected in both samples ≥LOQ
Analytical Precision	LCSD	RPD ≤25%
Accuracy/Bias	LCS	See worksheet #28
Matrix Interference	MS/MSD	See worksheet #28
Cross Contamination	Equipment blank	No detections >LOQ
	Trip Blank	No detections >LOQ
Sensitivity	LOQ verification	Compare to worksheet #15
Completeness	See worksheet #34	See worksheet #34

Matrix: Soil/sediment/concrete
Analytical Group or Method: PCBs by EPA Method 8082A

Data Quality Indicator	QC Sample or Measurement Performance Activity	Performance Criteria
Precision	Field duplicate	RPD ≤50% when Aroclors detected in both samples ≥LOQ
Analytical Precision	LCSD	RPD ≤30%
Accuracy/Bias	LCS	See worksheet #28
Matrix Interference	MS/MSD	See worksheet #28
Cross Contamination	Equipment blank	No detections >LOQ
Sensitivity	LOQ verification	Compare to worksheet #15
Completeness	See worksheet #34	See worksheet #34

Matrix: Soil/sediment
Analytical Group or Method: Explosives (with nitroglycerin) by EPA Method 8330B

Data Quality Indicator	QC Sample or Measurement Performance Activity	Performance Criteria
Precision	Field duplicate	RPD ≤50% when analyte detected in both samples ≥LOQ
Analytical Precision	LCSD	RPD ≤20%
Accuracy/Bias	LCS	See worksheet #28
Matrix Interference	MS/MSD	See worksheet #28
Cross Contamination	Equipment blank	No detections >LOQ
Sensitivity	LOQ verification	Compare to worksheet #15
Completeness	See worksheet #34	See worksheet #34

Matrix: Soil
Analytical Group or Method: Dioxins and furans by EPA Method 1613B

Data Quality Indicator	QC Sample or Measurement Performance Activity	Performance Criteria
Precision	Field duplicate	RPD ≤50% when analyte detected in both samples ≥LOQ
Analytical Precision	LCSD	RPD ≤20%
Accuracy/Bias	LCS	See worksheet #28
Matrix Interference	MS/MSD	See worksheet #28
Cross Contamination	Equipment blank	No detections >LOQ
Sensitivity	LOQ verification	Compare to worksheet #15
Completeness	See worksheet #34	See worksheet #34

Matrix: Soil/sediment
Analytical Group or Method: Pesticides by EPA Method 8081B

Data Quality Indicator	QC Sample or Measurement Performance Activity	Performance Criteria
Precision	Field duplicate	RPD ≤50% when analyte detected in both samples ≥LOQ
Analytical Precision	LCSD	RPD ≤30%
Accuracy/Bias	LCS	See worksheet #28
Matrix Interference	MS/MSD	See worksheet #28
Cross Contamination	Equipment blank	No detections >LOQ
Sensitivity	LOQ verification	Compare to worksheet #15
Completeness	See worksheet #34	See worksheet #34

Matrix: Soil/sediment
Analytical Group or Method: pH by EPA Method 9045C

Data Quality Indicator	QC Sample or Measurement Performance Activity	Performance Criteria
Precision	Field duplicate	0.2 pH units of parent sample
Completeness	See worksheet #34	See worksheet #34

GROUNDWATER

Matrix: Groundwater
Analytical Group or Method: VOCs by EPA Method 8260C

Data Quality Indicator	QC Sample or Measurement Performance Activity	Performance Criteria
Precision	Field duplicate	RPD ≤30% when compound detected in both samples ≥LOQ
Analytical Precision	LCSD	RPD ≤20%
Accuracy/Bias	LCS	See worksheet #28
Matrix Interference	MS/MSD	See worksheet #28
Cross Contamination	Equipment blank	No detections >LOQ
	Trip Blank	No detections >LOQ
Sensitivity	LOQ verification	Compare to worksheet #15
Completeness	See worksheet #34	See worksheet #34

Matrix: Groundwater
Analytical Group or Method: PAHs by EPA Method 8270D with SIM

Data Quality Indicator	QC Sample or Measurement Performance Activity	Performance Criteria
Precision	Field duplicate	RPD ≤30% when compound detected in both samples ≥LOQ
Analytical Precision	LCSD	RPD ≤40%
Accuracy/Bias	LCS	See worksheet #28
Matrix Interference	MS/MSD	See worksheet #28
Cross Contamination	Equipment blank	No detections >LOQ
Sensitivity	LOQ verification	Compare to worksheet #15
Completeness	See worksheet #34	See worksheet #34

Matrix: Groundwater
Analytical Group or Method: SVOCs by EPA Method 8270D

Data Quality Indicator	QC Sample or Measurement Performance Activity	Performance Criteria
Precision	Field duplicate	RPD ≤30% when compound detected in both samples ≥LOQ
Analytical Precision	LCSD	RPD ≤40%
Accuracy/Bias	LCS	See worksheet #28
Matrix Interference	MS/MSD	See worksheet #28
Cross Contamination	Equipment blank	No detections >LOQ
Sensitivity	LOQ verification	Compare to worksheet #15
Completeness	See worksheet #34	See worksheet #34

Matrix: Groundwater
Analytical Group or Method: Metals by EPA Method 6020A

Data Quality Indicator	QC Sample or Measurement Performance Activity	Performance Criteria
Precision	Field duplicate	RPD ≤30% when metal detected in both samples ≥LOQ
Analytical Precision	LCSD	RPD ≤20%
Accuracy/Bias	LCS	See worksheet #28
Matrix Interference	MS/MSD	See worksheet #28
Cross Contamination	Equipment blank	No detections >LOQ
Sensitivity	LOQ verification	Compare to worksheet #15
Completeness	See worksheet #34	See worksheet #34

Matrix: Groundwater
Analytical Group or Method: Mercury by EPA Method 7470A

Data Quality Indicator	QC Sample or Measurement Performance Activity	Performance Criteria
Precision	Field duplicate	RPD ≤30% when metal detected in both samples ≥LOQ
Analytical Precision	LCSD	RPD ≤20%
Accuracy/Bias	LCS	See worksheet #28
Matrix Interference	MS/MSD	See worksheet #28
Cross Contamination	Equipment blank	No detections >LOQ
Sensitivity	LOQ verification	Compare to worksheet #15
Completeness	See worksheet #34	See worksheet #34

Matrix: Groundwater
Analytical Group or Method: Hexavalent chromium by EPA Method 7196A

Data Quality Indicator	QC Sample or Measurement Performance Activity	Performance Criteria
Precision	Field duplicate	RPD ≤30% when metal detected in both samples ≥LOQ
Analytical Precision	LCSD	RPD ≤20%
Accuracy/Bias	LCS	See worksheet #28
Matrix Interference	MS/MSD	See worksheet #28
Cross Contamination	Equipment blank	No detections >LOQ
Sensitivity	LOQ verification	Compare to worksheet #15
Completeness	See worksheet #34	See worksheet #34

Matrix: Groundwater
Analytical Group or Method: EPH by MADEP EPH-04-1.1

Data Quality Indicator	QC Sample or Measurement Performance Activity	Performance Criteria
Precision	Field duplicate	RPD ≤30% when TPH detected in both samples ≥LOQ
Analytical Precision	LCSD	RPD ≤25%
Accuracy/Bias	LCS	See worksheet #28
Matrix Interference	MS/MSD	See worksheet #28
Cross Contamination	Equipment blank	No detections >LOQ
Sensitivity	LOQ verification	Compare to worksheet #15
Completeness	See worksheet #34	See worksheet #34

Matrix: Groundwater
Analytical Group or Method: VPH by MADEP VPH-04-1.1

Data Quality Indicator	QC Sample or Measurement Performance Activity	Performance Criteria
Precision	Field duplicate	RPD ≤30% when TPH detected in both samples ≥LOQ
Analytical Precision	LCSD	RPD ≤25%
Accuracy/Bias	LCS	See worksheet #28
Matrix Interference	MS/MSD	See worksheet #28
Cross Contamination	Equipment blank	No detections >LOQ
	Trip blank	No detections >LOQ
Sensitivity	LOQ verification	Compare to worksheet #15
Completeness	See worksheet #34	See worksheet #34

Matrix: Groundwater
Analytical Group or Method: PCBs by EPA Method 8082A

Data Quality Indicator	QC Sample or Measurement Performance Activity	Performance Criteria
Precision	Field duplicate	RPD ≤30% when Aroclors detected in both samples ≥LOQ
Analytical Precision	LCSD	RPD ≤20%
Accuracy/Bias	LCS	See worksheet #28
Matrix Interference	MS/MSD	See worksheet #28
Cross Contamination	Equipment blank	No detections >LOQ
Sensitivity	LOQ verification	Compare to worksheet #15
Completeness	See worksheet #34	See worksheet #34

Matrix: Groundwater
Analytical Group or Method: Explosives (with nitroglycerin) by EPA Method 8330B

Data Quality Indicator	QC Sample or Measurement Performance Activity	Performance Criteria
Precision	Field duplicate	RPD ≤30% when analyte detected in both samples ≥LOQ
Analytical Precision	LCSD	RPD ≤20%
Accuracy/Bias	LCS	See worksheet #28
Matrix Interference	MS/MSD	See worksheet #28
Cross Contamination	Equipment blank	No detections >LOQ
Sensitivity	LOQ verification	Compare to worksheet #15
Completeness	See worksheet #34	See worksheet #34

Matrix: Groundwater
Analytical Group or Method: Pesticides by EPA Method 8081B

Data Quality Indicator	QC Sample or Measurement Performance Activity	Performance Criteria
Precision	Field duplicate	RPD ≤30% when analyte detected in both samples ≥LOQ
Analytical Precision	LCSD	RPD ≤20%
Accuracy/Bias	LCS	See worksheet #28
Matrix Interference	MS/MSD	See worksheet #28
Cross Contamination	Equipment blank	No detections >LOQ
Sensitivity	LOQ verification	Compare to worksheet #15
Completeness	See worksheet #34	See worksheet #34

WORKSHEET #13 – SECONDARY DATA USES AND LIMITATIONS

The following table summarizes secondary data previously generated for this project under a separate QAPP. Information is summarized relevant to its current project use.

Data Type	Source	Data Use for Project	Factors Affecting Data
Geological Data	Hermes et al, 1994	Comparison to observed geologic conditions	Variability from mapped data due to scale of map
Island Physical Setting	Tetra Tech, 2006	Consideration with Site data to better define physical setting of the island and CSM	Specific to island but offsite data
Historical data	SWETS, 1997	Target locations for sampling	Location reproducibility, differing standard of care since 1997
Munition Assessment	Alion, 2009	Health and Safety purposes and the extent of Disposal Area #14	Overgrown vegetation limiting reproduction/confirmation of the geophysical limits.

WORKSHEET # 14 & 16 – PROJECT TASKS AND SCHEDULE

The following table summarizes the schedule for project activities from completion of review of this QAPP through implementation of field activities by Credere.

Date	Task
October 2017	Submit Draft QAPP/FSP and Accident Prevention Plan (APP)/Site-Specific Safety and Health Plan (SSHP)
November 2017	Receive QAPP/FSP and APP/SSHP comments from CENAE
November 2017	Incorporate comments and submit response to comment (RTC) and Stakeholder-Draft version of QAPP/FSP and Final version of APP/SSHP
November – December 2017	Site clearing and initial site visit
December 2017 – February 2018	Submit QAPP/FSP to RIDEM
February 2018	Incorporate RIDEM comments and submit RTC and Final version of QAPP/FSP
February 2018	Complete electronic QAPP (eQAPP) and event planning in FUDSChem
February – March 2018	Perform remedial investigation field work
April 1st – August 15th (RIDEM, 2010)	ISLAND INACCESSIBLE TO HEAVY EQUIPMENT DUE TO BIRD NESTING (low impact hand tools may be allowed)
March-April 2018	Chain of custody reconciliation and upload to FUDSChem (within 3 days of field demobilization)
May 2018	RI Field Report and Project Team Meeting
July 2018	Submit Draft RI Report (if additional investigation is not warranted)
July-August 2018	CENAE Review Period and RTC Period for Draft RI Report
August - October 2018	RIDEM and RAB review period for Stakeholder-Draft RI Report
November 2018	Final RI
Fall 2018	Alternatively, additional remedial investigation activities after August 15th

WORKSHEET #15 – PROJECT ACTION LIMITS AND LABORATORY SPECIFIC DETECTION/QUANTITATION LIMITS

Laboratory analytical analysis will be performed by ARA with some analyses subcontracted to TAS, both DoD Environmental Laboratory Accreditation Program (ELAP) accredited laboratories. Certification documentation is included as **Appendix C**. Referenced laboratory standard operating procedures (SOPs) are included in **Appendix D**.

SOIL

Soil PALs indicated in the following tables are EPA Regional Screening Levels (RSLs) at the target hazard quotient of 0.1 for the residential (unrestricted) exposure scenario (EPA, 2017a), which are the lowest published RSL values. Petroleum hydrocarbon fraction PALs are those established by the Massachusetts Contingency Plan (MCP) for use associated with the MADEP EPH and VPH methods, those PALs are indicated with a ^. PALs and/or project quantitation limits (PQLs) that cannot be achieved by the selected analytical laboratory/method are highlighted bold. For these compounds, non-detect results with the limit of detection (LOD) greater than the PQL will be considered Site-specific COPC and will be further evaluated in the risk assessment. If the risk assessment identifies these compounds as COCs, alternative methods will be evaluated. Laboratory LODs may vary as new studies are performed and limits are updated.

Lab: ARA
Matrix: Soil
Method: VOCs by EPA Method 8260C (SOP QA-5120)

Compound	PAL (mg/kg)	PQL (mg/kg)	LOQ (mg/kg)	LOD (mg/kg)
1,1,1,2-tetrachloroethane	2.0	1.0	0.1	0.025
1,1,1-trichloroethane	810	405	0.1	0.025
1,1,2,2-tetrachloroethane	0.6	0.3	0.1	0.025
1,1,2-trichloroethane	0.15	0.075	0.1	0.025
1,1-dichloroethane	3.6	1.8	0.1	0.025
1,1-dichloroethene	23	11.5	0.1	0.025
1,1-dichloropropene	NE	0.025	0.1	0.025
1,2,3-trichlorobenzene	6.3	3.15	0.1	0.025
1,2,3-trichloropropane	**0.0051**	**0.0026**	0.1	0.025
1,2,4-trichlorobenzene	5.8	2.9	0.1	0.025
1,2,4-trimethylbenzene	30	15	0.1	0.025
1,2-dibromo-3-chloropropane/DBCP	**0.0053**	**0.0027**	0.1	0.1
1,2-dibromoethane/EDB	0.036	**0.018**	0.1	0.025
1,2-dichlorobenzene	180	90	0.1	0.025
1,2-dichloroethane	0.46	0.23	0.1	0.025
1,2-dichloropropane	0.28	0.14	0.1	0.025
1,3,5-trimethylbenzene	27	13.5	0.1	0.025
1,3-dichlorobenzene	NE	0.025	0.1	0.025
1,3-dichloropropane	160	80	0.1	0.025
1,4-dichlorobenzene	2.6	1.4	0.1	0.025

Compound	PAL (mg/kg)	PQL (mg/kg)	LOQ (mg/kg)	LOD (mg/kg)
1,4-dioxane	5.3	2.65	2	2.5
2,2-dichloropropane	NE	0.025	0.1	0.025
2-butanone (methyl ethyl ketone)	2,700	1350	0.3	0.025
2-chlorotoluene	160	80	0.1	0.025
2-hexanone	20	10	0.5	0.025
4-chlorotoluene	160	80	0.1	0.025
4-isopropyltoluene	NE	0.025	0.1	0.025
4-methyl-2-pentanone/MIBK	3,300	1,650	0.4	0.025
acetone	6,100	3,050	2	0.2
benzene	1.2	0.6	0.1	0.025
bromobenzene	29	14.5	0.1	0.025
bromochloromethane	15	7.5	0.1	0.025
bromodichloromethane	0.29	0.145	0.1	0.025
bromoform	19	8.5	0.1	0.025
bromomethane	0.68	0.34	0.2	0.025
carbon disulfide	77	38.5	0.1	0.025
carbon tetrachloride	0.65	0.33	0.1	0.025
chlorobenzene	28	14	0.1	0.025
chloroethane	1,400	700	0.1	0.025
chloroform	0.32	0.16	0.1	0.025
chloromethane	11	5.5	0.1	0.025
cis-1,2-dichloroethene	16	8	0.1	0.025
cis-1,3-dichloropropene	NE	0.025	0.1	0.025
dibromochloromethane	8.3	4.15	0.1	0.025
dibromomethane	2.4	1.2	0.1	0.025
dichlorodifluoromethane	8.7	4.35	0.1	0.025
diethyl ether	1,600	800	0.1	0.025
ethylbenzene	5.8	2.9	0.1	0.025
hexachlorobutadiene	1.2	0.6	0.1	0.025
Isopropylbenzene (cumene)	190	95	0.1	0.025
m&p-xylenes	55	27.5	0.1	0.05
methyl t-butyl ether/MTBE	47	23.5	0.1	0.025
methylene chloride	35	17.5	0.1	0.025
naphthalene	3.8	1.9	0.1	0.025
n-butylbenzene	390	170	0.1	0.025
n-propylbenzene	NE	0.025	0.1	0.025
o-xylene	65	32.5	0.1	0.025
sec-butylbenzene	780	390	0.1	0.025
styrene	600	300	0.1	0.025
tert-butylbenzene	780	390	0.1	0.025
tetrachloroethene	8.1	4.05	0.1	0.025
tetrahydrofuran	1,800	600	0.5	0.2
toluene	490	245	0.1	0.025
trans-1,2-dichloroethene	160	80	0.1	0.025
trans-1,3-dichloropropene	NE	0.025	0.1	0.025
trichloroethene	0.41	0.205	0.1	0.025
trichlorofluoromethane	2,300	1,150	0.1	0.025

Compound	PAL (mg/kg)	PQL (mg/kg)	LOQ (mg/kg)	LOD (mg/kg)
vinyl chloride	0.059	0.0295	0.1	0.025

mg/kg – milligrams per kilogram
PAL – Project action limit
PQL – Project quantitation limit (half the PAL, or equal to LOD if no PAL)
LOQ – Limit of quantification
NE – not established
For compounds with no PAL, the LOD is considered the PQL

Lab: ARA
Matrix: Soil
Method: SVOCs by EPA Method 8270D (SOP QA-5515)

Compound	PAL (mg/kg)	PQL (mg/kg)	LOQ (mg/kg)	LOD (mg/kg)
1,2,4-trichlorobenzene	5.8	2.9	0.5	0.20
1,2-dichlorobenzene	180	90	0.2	0.20
1,3-dichlorobenzene	NE	0.2	0.2	0.20
1,4-dichlorobenzene	2.6	1.3	0.2	0.20
1,4-dioxane	5.3	2.65	2	2
2,4,5-trichlorophenol	630	315	0.2	0.20
2,4,6-trichlorophenol	6.3	3.15	0.2	0.20
2,4-dichlorophenol	19	8.5	0.5	0.20
2,4-dimethylphenol	130	65	0.2	0.20
2,4-dinitrophenol	13	6.5	5	0.20
2,4-dinitrotoluene	1.7	0.85	0.2	0.20
2,6-dinitrotoluene	0.36	**0.18**	0.2	0.20
2-chloronaphthalene	NE	0.20	0.5	0.20
2-chlorophenol	39	19.5	0.5	0.20
2-methylnaphthalene	24	12	0.05	0.05
2-methylphenol	320	160	0.2	0.20
2-nitroaniline	63	31.5	0.2	0.20
2-nitrophenol	NE	0.20	0.2	0.20
3,3'-dichlorobenzidine	**1.2**	**0.6**	3	2
3-nitroaniline	NE	0.2	0.2	0.20
4,6-dinitro-2-methylphenol	0.51	**0.255**	2	0.40
4-bromophenyl phenyl ether	NE	0.20	0.2	0.20
4-chloro-3-methylphenol	630	315	0.2	0.20
4-chloroaniline	2.7	1.35	0.2	0.20
4-chlorophenyl phenyl ether	NE	0.20	0.5	0.20
4-methylphenol	630	315	0.2	0.20
4-nitroaniline	25	12.5	0.5	0.20
4-nitrophenol	NE	0.40	2	0.40
Acenaphthene	360	180	0.05	0.05
Acenaphthylene	NE	0.05	0.05	0.05
Aniline	44	22	0.2	0.20
Anthracene	1,800	900	0.05	0.05
Azobenzene	5.6	2.8	0.2	0.20
Benzidine	**0.00053**	**0.000265**	3	0.2
Benzo(a)anthracene	1.1	0.55	0.05	0.05

Compound	PAL (mg/kg)	PQL (mg/kg)	LOQ (mg/kg)	LOD (mg/kg)
Benzo(a)pyrene	0.11	0.055	0.05	0.05
Benzo(b)fluoranthene	1.1	0.55	0.05	0.05
Benzo(g,h,i)perylene	NE	1	0.05	0.05
Benzo(k)fluoranthene	1.1	0.55	0.05	0.05
Benzoic acid	25,000	12,500	5	0.2
Benzyl alcohol	630	315	0.2	0.20
Bis(2-chloroethoxy)methane	19	9.5	0.2	0.20
Bis(2-chloroethyl)ether	0.23	0.115	0.2	0.20
Bis(2-chloroisopropyl)ether	NE	0.20	0.2	0.20
Bis(2-ethylhexyl)phthalate	63	31.5	0.5	0.20
Butyl benzyl phthalate	290	145	0.5	0.20
Carbazole	NE	0.20	0.2	0.20
Chrysene	110	55	0.05	0.05
Dibenzo(a,h)anthracene	0.11	0.055	0.05	0.05
Dibenzofuran	7.3	3.65	0.05	0.05
Diethyl phthalate	5,100	2,550	0.5	0.20
Dimethylphthalate	780	370	0.5	0.20
Di-n-butylphthalate	630	315	0.5	0.20
Di-n-octyl phthalate	63	31.5	0.5	0.20
Fluoranthene	240	120	0.05	0.05
Fluorene	240	120	0.05	0.05
Hexachlorobenzene	**0.21**	**0.105**	0.2	0.20
Hexachlorobutadiene	1.2	0.6	0.2	0.20
Hexachlorocyclopentadiene	**0.18**	**0.09**	1	0.20
Hexachloroethane	1.8	0.9	0.2	0.20
Indeno(1,2,3-cd)pyrene	1.1	0.55	0.05	0.05
Isophorone	NE	0.2	0.5	0.20
Naphthalene	3.8	1.9	0.05	0.05
Nitrobenzene	5.1	2.55	0.2	0.20
N-nitrosodimethylamine	**0.00081**	**0.000405**	0.2	0.20
N-nitroso-di-N-propylamine	**0.078**	**0.039**	0.2	0.20
N-nitrosodiphenylamine	110	55	0.2	0.20
Pentachlorophenol	1.0	0.5	1	0.40
Phenanthrene	NE	0.05	0.05	0.05
Phenol	1,900	800	0.2	0.20
Pyrene	180	90	0.05	0.05

Lab: ARA
Matrix: Soil
Method: Metals by EPA Method 6020A (SOP QA-5605)

Compound	PAL (mg/kg)	PQL (mg/kg)	LOQ (mg/kg)	LOD (mg/kg)
Antimony	3.1	1.55	0.5	0.05
Arsenic	0.68	**0.34**	2.5	0.5
Beryllium	16	8	0.5	0.05
Cadmium	7.1	3.55	0.5	0.05
Chromium, total	0.3	0.15	5	0.05
Chromium^{+3} (as total)	12,000	6,000		
Copper	310	155	5	0.05
Lead	400	200	2.5	0.05
Nickel	NE	0.05	5	0.05
Selenium	39	19.5	5	0.5
Silver	39	19.5	2.5	0.03
Thallium	NE	0.03	0.5	0.03
Zinc	2,300	1,150	5	0.5

Lab: ARA
Matrix: Soil
Method: Mercury by EPA Method 7471B (SOP QA-5600)

Compound	PAL (mg/kg)	PQL (mg/kg)	LOQ (mg/kg)	LOD (mg/kg)
Mercury	0.78	0.39	0.02	0.016

Lab: ARA
Matrix: Soil
Method: Hexavalent chromium by EPA Method 7196A (SOP QA-5813)

Compound	PAL (mg/kg)	PQL (mg/kg)	LOQ (mg/kg)	LOD (mg/kg)
Chromium $^{+6}$	**0.3**	**0.15**	0.4	0.4

Lab: ARA
Matrix: Soil
Method: EPH by MADEP Method EPH-04-1.1 (SOP QA-5313)

Compound	PAL (mg/kg)	PQL (mg/kg)	LOQ (mg/kg)	LOD (mg/kg)
Naphthalene	3.8	1.9	0.2	0.1
2-Methylnaphthalene	24	12	0.2	0.1
Phenanthrene	NE	0.1	0.2	0.1
Acenaphthene	360	180	0.2	0.1
Acenaphthylene	NE	0.1	0.2	0.1
Fluorene	240	120	0.2	0.1
Anthracene	1,800	900	0.2	0.1
Fluoranthene	240	120	0.2	0.1
Pyrene	180	90	0.2	0.1
Benzo(a)anthrancene	1.1	0.55	0.2	0.1
Chrysene	110	55	0.2	0.1
Benzo(b)fluoranthene	1.1	0.55	0.2	0.1
Benzo(k)fluoranthene	11	5.5	0.2	0.1
Benzo(a)pyrene	0.11	**0.055**	0.2	0.1
Indeno(1,2,3-cd) pyrene	1.1	0.55	0.2	0.1
Dibenzo(a,h)anthracene	0.11	**0.055**	0.2	0.1
Benzo(g,h,i)perylene	NE	0.1	0.2	0.1
C9-C18 Aliphatics	1,000^	500	20	6
C19-C36 Aliphatics	3,000^	1,500	20	10
C11-C22 Aromatics	1,000^	500	20	6

Lab: ARA
Matrix: Soil
Method: VPH by MADEP Method VPH-04-1.1 (SOP QA-5130)

Compound	PAL (mg/kg)	PQL (mg/kg)	LOQ (mg/kg)	LOD (mg/kg)
Methyl tert-butyl ether	47	23.5	0.1	0.05
Benzene	1.2	0.6	0.1	0.05
Toluene	490	245	0.1	0.05
Ethylbenzene	5.8	2.9	0.1	0.05
m&p Xylenes	55	27.5	0.1	0.05
o-Xylenes	65	32.5	0.1	0.05
Naphthalene	3.8	1.9	0.2	0.05
C5-C8 Aliphatics	100^	50	4	2
C9-C12 Aliphatics	1,000^	500	4	3
C9-C10 Aromatics	100^	50	4	0.4

Lab: ARA
Matrix: Soil
Method: PCBs by EPA Method 8082A (SOP QA-5303)

Compound	PAL (mg/kg)	PQL (mg/kg)	LOQ (mg/kg)	LOD (mg/kg)
PCB-1016	0.41	0.205	0.2	0.04
PCB-1221	0.2	0.1	0.2	0.04
PCB-1232	0.17	0.085	0.2	0.04
PCB-1242	0.23	0.115	0.2	0.04
PCB-1248	0.23	0.115	0.2	0.04
PCB-1254	0.12	0.06	0.2	0.04
PCB-1260	0.24	0.12	0.2	0.04

Lab: TAS
Matrix: Soil
Method: Explosives by EPA Method 8330B (SOP WS-LC-0009)

Compound	PAL (mg/kg)	PQL (mg/kg)	LOQ (mg/kg)	LOD (mg/kg)
1,3,5-trinitrobenzene	220	110	0.25	0.05
1,3-dinitrobenzene	0.63	0.315	0.25	0.05
2,4,6-trinitrotoluene/TNT	0.8	0.4	0.25	0.05
2,4-dinitrotoluene	1.7	0.85	0.25	0.05
2,6-dinitrotoluene	0.36	0.18	0.25	0.05
2-amino-4,6-dinitrotoluene	15	7.5	0.25	0.05
4-amino-2,6-dinitrotoluene	15	7.5	0.25	0.05
2-nitrotoluene	3.2	1.6	0.25	0.05
3-nitrotoluene	0.63	0.315	0.25	0.05
4-nitrotoluene	25	12.5	0.25	0.05
nitrobenzene	5.1	2.55	0.25	0.05
nitroglycerin	0.63	0.315	0.5	0.25
pentaerythritol tetranitrate/PETN	13	6.5	0.5	0.25
hexahydro-1,3,5-trinitro-1,3,5-triazine/RDX	6.1	3.05	0.25	0.05
trinitrophenylmethylnitramine (tetryl)	16	8	0.25	0.05

Lab: TAS
Matrix: Soil
Method: Dioxin/furans by EPA Method 1613B (SOP WS-ID-0007)

Compound	PAL (mg/kg)	PQL (mg/kg)	LOQ (mg/kg)	LOD (mg/kg)
2,3,7,8-TCDD	0.0000045	0.0000045	0.000001	0.0000004
2,3,7,8-TCDF	NE	0.0000004	0.000001	0.0000004
1,2,3,7,8-PeCDD	NE	0.00000075	0.000005	0.00000075
1,2,3,7,8-PeCDF	NE	0.00000075	0.000005	0.00000075
2,3,4,7,8-PeCDF	NE	0.00000075	0.000005	0.00000075
1,2,3,4,7,8-HxCDD	0.0001	0.0001	0.000005	0.000002
1,2,3,6,7,8-HxCDD	0.0001	0.0001	0.000005	0.000002
1,2,3,7,8,9-HxCDD	0.0001	0.0001	0.000005	0.000002
1,2,3,4,7,8-HxCDF	0.0001	0.0001	0.000005	0.00000075
1,2,3,6,7,8-HxCDF	0.0001	0.0001	0.000005	0.000001
1,2,3,7,8,9-HxCDF	0.0001	0.0001	0.000005	0.000001

Compound	PAL (mg/kg)	PQL (mg/kg)	LOQ (mg/kg)	LOD (mg/kg)
2,3,4,6,7,8-HxCDF	0.0001	0.0001	0.000005	0.00000075
1,2,3,4,6,7,8-HpCDD	NE	0.000001	0.000005	0.000001
1,2,3,4,6,7,8-HpCDF	NE	0.000001	0.000005	0.000001
1,2,3,4,7,8,9-HpCDF	NE	0.000002	0.000005	0.000002
OCDD	NE	0.000004	0.000010	0.000004
OCDF	NE	0.000004	0.000010	0.000004

PQL is equal to the PAL for dioxins because the PAL is already at the low end of the analytical limits.

Lab: ARA
Matrix: Soil
Method: Pesticides by EPA Method 8081B (SOP QA-5304)

Compound	PAL (mg/kg)	PQL (mg/kg)	LOQ (mg/kg)	LOD (mg/kg)
4,4'-DDD	2.3	1.15	0.04	0.03
4,4'-DDE	2.0	1.0	0.04	0.03
4,4'-DDT	1.9	0.95	0.04	0.03
Aldrin	0.039	**0.0195**	0.04	0.03
alpha-BHC	0.086	0.043	0.04	0.03
alpha-chlordane	1.7	0.85	0.04	0.03
gamma-chlordane	1.7	0.85	0.04	0.03
beta-BHC	0.3	0.15	0.04	0.03
delta-BHC	NE	0.03	0.04	0.03
Dieldrin	0.034	**0.017**	0.04	0.03
Endosulfan I	47	23.5	0.04	0.03
Endosulfan II	47	23.5	0.04	0.03
Endosulfan sulfate	47	23.5	0.04	0.03
Endrin	1.9	0.95	0.04	0.03
Endrin ketone	NE	0.03	0.04	0.03
Endrin aldehyde	NE	0.03	0.04	0.03
gamma-BHC (Lindane)	0.57	0.285	0.04	0.03
Heptachlor	0.13	0.065	0.04	0.03
Heptachlor epoxide	0.07	0.035	0.04	0.03
Methoxychlor	32	16	0.04	0.03
Toxaphene	0.49	0.245	0.2	0.2

Lab: ARA
Matrix: Soil
Method: pH by EPA Method 9045C (SOP QA-5851)

Compound	PAL	PQL	LOQ	LOD
pH	6-8	NA	0-14	0-14

GROUNDWATER

Groundwater PALs summarized in the following tables are the EPA Maximum Contaminant Levels (MCLs; EPA, 2017b), where established, at the target hazard quotient (THQ) 0.1, which are the lowest applicable published values. Where MCLs are not established, RIDEM GA groundwater objectives (GWOs) are used to conservatively screen groundwater conditions for unrestricted future use, those PALs are indicated with an *. Petroleum hydrocarbon fraction action levels are those established by the MCP for use associated with the MADEP EPH and VPH methods; those PALs are indicated with a ^. PALs and/or PQLs that cannot be achieved by

the selected analytical laboratory/method are highlighted bold. For these compounds, non-detect results with the LOD greater than the PQL will be considered Site-specific COPCs and will be further evaluated in the risk assessment. If the risk assessment identifies these compounds as COCs, alternative methods will be evaluated. COPCs will be identified based on exceedances of applicable PALs, exceedances or detections of other similarly grouped analytes (e.g., other PAHs), and based on observations of evidence of releases of certain compounds in the field (e.g., PID response, labeled containers, evidence of spilled drums, etc). Laboratory LODs may vary as new studies are performed and limits are updated.

Lab: ARA
Matrix: Groundwater
Method: VOCs by EPA Method 8260C (SOP QA-5120)

Compound	PAL (µg/L)	PQL (µg/L)	LOQ (µg/L)	LOD (µg/L)
1,1,1,2-tetrachloroethane	NE	1	2	1
1,1,1-trichloroethane	200	100	2	1
1,1,2,2-tetrachloroethane	NE	1	2	1
1,1,2-trichloroethane	5.0	2.5	2	1
1,1-dichloroethane	NE	1	2	1
1,1-dichloroethene	7.0	3.5	1	1
1,1-dichloropropene	NE	1	2	1
1,2,3-trichlorobenzene	NE	1	2	1
1,2,3-trichloropropane	NE	1	2	1
1,2,4-trichlorobenzene	70	35	2	1
1,2,4-trimethylbenzene	NE	1	2	1
1,2-dibromo-3-chloropropane/DBCP)	**0.2**	**0.1**	2	1
1,2-dibromoethane /EDB	**0.05**	**0.025**	2	1
1,2-dichlorobenzene	600	300	2	1
1,2-dichloroethane	5.0	2.5	2	1
1,2-dichloropropane	5.0	2.5	2	1
1,3,5-trimethylbenzene	NE	1	2	1
1,3-dichlorobenzene	NE	1	2	1
1,3-dichloropropane	NE	1	2	1
1,4-dichlorobenzene	75	37.5	2	1
1,4-dioxane	NE	10	50	10
2,2-dichloropropane	NE	1	2	1
2-butanone (MEK)	NE	5	10	5
2-chlorotoluene	NE	1	2	1
2-hexanone	NE	1	10	1
4-chlorotoluene	NE	1	2	1
4-isopropyltoluene	NE	1	2	1
4-methyl-2-pentanone/MIBK	NE	1	10	1
acetone	NE	5	50	5
benzene	5.0	2.5	2	1
bromobenzene	NE	1	2	1
bromochloromethane	NE	1	2	1

Compound	PAL (µg/L)	PQL (µg/L)	LOQ (µg/L)	LOD (µg/L)
bromodichloromethane	80	40	0.6	0.5
bromoform	80	40	2	1
bromomethane	NE	2	2	2
carbon disulfide	NE	1	2	1
carbon tetrachloride	5.0	2.5	2	1
chlorobenzene	100	50	2	1
chloroethane	NE	1	2	1
chloroform	80	40	2	1
chloromethane	NE	1	2	1
cis-1,2-dichloroethene	70	35	2	1
cis-1,3-dichloropropene	NE	1	2	1
dibromochloromethane	80	40	2	1
dibromomethane	NE	1	2	1
dichlorodifluoromethane	NE	1	2	1
diethyl ether	NE	1	5	1
ethylbenzene	700	350	2	1
hexachlorobutadiene	NE	1	2	1
isopropylbenzene	NE	1	2	1
m&p-xylenes	10,000	5,000	2	2
methyl t-butyl etherMTBE	40*	20	2	1
methylene chloride	5.0	2.5	5	1
naphthalene	100*	50	5	1
n-butylbenzene	NE	1	2	1
n-propylbenzene	NE	1	2	1
o-xylene	10,000	5,000	2	1
sec-butylbenzene	NE	1	2	1
styrene	100	50	2	1
tert-butylbenzene	NE	1	2	1
tetrachloroethene	5.0	2.5	2	1
tetrahydrofuran	NE	5	10	5
toluene	1,000	500	2	1
trans-1,2-dichloroethene	100	50	2	1
trans-1,3-dichloropropene	NE	1	2	1
trichloroethene	5.0	2.5	2	1
trichlorofluoromethane	NE	1	2	1
vinyl chloride	2.0	1.0	2	1

µg/L – micrograms per liter

Lab: TAS
Matrix: Groundwater
Method: PAHs by EPA Method 8270D with SIM (SOP WS-MW-0008)

Compound	PAL (µg/L)	PQL (µg/L)	LOQ (µg/L)	LOD (µg/L)
1-Methylnaphthalene	NE	0.015	0.05	0.015

Compound	PAL (µg/L)	PQL (µg/L)	LOQ (µg/L)	LOD (µg/L)
2-Methylnaphthalene	NE	0.015	0.05	0.015
Acenaphthene	NE	0.015	0.05	0.015
Acenaphthylene	NE	0.015	0.05	0.015
Anthracene	NE	0.015	0.05	0.015
Benzo[a]anthracene	NE	0.015	0.05	0.015
Benzo[a]pyrene	0.2	0.1	0.05	0.015
Benzo[b]fluoranthene	NE	0.030	0.05	0.030
Benzo[g,h,i]perylene	NE	0.015	0.05	0.015
Benzo[k]fluoranthene	NE	0.030	0.05	0.030
Chrysene	NE	0.015	0.05	0.015
Dibenz(a,h)anthracene	NE	0.030	0.05	0.030
Fluoranthene	NE	0.015	0.05	0.015
Fluorene	NE	0.015	0.05	0.015
Indeno[1,2,3-cd]pyrene	NE	0.030	0.05	0.030
Naphthalene	NE	0.015	0.05	0.015
Phenanthrene	NE	0.015	0.05	0.015
Pyrene	NE	0.015	0.05	0.015

Lab: ARA
Matrix: Soil
Method: SVOCs by EPA Method 8270D (SOP QA-5515)

Compound	PAL (µg/L)	PQL (µg/L)	LOQ (µg/L)	LOD (µg/L)
1,2,4-trichlorobenzene	70	35	5	1
1,2-dichlorobenzene	600	300	2	1
1,3-dichlorobenzene	NE	1	2	1
1,4-dichlorobenzene	75	37.5	2	1
1,4-dioxane	NE	50	50	50
2,4,5-trichlorophenol	NE	2	2	2
2,4,6-trichlorophenol	NE	2	2	2
2,4-dichlorophenol	NE	2	5	2
2,4-dimethylphenol	NE	2	2	2
2,4-dinitrophenol	NE	1	50	1
2,4-dinitrotoluene	NE	2	2	2
2,6-dinitrotoluene	NE	1	2	1
2-chloronaphthalene	NE	1	5	1
2-chlorophenol	NE	4	5	4
2-methylnaphthalene	NE	1	1	1
2-methylphenol	NE	2	2	2
2-nitroaniline	NE	1	2	1
2-nitrophenol	NE	2	2	2
3,3'-dichlorobenzidine	NE	20	30	20
3-nitroaniline	NE	1	2	1
4,6-dinitro-2-methylphenol	NE	4	20	4

Compound	PAL (µg/L)	PQL (µg/L)	LOQ (µg/L)	LOD (µg/L)
4-bromophenyl phenyl ether	NE	1	2	1
4-chloro-3-methylphenol	NE	2	2	2
4-chloroaniline	NE	1	2	1
4-chlorophenyl phenyl ether	NE	1	5	1
4-methylphenol	NE	2	2	2
4-nitroaniline	NE	4	5	4
4-nitrophenol	NE	4	10	4
Acenaphthene	NE	0.5	0.5	0.5
Acenaphthylene	NE	0.5	0.5	0.5
Aniline	NE	1	2	1
Anthracene	NE	0.5	0.5	0.5
Azobenzene	NE	1	2	1
Benzidine	NE	30	30	30
Benzo(a)anthracene	NE	0.5	0.5	0.5
Benzo(a)pyrene	0.2	**0.1***	0.2	0.2
Benzo(b)fluoranthene	NE	0.5	0.5	0.5
Benzo(g,h,i)perylene	NE	0.5	0.5	0.5
Benzo(k)fluoranthene	NE	0.5	0.5	0.5
Benzoic acid	NE	50	50	50
Benzyl alcohol	NE	1	2	1
Bis(2-chloroethoxy)methane	NE	1	5	1
Bis(2-chloroethyl)ether	NE	1	2	1
Bis(2-chloroisopropyl) ether	NE	1	2	1
Bis(2-ethylhexyl)phthalate	6.0	3.0	5	1
Butyl benzyl phthalate	NE	1	5	1
Carbazole	NE	1	2	1
Chrysene	NE	0.5	0.5	0.5
Dibenzo(a,h)anthracene	NE	0.5	0.5	0.5
Dibenzofuran	NE	0.5	0.5	0.5
Diethyl phthalate	NE	1	5	1
Dimethylphthalate	NE	1	5	1
Di-n-butylphthalate	NE	1	5	1
Di-n-octyl phthalate	NE	1	2	1
Fluoranthene	NE	0.5	0.5	0.5
Fluorene	NE	0.5	0.5	0.5
Hexachlorobenzene	1.0	**0.5**	2	1
Hexachlorobutadiene	NE	1	2	1
Hexachlorocyclopentadiene	50	25	10	1
Hexachloroethane	NE	1	2	1
Indeno(1,2,3-cd)pyrene	NE	0.5	0.5	0.5
Isophorone	NE	1	5	1
Naphthalene	NE	0.5	0.5	0.5
Nitrobenzene	NE	1	2	1
N-nitrosodimethylamine	NE	2	2	2
N-nitroso-di-N-propylamine	NE	1	2	1
N-nitrosodiphenylamine	NE	1	2	1

Compound	PAL (µg/L)	PQL (µg/L)	LOQ (µg/L)	LOD (µg/L)
Pentachlorophenol	**1.0**	**0.5**	10	4
Phenanthrene	NE	0.5	0.5	0.5
Phenol	NE	2	2	2
Pyrene	NE	1	0.5	1

*PQL achieved by PAH with SIM analysis

Lab: ARA
Matrix: Groundwater
Method: Metals by EPA Method 6020A (SOP QA-5605)

Compound	PAL (µg/L)	PQL (µg/L)	LOQ (µg/L)	LOD (µg/L)
Antimony	6	3	1	0.1
Arsenic	10	5	5	1
Beryllium	4	2	1	0.1
Cadmium	5	2.5	1	0.1
Chromium, total	100	50	10	0.5
$Chromium^{+3}$ (as total)	NE	NE		
Copper	1,300	650	10	0.1
Lead	15	7.5	5	0.1
Nickel	NE	0.1	10	0.1
Selenium	50	25	10	10
Silver	NE	0.05	5	0.05
Thallium	2	1	1	0.5
Zinc	NE	1	10	1

Lab: ARA
Matrix: Groundwater
Method: Mercury by EPA Method 7470A (SOP QA-5600)

Compound	PAL (µg/L)	PQL (µg/L)	LOQ (µg/L)	LOD (µg/L)
Mercury	2	1	0.2	0.1

Lab: ARA
Matrix: Groundwater
Method: Hexavalent chromium by SM 3500 Cr-B (SOP QA-5813)

Compound	PAL (µg/L)	PQL (µg/L)	LOQ (µg/L)	LOD (µg/L)
$Chromium^{+6}$	100	50	10	10

Lab: ARA
Matrix: Groundwater
Method: EPH by MADEP Method EPH-04-1.1 (SOP QA-5313)

Compound	PAL (µg/L)	PQL (µg/L)	LOQ (µg/L)	LOD (µg/L)
Naphthalene	100*	50	1	0.5
2-Methylnaphthalene	NE	0.5	1	0.5
Phenanthrene	NE	0.5	1	0.5
Acenaphthene	NE	0.5	1	0.5
Acenaphthylene	NE	0.5	1	0.5
Fluorene	NE	0.5	1	0.5
Anthracene	NE	0.5	1	0.5
Fluoranthene	NE	0.5	1	0.5
Pyrene	NE	0.5	1	0.5
Benzo(a)anthrancene	NE	0.5	1	0.5
Chrysene	NE	0.5	1	0.5
Benzo(b)fluoranthene	NE	0.5	1	0.5
Benzo(k)fluoranthene	NE	0.5	1	0.5
Benzo(a)pyrene	**0.2**	**0.1**	0.4	0.4
Indeno(1,2,3-cd) pyrene	NE	0.5	1	0.5
Dibenzo(a,h)anthracene	NE	0.5	1	0.5
Benzo(g,h,i)perylene	NE	0.5	1	0.5
C9-C18 Aliphatics	5,000^	2,500	100	100
C19-C36 Aliphatics	50,000^	25,000	100	100
C11-C22 Aromatics	5,000^	2,500	100	100

Lab: ARA
Matrix: Groundwater
Method: VPH by MADEP Method VPH-04-1.1 (SOP QA-5130)

Compound	PAL (µg/L)	PQL (µg/L)	LOQ (µg/L)	LOD (µg/L)
Methyl tert-butyl ether	NE	NE	2	0.5
Benzene	5	2.5	1	0.5
Toluene	1,000	500	2	0.5
Ethylbenzene	700	350	2	0.5
m&p Xylenes	10,000	5,000	2	1
o-Xylenes	10,000	5,000	2	0.5
Naphthalene	100*	50	5	0.03
C5-C8 Aliphatics	3,000^	1,500	100	100
C9-C12 Aliphatics	5,000^	2,500	100	100
C9-C10 Aromatics	4,000^	2,000	100	10

Lab: ARA
Matrix: Groundwater
Method: PCBs by EPA Method 8082A (SOP QA-5303)

Compound	PAL (µg/L)	PQL (µg/L)	LOQ (µg/L)	LOD (µg/L)
PCB-1016	Total 0.5	0.25	0.2	0.1
PCB-1221		0.25	0.2	0.1
PCB-1232		0.25	0.2	0.1
PCB-1242		0.25	0.2	0.1
PCB-1248		0.25	0.2	0.1
PCB-1254		0.25	0.2	0.1
PCB-1260		0.25	0.2	0.1

Lab: TAS
Matrix: Groundwater
Method: Explosives by EPA Method 8330B (SOP WS-LC-0009)

Compound	PAL (µg/L)	PQL (µg/L)	LOQ (µg/L)	LOD (µg/L)
1,3,5-trinitrobenzene	NE	0.1	0.15	0.1
1,3-dinitrobenzene	NE	0.1	0.15	0.1
2,4,6-trinitrotoluene (TNT)	NE	0.1	0.15	0.1
2,4-dinitrotoluene	NE	0.1	0.13	0.1
2,6-dinitrotoluene	NE	0.1	0.13	0.1
2-amino-4,6-dinitrotoluene	NE	0.1	0.15	0.1
4-amino-2,6-dinitrotoluene	NE	0.1	0.15	0.1
2-nitrotoluene	NE	0.2	0.5	0.2
3-nitrotoluene	NE	0.2	0.5	0.2
4-nitrotoluene	NE	0.2	0.5	0.2
nitrobenzene	NE	0.1	0.15	0.1
nitroglycerin	NE	0.75	1	0.75
pentaerythritol tetranitrate (PETN)	NE	0.75	1	0.75
hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	NE	0.1	0.15	0.1
trinitrophenylmethylnitramine (tetryl)	NE	0.1	0.15	0.1

Lab: ARA
Matrix: Groundwater
Method: Pesticides by EPA Method 8081B (SOP QA-5304)

Compound	PAL (µg/L)	PQL (µg/L)	LOQ (µg/L)	LOD (µg/L)
4,4'-DDD	NE	0.05	0.1	0.05
4,4'-DDE	NE	0.05	0.1	0.05
4,4'-DDT	NE	0.05	0.1	0.05
Aldrin	NE	0.05	0.1	0.05
alpha-BHC	NE	0.05	0.1	0.05
alpha-chlordane	2.0	1.0	0.1	0.05
gamma-chlordane	2.0	1.0	0.1	0.05
beta-BHC	NE	0.05	0.1	0.05
delta-BHC	NE	0.05	0.1	0.05
Dieldrin	NE	0.05	0.1	0.05
Endosulfan I	NE	0.05	0.1	0.05
Endosulfan II	NE	0.05	0.1	0.05
Endosulfan sulfate	NE	0.05	0.1	0.05
Endrin	2.0	1.0	0.1	0.05
Endrin ketone	NE	0.05	0.1	0.05
Endrin aldehyde	NE	0.05	0.1	0.05
gamma-BHC (Lindane)	0.2	0.1	0.1	0.05
Heptachlor	0.4	0.2	0.1	0.05
Heptachlor epoxide	0.2	0.1	0.1	0.05
Methoxychlor	40	20	0.1	0.05
Toxaphene	3.0	1.5	0.4	0.4

SEDIMENT

Sediment PALs are the consensus-based threshold effect concentration (TEC)/probable effect concentrations (PEC) criteria established by MacDonald *et al* (MacDonald *et al*, 2000). TECs, which are the lower of the two values are shown below as the PAL. PALs and/or PQLs that cannot be achieved by the selected analytical laboratory/method are highlighted bold. For these compounds the LOD will be considered the PQL until they are identified as a Site-specific COPC, at which time alternative methods will be evaluated. Laboratory LODs may vary as new studies are performed and limits are updated.

Lab: ARA
Matrix: Sediment
Method: VOCs by EPA Method 8260C (SOP QA-5120)

Compound	PAL (mg/kg)	PQL (mg/kg)	LOQ (mg/kg)	LOD (mg/kg)
1,1,1,2-tetrachloroethane	NE	0.025	0.1	0.025
1,1,1-trichloroethane	NE	0.025	0.1	0.025
1,1,2,2-tetrachloroethane	NE	0.025	0.1	0.025
1,1,2-trichloroethane	NE	0.025	0.1	0.025
1,1-dichloroethane	NE	0.025	0.1	0.025
1,1-dichloroethene	NE	0.025	0.1	0.025
1,1-dichloropropene	NE	0.025	0.1	0.025

Compound	PAL (mg/kg)	PQL (mg/kg)	LOQ (mg/kg)	LOD (mg/kg)
1,2,3-trichlorobenzene	NE	0.025	0.1	0.025
1,2,3-trichloropropane	NE	0.025	0.1	0.025
1,2,4-trichlorobenzene	NE	0.025	0.1	0.025
1,2,4-trimethylbenzene	NE	0.025	0.1	0.025
1,2-dibromo-3-chloropropane (DBCP)	NE	0.1	0.1	0.1
1,2-dibromoethane (EDB)	NE	0.025	0.1	0.025
1,2-dichlorobenzene	NE	0.025	0.1	0.025
1,2-dichloroethane	NE	0.025	0.1	0.025
1,2-dichloropropane	NE	0.025	0.1	0.025
1,3,5-trimethylbenzene	NE	0.025	0.1	0.025
1,3-dichlorobenzene	NE	0.025	0.1	0.025
1,3-dichloropropane	NE	0.025	0.1	0.025
1,4-dichlorobenzene	NE	0.025	0.1	0.025
1,4-dioxane	NE	???	2.5	???
2,2-dichloropropane	NE	0.025	0.1	0.025
2-butanone (MEK)	NE	0.025	0.3	0.025
2-chlorotoluene	NE	0.025	0.1	0.025
2-hexanone	NE	0.025	0.5	0.025
4-chlorotoluene	NE	0.025	0.1	0.025
4-isopropyltoluene	NE	0.025	0.1	0.025
4-methyl-2-pentanone (MIBK)	NE	0.025	0.45	0.025
acetone	NE	0.2	2.5	0.2
benzene	NE	0.025	0.1	0.025
bromobenzene	NE	0.025	0.1	0.025
bromochloromethane	NE	0.025	0.1	0.025
bromodichloromethane	NE	0.025	0.1	0.025
bromoform	NE	0.025	0.1	0.025
bromomethane	NE	0.025	0.25	0.025
carbon disulfide	NE	0.025	0.1	0.025
carbon tetrachloride	NE	0.025	0.1	0.025
chlorobenzene	NE	0.025	0.1	0.025
chloroethane	NE	0.025	0.1	0.025
chloroform	NE	0.025	0.1	0.025
chloromethane	NE	0.025	0.1	0.025
cis-1,2-dichloroethene	NE	0.025	0.1	0.025
cis-1,3-dichloropropene	NE	0.025	0.1	0.025
dibromochloromethane	NE	0.025	0.1	0.025
dibromomethane	NE	0.025	0.1	0.025
dichlorodifluoromethane	NE	0.025	0.1	0.025
diethyl ether	NE	0.025	0.1	0.025
ethylbenzene	NE	0.025	0.1	0.025
hexachlorobutadiene	NE	0.025	0.1	0.025
isopropylbenzene	NE	0.025	0.1	0.025
m&p-xylenes	NE	0.05	0.1	0.05

Compound	PAL (mg/kg)	PQL (mg/kg)	LOQ (mg/kg)	LOD (mg/kg)
methyl t-butyl ether (MTBE)	NE	0.025	0.1	0.025
methylene chloride	NE	0.025	0.1	0.025
naphthalene	NE	0.025	0.1	0.025
n-butylbenzene	NE	0.025	0.1	0.025
n-propylbenzene	NE	0.025	0.1	0.025
o-xylene	NE	0.025	0.1	0.025
sec-butylbenzene	NE	0.025	0.1	0.025
styrene	NE	0.025	0.1	0.025
tert-butylbenzene	NE	0.025	0.1	0.025
tetrachloroethene	NE	0.025	0.1	0.025
tetrahydrofuran (THF)	NE	0.2	0.5	0.2
toluene	NE	0.025	0.1	0.025
trans-1,2-dichloroethene	NE	0.025	0.1	0.025
trans-1,3-dichloropropene	NE	0.025	0.1	0.025
trichloroethene	NE	0.025	0.1	0.025
trichlorofluoromethane	NE	0.025	0.1	0.025
vinyl chloride	NE	0.025	0.1	0.025

Lab: TAS
Matrix: Sediment
Method: PAHs by EPA Method 8270D with SIM (SOP WS-MS-0008)

Compound	PAL (mg/kg)	PQL (mg/kg)	LOQ (mg/kg)	LOD (mg/kg)
1-Methylnaphthalene	NE	0.001	0.005	0.001
2-Methylnaphthalene	NE	0.001	0.005	0.001
Acenaphthene	NE	0.001	0.005	0.001
Acenaphthylene	NE	0.001	0.005	0.001
Anthracene	0.0572	0.0286	0.005	0.001
Benzo[a]anthracene	0.108	0.054	0.005	0.001
Benzo[a]pyrene	0.150	0.075	0.005	0.001
Benzo[b]fluoranthene	NE	0.002	0.005	0.002
Benzo[g,h,i]perylene	NE	0.003	0.005	0.003
Benzo[k]fluoranthene	NE	0.002	0.005	0.002
Chrysene	0.166	0.083	0.005	0.001
Dibenz(a,h)anthracene	0.033	0.0165	0.005	0.003
Fluoranthene	0.423	0.2115	0.005	0.001
Fluorene	0.0774	0.0387	0.005	0.001
Indeno[1,2,3-cd]pyrene	NE	0.001	0.005	0.001
Naphthalene	0.176	0.088	0.005	0.001
Phenanthrene	0.204	0.102	0.005	0.001
Pyrene	0.195	0.0975	0.005	0.001
Total	1.61	NA	0.005	0.001

Lab: ARA
Matrix: Sediment
Method: SVOCs by EPA Method 8270D (SOP QA-5515)

Compound	PAL (mg/kg)	PQL (mg/kg)	LOQ (mg/kg)	LOD (mg/kg)
1,2,4-trichlorobenzene	NE	0.20	0.5	0.20
1,2-dichlorobenzene	NE	0.20	0.2	0.20
1,3-dichlorobenzene	NE	0.20	0.2	0.20
1,4-dichlorobenzene	NE	0.20	0.2	0.20
1,4-dioxane	NE	2	2	2
2,4,5-trichlorophenol	NE	0.20	0.2	0.20
2,4,6-trichlorophenol	NE	0.20	0.2	0.20
2,4-dichlorophenol	NE	0.20	0.5	0.20
2,4-dimethylphenol	NE	0.20	0.2	0.20
2,4-dinitrophenol	NE	0.20	5	0.20
2,4-dinitrotoluene	NE	0.20	0.2	0.20
2,6-dinitrotoluene	NE	0.20	0.2	0.20
2-chloronaphthalene	NE	0.20	0.5	0.20
2-chlorophenol	NE	0.20	0.5	0.20
2-methylnaphthalene	NE	0.05	0.05	0.05
2-methylphenol	NE	0.20	0.2	0.20
2-nitroaniline	NE	0.20	0.2	0.20
2-nitrophenol	NE	0.20	0.2	0.20
3,3'-dichlorobenzidine	NE	2	3	2
3-nitroaniline	NE	0.20	0.2	0.20
4,6-dinitro-2-methylphenol	NE	0.40	2	0.40
4-bromophenyl phenyl ether	NE	0.20	0.2	0.20
4-chloro-3-methylphenol	NE	0.20	0.2	0.20
4-chloroaniline	NE	0.20	0.2	0.20
4-chlorophenyl phenyl ether	NE	0.20	0.5	0.20
4-methylphenol	NE	0.20	0.2	0.20
4-nitroaniline	NE	0.20	0.5	0.20
4-nitrophenol	NE	0.40	2	0.40
Acenaphthene	NE	0.05	0.05	0.05
Acenaphthylene	NE	0.05	0.05	0.05
Aniline	NE	0.20	0.2	0.20
Anthracene	0.0572	**0.0286***	0.05	0.05
Azobenzene	NE	0.20	0.2	0.20
Benzidine	NE	0.2	3	0.2
Benzo(a)anthracene	0.108	0.054	0.05	0.05
Benzo(a)pyrene	0.150	0.075	0.05	0.05
Benzo(b)fluoranthene	NE	0.05	0.05	0.05
Benzo(g,h,i)perylene	NE	0.05	0.05	0.05
Benzo(k)fluoranthene	NE	0.05	0.05	0.05
Benzoic acid	NE	0.2	5	0.2
Benzyl alcohol	NE	0.20	0.2	0.20
Bis(2-chloroethoxy)methane	NE	0.20	0.2	0.20
Bis(2-chloroethyl)ether	NE	0.20	0.2	0.20

Compound	PAL (mg/kg)	PQL (mg/kg)	LOQ (mg/kg)	LOD (mg/kg)
Bis(2-chloroisopropyl) ether	NE	0.20	0.2	0.20
Bis(2-ethylhexyl)phthalate	NE	0.20	0.5	0.20
Butyl benzyl phthalate	NE	0.20	0.5	0.20
Carbazole	NE	0.20	0.2	0.20
Chrysene	0.166	0.083	0.05	0.05
Dibenzo(a,h)anthracene	**0.033**	**0.0165***	0.05	0.05
Dibenzofuran	NE	0.05	0.05	0.05
Diethyl phthalate	NE	0.20	0.5	0.20
Dimethylphthalate	NE	0.20	0.5	0.20
Di-n-butylphthalate	NE	0.20	0.5	0.20
Di-n-octyl phthalate	NE	0.20	0.5	0.20
Fluoranthene	0.423	0.2115	0.05	0.05
Fluorene	0.0774	**0.0387***	0.05	0.05
Hexachlorobenzene	NE	0.20	0.2	0.20
Hexachlorobutadiene	NE	0.20	0.2	0.20
Hexachlorocyclopentadiene	NE	0.20	1	0.20
Hexachloroethane	NE	0.20	0.2	0.20
Indeno(1,2,3-cd)pyrene	NE	0.05	0.05	0.05
Isophorone	NE	0.20	0.5	0.20
Naphthalene	0.176	0.088	0.05	0.05
Nitrobenzene	NE	0.20	0.2	0.20
N-nitrosodimethylamine	NE	0.20	0.2	0.20
N-nitroso-di-N-propylamine	NE	0.20	0.2	0.20
N-nitrosodiphenylamine	NE	0.20	0.2	0.20
Pentachlorophenol	NE	0.40	1	0.40
Phenanthrene	0.204	0.102	0.05	0.05
Phenol	NE	0.20	0.2	0.20
Pyrene	0.195	0.0975	0.05	0.05

*PQL achieved by PAH with SIM analysis

Lab: ARA
Matrix: Sediment
Method: Metals by EPA Method 6020A (SOP QA-5605)

Compound	PAL (mg/kg)	PQL (mg/kg)	LOQ (mg/kg)	LOD (mg/kg)
Antimony	NE	0.05	0.5	0.05
Arsenic	9.79	4.85	2.5	0.5
Beryllium	NE	0.05	0.5	0.05
Cadmium	0.99	0.495	0.5	0.05
Chromium, total	43.4	21.7	5	0.05
$Chromium^{+3}$	NE	0.05		
Copper	31.6	15.8	5	0.05
Lead	35.8	17.9	2.5	0.05
Nickel	22.7	11.35	5	0.05
Selenium	NE	0.5	5	0.5
Silver	NE	0.03	2.5	0.03
Thallium	NE	0.03	0.5	0.03
Zinc	121	60.5	5	0.5

Lab: ARA
Matrix: Sediment
Method: Mercury by EPA Method 7471B (SOP QA-5600)

Compound	PAL (mg/kg)	PQL (mg/kg)	LOQ (mg/kg)	LOD (mg/kg)
Mercury	0.18	0.09	0.02	0.016

Lab: ARA
Matrix: Sediment
Method: Hexavalent chromium by EPA Method 7196A (SOP QA-5813)

Compound	PAL (mg/kg)	PQL (mg/kg)	LOQ (mg/kg)	LOD (mg/kg)
$Chromium^{+6}$	NE	0.4	0.4	0.4

Lab: ARA
Matrix: Sediment
Method: EPH by MADEP Method EPH-04-1.1 (SOP QA-5313)

Compound	PAL (mg/kg)	PQL (mg/kg)	LOQ (mg/kg)	LOD (mg/kg)
Naphthalene	0.176	**0.088**	0.2	0.1
2-Methylnaphthalene	NE	0.1	0.2	0.1
Phenanthrene	0.204	0.102	0.2	0.1
Acenaphthene	NE	0.1	0.2	0.1
Acenaphthylene	NE	0.1	0.2	0.1
Fluorene	**0.0774**	**0.0387**	0.2	0.1
Anthracene	**0.0572**	**0.0286**	0.2	0.1
Fluoranthene	0.423	0.2115	0.2	0.1
Pyrene	0.195	**0.0975**	0.2	0.1
Benzo(a)anthrancene	0.108	**0.054**	0.2	0.1
Chrysene	0.166	**0.083**	0.2	0.1
Benzo(b)fluoranthene	NE	0.1	0.2	0.1
Benzo(k)fluoranthene	NE	0.1	0.2	0.1
Benzo(a)pyrene	0.150	**0.075**	0.2	0.1
Indeno(1,2,3-cd) pyrene	NE	0.1	0.2	0.1
Dibenzo(a,h)anthracene	**0.033**	**0.0165**	0.2	0.1
Benzo(g,h,i)perylene	NE	0.1	0.2	0.1
C9-C18 Aliphatics	NE	6	20	6
C19-C36 Aliphatics	NE	10	20	10
C11-C22 Aromatics	NE	6	20	6

Lab: ARA
Matrix: Sediment
Method: VPH by MADEP Method VPH-04-1.1 (SOP QA-5130)

Compound	PAL (mg/kg)	PQL (mg/kg)	LOQ (mg/kg)	LOD (mg/kg)
Methyl tert-butyl ether	NE	0.05	0.1	0.05
Benzene	5.0	2.5	0.1	0.05
Toluene	1,000	500	0.1	0.05
Ethylbenzene	700	350	0.1	0.05
m&p Xylenes	Total 1,000	500	0.1	0.05
o-Xylenes		500	0.1	0.05
Naphthalene	NE	0.05	0.2	0.05
C5-C8 Aliphatics	NE	2	4	2
C9-C12 Aliphatics	NE	3	4	3
C9-C10 Aromatics	NE	0.4	4	0.4

Lab: ARA
Matrix: Sediment
Method: PCBs by EPA Method 8082A (SOP QA-5303)

Compound	PAL (mg/kg)	PQL (mg/kg)	LOQ (mg/kg)	LOD (mg/kg)
PCB-1016	Total 0.0598	**0.0299**	0.2	0.04
PCB-1221			0.2	0.04
PCB-1232			0.2	0.04
PCB-1242			0.2	0.04
PCB-1248			0.2	0.04
PCB-1254			0.2	0.04
PCB-1260			0.2	0.04

Lab: TAS
Matrix: Sediment
Method: Explosives by EPA Method 8330B (SOP WS-LC-0009)

Compound	PAL (mg/kg)	PQL (mg/kg)	LOQ (mg/kg)	LOD (mg/kg)
1,3,5-trinitrobenzene	NE	0.05	0.25	0.05
1,3-dinitrobenzene	NE	0.05	0.25	0.05
2,4,6-trinitrotoluene (TNT)	NE	0.05	0.25	0.05
2,4-dinitrotoluene	NE	0.05	0.25	0.05
2,6-dinitrotoluene	NE	0.05	0.25	0.05
2-amino-4,6-dinitrotoluene	NE	0.05	0.25	0.05
4-amino-2,6-dinitrotoluene	NE	0.05	0.25	0.05
2-nitrotoluene	NE	0.05	0.25	0.05
3-nitrotoluene	NE	0.05	0.25	0.05
4-nitrotoluene	NE	0.05	0.25	0.05
nitrobenzene	NE	0.05	0.25	0.05
nitroglycerin	NE	0.25	0.5	0.25
pentaerythritol tetranitrate (PETN)	NE	0.25	0.5	0.25
hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	NE	0.05	0.25	0.05
trinitrophenylmethylnitramine (tetryl)	NE	0.05	0.25	0.05

Lab: ARA
Matrix: Sediment
Method: Pesticides by EPA Method 8081B (SOP QA-5304)

Compound	PAL (mg/kg)	PQL (mg/kg)	LOQ (mg/kg)	LOD (mg/kg)
4,4'-DDD	**0.00488**	**0.00244**	0.04	0.03
4,4'-DDE	**0.00316**	**0.00158**	0.04	0.03
4,4'-DDT	**0.00416**	**0.00208**	0.04	0.03
Aldrin	NE	0.03	0.04	0.03
alpha-BHC	NE	0.03	0.04	0.03
alpha-chlordane	**0.00324**	**0.00162**	0.04	0.03
gamma-chlordane	**0.00324**	**0.00162**	0.04	0.03
beta-BHC	NE	0.03	0.04	0.03
delta-BHC	NE	0.03	0.04	0.03
Dieldrin	**0.00190**	**0.00095**	0.04	0.03
Endosulfan I	NE	0.03	0.04	0.03

Compound	PAL (mg/kg)	PQL (mg/kg)	LOQ (mg/kg)	LOD (mg/kg)
Endosulfan II	NE	0.03	0.04	0.03
Endosulfan sulfate	NE	0.03	0.04	0.03
Endrin	**0.00222**	**0.00111**	0.04	0.03
Endrin ketone	NE	0.03	0.04	0.03
Endrin aldehyde	NE	0.03	0.04	0.03
gamma-BHC (Lindane)	**0.00237**	**0.001185**	0.04	0.03
Heptachlor	NE	0.03	0.04	0.03
Heptachlor epoxide	**0.00247**	**0.001235**	0.04	0.03
Methoxychlor	NE	0.03	0.04	0.03
Toxaphene	NE	0.2	0.2	0.2

Lab: ARA
Matrix: Sediment
Method: pH by EPA Method 9045C (SOP QA-5851)

Compound	PAL	PQL	LOQ	LOD
pH	6-8	NA	0-14	0-14

WORKSHEET #17 – SAMPLING DESIGN AND RATIONALE

Credere has designed the following sampling program to meet the objectives outlined in **Worksheet #11** and in accordance with the PWS (CENAE, 2017). Tasks outline below will be completed in the late winter/spring of 2018 if review and approval of the FSP and QAPP allow, based on the project Schedule outlined in **Worksheets #14 & #16**.

The physical boundaries for the area under study are depicted on **Figure 2**. Data gaps for each specific AOC within the Site that require additional assessment are identified in the **Appendix B** Conceptual Site Model and Data Gap Analysis table. The data gaps identified below as rationale (See "Data Gap Justifying Sampling" column in below table) are justified in **Appendix B**. These specific NED Sites were designated during an early investigation and have been carried through for consistency. Each specific NED Site No. has a unique set of COPCs, physical conditions, and COPC mechanics that warrant individual as opposed to Site-wide assessment. This investigation will seek to identify more definitive boundaries for each NED Site. Proposed sampling locations for each NED Site No. and proposed methodology are summarized below.

GEOPHYSICS

Credere will subcontract Hager-Richter Geosciences (HRG) of Salem, New Hampshire, to perform GPR, magnetometry, and metal detection at the Site to clear all subsurface exploration locations for utilities and other anomalies, and to confirm the previously surveyed boundaries of Disposal Area #14 (NED Site No. 10) in the field for confirmation of the georeferenced location with a global positioning system (GPS) handheld unit. Any identified anomalies will be marked in the field and avoided during sampling activities unless field conditions (e.g., availability of an excavator associated with Site clearing that can penetrate frozen ground during the geophysical mobilization) allow for exploration of anomalies. If suspected MC are identified, an UXO specialist will be contacted to identify the suspect material.

CONTAINER CONSOLIDATION

During RI activities, any encountered containers containing liquid or solid will be overpacked, if needed, and consolidated by NRC of Franklin, Massachusetts. Where empty containers are encountered, their location will be marked for further evaluation/sampling. Empty containers will be consolidated to a single area on the island to be removed and recycled as scrap to reduce the debris present and facilitate future investigation activities. Any solid and liquid waste will be characterized for disposal according to disposal facility requirements and in accordance with RIDEM's Rules and Regulations for Hazardous Waste Management (RIDEM, 2016).

SOIL AND CONCRETE

The following table summarizes the sample design and rationale for soil and concrete samples. Soil samples will generally be collected from soil borings advanced by GeoSearch, Inc. of Fitchburg, Massachusetts, or collected using hand tools. Concrete samples will be collected using hand tools. Specific sample details for each sample ID listed below are provided in **Worksheet #18**. Sample locations or DUs are depicted on **Figures 4A through 4C**. If proposed locations are not achievable due to field constraints, the field team will communicate these limitations according to **Worksheet #6** to evaluate changes or elimination to the design.

NED Site No.	Data Gap Justifying Sampling	Sample Type	Sample ID	Proposed Analyses	Sample Decision Process
1 Coal Storage North	Nature and extent of previously identified surface contamination in DUs for average exposure assessment	ISM	01DU1-1	PAHs, metals, EPH	Source Unit. 01DU1 is depicted on **Figure 4A** and will consist of 30 0 to 1-foot aliquots per replicate
			01DU1-2		
			01DU1-3		
			01DU2-1		01DU2 is depicted on **Figure 4A** and will consist of 30 0 to 1-foot aliquots per replicate
			01DU2-2		
			01DU2-3		
			01DU3-1		01DU3 is depicted on **Figure 4A** and will consist of 30 0 to 1-foot aliquots per replicate
			01DU3-2		
			01DU3-3		
	Horizontal and vertical extent of coal in soil by visual examination and discrete sampling for comparison to UCLs and comparability of ISM and discrete samples	Soil boring/ Grab	01SB1-1	PAHs, metals, EPH	Sample to be collected from the entire thickness of observed coal up to a 4 foot interval (e.g., 0 to 4 feet bgs). If greater than 4 feet of coal is encountered then 2 samples will be collected (e.g., 4 to 8 feet bgs, etc.).
			01SB1-2*		
			01SB2-1		
			01SB2-2*		
			01SB3-1		
			01SB3-2*		
			01SB4-1		
			01SB4-2*		
			01SB5-1		
			01SB5-2*		
			01SB6-1		
			01SB6-2*		
			01SB7-1		
			01SB7-2*		
			01SB8-1		
			01SB8-2*		
			01SB9-1		
			01SB9-2*		
			01SB10-1		
			01SB10-2*		
4 Pump House	Removal of drum and confirmatory sampling	Composite	04CP-1	VOCs, SVOCs, EPH, VPH, PCBs, metals	16 point composite to be collected around pump house on a 10-foot grid with aliquots bias to drum locations. If drums are no longer present, the aliquots will be evenly spaced.

NED Site No.	Data Gap Justifying Sampling	Sample Type	Sample ID	Proposed Analyses	Sample Decision Process
8 Incinerator #49	Assessment of incinerator floor debris/ equipment contents	Grab	08W-1*	PAHs, Dioxins/furans, metals, PCBs	Samples to be collected from incinerator ash on the building floor, in the stack, or other incinerator equipment. Samples will not be collected if no floor debris or equipment remains.
			08W-2*		
			08W-3*		
			08W-4*		
	Assessment of perimeter soil for average surface soil exposure	ISM	08DU1-1		Source Unit. 08DU1 is depicted on **Figure 4B** east of the incinerator and will consist of 30 0 to 1-foot aliquots per replicate
			08DU1-2		
			08DU1-3		
			08DU2-1		08DU2 is depicted on **Figure 4B** north of the incinerator and will consist of 30 0 to 1-foot aliquots
			08DU2-2		
			08DU2-3		
			08DU3-1		08DU3 is depicted on **Figure 4B** south of the incinerator and will consist of 30 0 to 1-foot aliquots
			08DU3-2		
			08DU3-3		
			08DU4-1		08DU4 is depicted on **Figure 4B** west of the incinerator and will consist of 30 0 to 1-foot aliquots
			08DU4-2		
			08DU4-3		
	Assessment of perimeter soil from discrete locations for comparison to UCLs and assess comparability of discrete and ISM samples	Soil boring/ Grab	08SB1-1		Will be sampled from interval of observable contamination or 0-1 foot
			08SB2-1		
			08SB3-1		
			08SB4-1		
			08SB5-1		
			08SB6-1		
			08SB7-1		
9 Magazine Ignitor Storage #37	Assessment of soil	See NED Site No. 48			

NED Site No.	Data Gap Justifying Sampling	Sample Type	Sample ID	Proposed Analyses	Sample Decision Process
10 Disposal Area #14	Vertical delineation and characterization of waste	Soil boring	10SB1-1	Explosives, VOCs, SVOCs, EPH, VPH, pH, PCBs, metals, pesticides	Continuous sampling in 2 foot interval through waste material.
			10SB1-2*,^		
			10SB1-3*, ^		
			10SB1-4*,^		Greatest observed contamination beyond waste material, if any. If no evidence of contamination (e.g., PID response or free product) then no samples will be collected.
			10SB1-5*,^		2-foot interval above bedrock refusal
		Soil boring	10SB2-1	Explosives, VOCs, SVOCs, EPH, VPH, pH, metals, PCBs, pesticides	Continuous sampling in 2 foot interval through waste material.
			10SB2-2*,^		
			10SB2-3*, ^		
			10SB2-4*,^		Greatest observed contamination beyond waste material, if any. If no evidence of contamination (e.g., PID response or free product) then no samples will be collected.
			10SB2-5*,^		2-foot interval above bedrock refusal
		Soil boring	10SB3-1	Explosives, VOCs, SVOCs, EPH, VPH, pH, PCBs, metals, pesticides	Continuous sampling in 2 foot interval through waste material.
			10SB3-2*,^		
			10SB3-3*, ^		
			10SB3-4*,^		Greatest observed contamination beyond waste material, if any. If no evidence of contamination (e.g., PID response or free product) then no samples will be collected.
			10SB3-5*,^		2-foot interval above bedrock refusal
		Soil boring	10SB4-1	Explosives, VOCs, SVOCs, EPH, VPH, pH, PCBs, metals, pesticides	Continuous sampling in 2 foot interval through waste material.
			10SB4-2*,^		
			10SB4-3*, ^		
			10SB4-4*,^		Greatest observed contamination beyond waste material, if any. If no evidence of contamination (e.g., PID response or free product) then no samples will be collected.
			10SB4-5*,^		2-foot interval above bedrock refusal
		Soil boring	10SB5-1	Explosives, VOCs, SVOCs, EPH, VPH, pH, PCBs, metals, pesticides	Continuous sampling in 2 foot interval through waste material.
			10SB5-2*,^		
			10SB5-3*, ^		
			10SB5-4*,^		Greatest observed contamination beyond waste material, if any. If no evidence of contamination (e.g., PID response or free product) then no samples will be collected.
			10SB5-5*,^		2-foot interval above bedrock refusal

NED Site No.	Data Gap Justifying Sampling	Sample Type	Sample ID	Proposed Analyses	Sample Decision Process
10 Disposal Area #14 (cont.)	Assessment of impacts to the beach and extent of previously observed tar-like material	Test pits	10TP1-1	Explosives, VOCs, SVOCs, EPH[1], VPH[1], pH, PCBs, metals, pesticides	One sample from each test pit from the greatest observed contamination (i.e., PID response, staining, etc.). In the absence of evidence of contamination, sample will be collected from 0 to 2 feet bgs
			10TP2-1		
			10TP3-1		
			10TP4-1		
			10TP5-1		
			10TP5-2		2-foot interval above refusal or confining layer
			10TP6-1		One sample from each test pit from the greatest observed contamination (i.e., PID response, staining, etc.). In the absence of evidence of contamination, sample will be collected from 0 to 2 feet bgs
			10TP7-1		
			10TP8-1		
			10TP8-2		2-foot interval above refusal or confining layer
			10TP9-1		One sample from each test pit from the greatest observed contamination (i.e., PID response, staining, etc.). In the absence of evidence of contamination, sample will be collected from 0 to 2 feet bgs
			10TP10-1		
			10TP11-1		
			10TP12-1		
			10TP13-1		
11 Quonset/ Maintenance Shops	Assessment of heating and maintenance related impacts	ISM	11DU1-1	SVOCs, EPH, metals	11DU1 shown on **Figure 4A** and will consist of 50 0 to 1-foot aliquots per replicate
			11DU1-2		
			11DU1-3		
		Grab	11SS-1	VOCs, SVOCs, EPH, VPH, metals	Grab sample from three aliquot locations showing evidence of contamination (i.e., staining, PID response, presence of drums/containers in vicinity, etc.)
			11SS-2		
			11SS-3		

NED Site No.	Data Gap Justifying Sampling	Sample Type	Sample ID	Proposed Analyses	Sample Decision Process
12 Maintenance Shop/ Garage/ Fire Station	Assessment of extent of spilled transformer contents[2]	Grabs	12SS-1	PCBs, VPH, EPH	A 3-meter grid will be used to collect 9 soil/debris (0-0.5 feet) and 9 concrete samples (0-0.5 inches) around the spilled transformer contents in accordance with 40 CFR 761.265
			12CC-1		
			12SS-2		
			12CC-2		
			12SS-3		
			12CC-3		
			12SS-4		
			12CC-4		
			12SS-5		
			12CC-5		
			12SS-6		
			12CC-6		
			12SS-7		
			12CC-7		
			12SS-8		
			12CC-8		
			12SS-9		
			12CC-9		
	To assess impacts from the vehicle maintenance pit	Soil boring	12SB1-1	PCBs, EPH, VPH, VOCs, SVOCs, metals	Sample from greatest observed contamination or groundwater interface in the absence of contamination
		Grab	12W-1		Three samples to be collected from sludge or debris at base of maintenance pit.
			12W-2		
			12W-3		
	To assess around suspected UST	Soil boring	12SB2-1		Sample collected from greatest observed contamination or groundwater interface in absence of contamination
			12SB3-1		
			12SB4-1		
13 Electric Substation/ transformer Pen[2]	To assess impacts to concrete transformer pad	Concrete	13CC-1	PCBs	4 samples of top 0.5 inch of concrete transformer pad. Sample locations biased to areas of staining.
			13CC-2		
			13CC-3		
			13CC-4		
	To assess impacts surrounding transformer pad	Grab	13SS-3	PCBs, EPH, VPH, metals	4 samples of top 6 inches on each side of the transformer pad
			13SS-4		
			13SS-5		
			13SS-6		
16 Barracks	To assess beneath each transformer[2]	Grab	16T-1	PCBs, EPH, VPH	Additional samples may be warranted based on number of transformers identified. Samples biased to areas of staining.
			16T-2		
			16T-3*		
	Assessment of extent of spilled transformer contents[2]		16SS-1*	PCBs, VPH, EPH	Contingent on evidence of a release from buried transformers. A 3-meter grid will be used to collect 16 soil samples around the spilled transformer contents in accordance with 40 CFR 761.265
			16SS-2*		
			16SS-3*		
			16SS-4*		
			16SS-5*		
			16SS-6*		
			16SS-7*		
			16SS-8*		
			16SS-9*		
			16SS-10*		
			16SS-11*		
			16SS-12*		
			16SS-13*		
			16SS-14*		
			16SS-15*		
			16SS-16*		

NED Site No.	Data Gap Justifying Sampling	Sample Type	Sample ID	Proposed Analyses	Sample Decision Process
19 Torch Pot Storage #51	Confirm soil concentrations in vicinity of drum	ISM	19DU1-1 19DU1-2 19DU1-3	EPH	19DU1 shown on **Figure 4A** and will consist of 30 0 to 1-foot aliquots per replicate
21 Bunker #11	To assess within bunker for impacts associated with use as a firing range and possible releases from stored drums within the bunker	Grab	21SS-3* 21SS-4* 21SS-5* 21SS-6* 21SS-7*	EPH, VPH, metals, PCBs, explosives, VOCs, SVOCs, pesticides	To be collected of floor debris or 0-2 feet bgs within bunker depending if there is a concrete floor. SS-3 through SS-5 will be biased towards firing target end of the bunker (i.e., east or west end). Samples contingent upon removal of collapsed bunker roof and removal of other hazards identified beneath collapsed debris.
	Perimeter soil sampling to assess migration of contaminants possibly released to bunker	Soil boring/ Grab	21SB1-1 21SB2-1 21SB3-1 21SB4-1 21SB-5-1 21SB-6-1	EPH, VPH, metals, PCBs, explosives, VOCs, SVOCs, pesticides	One sample from each boring from the greatest observed contamination (i.e., PID response, staining, etc.). In the absence of contamination, sample to be collected from groundwater interface or 2 foot interval above refusal.
22 Bunker #12	To assess within the bunker for impacts associated with possible release from drums stored within the bunker.	Grab	22SS-3* 22SS-4* 22SS-5*	EPH, VPH, metals, PCBs, explosives, VOCs, SVOCs, pesticides	To be collected of floor debris or 0-2 feet bgs within bunker depending if there is a concrete floor. Samples contingent upon removal of collapsed bunker roof and removal of other hazards identified beneath collapsed debris.
	Perimeter soil sampling to assess migration of contaminants possibly released to bunker	Soil boring/ Grab	22SB1-1 22SB2-1 22SB3-1 22SB4-1 22SB-5-1 22SB-6-1	EPH, VPH, metals, PCBs, explosives, VOCs, SVOCs, pesticides	One sample from each boring from the greatest observed contamination (i.e., PID response, staining, etc.). In the absence of contamination, sample to be collected from groundwater interface or 2-foot interval above refusal.

NED Site No.	Data Gap Justifying Sampling	Sample Type	Sample ID	Proposed Analyses	Sample Decision Process
23 Coal Storage South	Nature and extent of previously identified surface contamination	ISM	23DU1-1	PAHs, metals, EPH	Source Unit. 23DU1 is depicted on **Figure 4A** and will consist of 50 0 to 1-foot aliquots per replicate
			23DU1-2		
			23DU1-3		
			23DU2-1		23DU2 is depicted on **Figure 4A** and will consist of 50 0 to 1-foot aliquots per replicate
			23DU2-2		
			23DU2-3		
	Horizontal and vertical extent of coal in soil by visual examination	Soil boring/ Grab	23SB1-1	PAHs, metals, EPH	Sample to be collected from the entire thickness of observed coal up to a 4-foot interval (e.g., 0 to 4 feet bgs). If greater than 4 feet of coal is encountered the -2 samples will be collected (e.g., 4 to 8, 4 to 6 feet bgs, etc.).
			23SB1-2*		
			23SB2-1		
			23SB2-2*		
			23SB3-1		
			23SB3-2*		
			23SB4-1		
			23SB4-2*		
			23SB5-1		
			23SB5-2*		
			23SB6-1		
			23SB6-2*		
			23SB7-1		
			23SB7-2*		
			23SB8-1		
			23SB8-2*		
24 Cable Terminal Bldg #16	Assess impacts from former transformer[2]	Concrete grab	24CC-1	PCBs	Sample top 0.5 inch of concrete transformer pad. Sample location biased to area of staining.
		Grab	24SS-2	PCBs, EPH, VPH	4 samples of top 6 inches on each side of the transformer pad
		Grab	24SS-3		
		Grab	24SS-4		
		Grab	24SS-5		
27 & 31 Three Gas Pits	Assess contents of gas pits and possible release to the subsurface	Sludge Grab	27SL-1	VOCs, SVOCs, EPH, VPH, metals, PCBs	Sludge grab sample from each of three gas pits. Two 27 pits located on east side of concrete pad, one 31 pit located on west side of pad.
			27SL-2		
			31SL-1		
		Soil boring/grab	27SB1-1		One sample from each boring from the greatest observed contamination (i.e., PID response, staining, etc.). In the absence of contamination, sample to be collected from groundwater interface or 2 foot interval above refusal.
			27SB2-1		
			31SB1-1		

NED Site No.	Data Gap Justifying Sampling	Sample Type	Sample ID	Proposed Analyses	Sample Decision Process
28, 30, 32, 33, 43, 44 General Aviation Pad	Generally assess soil beneath the concrete aviation pad	Soil boring/ grab	APSB1-1	VOCs, SVOCs, explosives, PCBs, EPH, VPH, metals	2-foot interval below the concrete pad
			APSB2-1		
			APSB3-1		
			APSB4-1		
			APSB5-1		
			APSB6-1		
			APSB7-1		
			APSB8-1		
			APSB9-1		
			APSB10-1		
			APSB11-1		
			APSB12-1		
	To assess subsurface conditions associated with NED combined in above sampling	Soil boring/ Grab	30SB1-1	VOCs, SVOCs, explosives, PCBs, EPH, VPH, metals	One sample from each boring from the greatest observed contamination (i.e., PID response, staining, etc.). In the absence of contamination, sample to be collected from groundwater interface or 2-foot interval above refusal.
			32SB1-1		
35 Boiler House #29	Assess contents of stack and fire boxes to characterize source	Grab	35W-1	PAHs, metals, dioxins/ furans	Sample ash and debris within stack and fire boxes. Sampling contingent upon safe access to these contents. Number of samples dependent on number of ash containing pieces of equipment remaining.
			35W-2		
			35W-3		
			35W-4		
	To generally assess surface disposal of coal ash surrounding the boiler house	ISM	35DU1-1	PAHs, metals, dioxins/ furans	35DU1 is depicted on **Figure 4C** and will consist of 30 0 to 1-foot aliquots per replicate
			35DU1-2		
			35DU1-3		
			35DU2-1		35DU2 is depicted on **Figure 4C** and will consist of 30 0 to 1-foot aliquots per replicate
			35DU2-2		
			35DU2-3		
			35DU3-1		35DU3 is depicted on **Figure 4C** and will consist of 30 0 to 1-foot aliquots per replicate
			35DU3-2		
			35DU3-3		
			35DU4-1		35DU4 is depicted on **Figure 4C** and will consist of 30 0 to 1-foot aliquots per replicate
			35DU4-2		
			35DU4-3		

NED Site No.	Data Gap Justifying Sampling	Sample Type	Sample ID	Proposed Analyses	Sample Decision Process
35 Boiler House #29 (cont.)	To vertically assess coal ash disposal around and downslope of the boiler house	Soil borings/ grab	35SB1-1 35SB1-2* 35SB2-1 35SB2-2* 35SB3-1 35SB3-2* 35SB4-1 35SB4-2* 35SB5-1 35SB5-2* 35SB6-1 35SB6-2* 35SB7-1 35SB7-2* 35SB8-1 35SB8-2* 35SB9-1 35SB9-2* 35SB10-1 35SB10-2* 35SB11-1 35SB11-2*	PAHs, metals, dioxins/ furans	Sample to be collected from the entire thickness of observed coal up to a 4-foot interval (e.g., 0 to 4 feet bgs). If greater than 4 feet of coal is encountered the -2 samples will be collected (e.g., 4 to 8, 4 to 6 feet bgs, etc.).
37 Misc. Storage Building	Assess possible release from tank of unknown use	Grab	37SB1-1 37SB2-1	EPH, VPH, VOCs, SVOCs, PCBs, metals	One sample from each boring from the greatest observed contamination (i.e., PID response, staining, etc.). In the absence of contamination, sample to be collected from groundwater interface or 2-foot interval above refusal.
38 Torpedo Storage #10	Assessment of extent of spilled transformer contents[2]	Grab	38SS-1 38CC-1* 38SS-2 38CC-2* 38SS-3 38CC-3* 38SS-4 38CC-4* 38SS-5 38CC-5* 38SS-6 38CC-6* 38SS-7 38CC-7* 38SS-8 38CC-8* 38SS-9 38CC-9*	PCBs (VPH and EPH for soil only)	A 3-meter grid will be used to collect 9 soil samples from 0 to 0.5-feet around the spilled transformer contents in accordance with 40 CFR 761.265. Concrete samples contingent upon underlying concrete, and will be collected from top 0.5 inches if present.
39 Well House #81	Assess impacts from former transformer[2]	Concrete grab	39CC-1	PCBs	Sample top 0.5 inch of concrete transformer pad. Sample location biased to area of staining.
		Grab	39SS-2 39SS-3 39SS-4 39SS-5	PCBs, EPH, VPH	4 samples of top 6 inches on each side of the transformer pad

NED Site No.	Data Gap Justifying Sampling	Sample Type	Sample ID	Proposed Analyses	Sample Decision Process
42 5,000-gallon Tank (Av Gas) Ordnance Test Facility Gasoline Outlet #30	Assess the location of former tank	Grab	42SB1-1 42SB1-2 42SB2-1 42SB2-2 42SB3-1 42SB3-2 42SB4-1 42SB4-2 42SB5-1 42SB5-2	VOCs, SVOCs, EPH, VPH, metals, PCBs	The first (-1) will be collected from 0-2 feet. The second (i.e. -2) sample from each boring from the greatest observed contamination (i.e., PID response, staining, etc.). In the absence of contamination, sample to be collected from groundwater interface or 2-foot interval above refusal.
48 & 9 Debris Stockpiles and Magazine Ignitor Storage	Assess possible landfill or stockpiling	ISM	48DU1-1 48DU1-2 48DU1-3	SVOCs, PCBs, metals, EPH, explosives	48DU1 is depicted on **Figure 4A** and will consist of 50 0 to 1-foot aliquots per replicate
		Soil boring/ Grab	48SB1-1 48SB2-1	VOCs, SVOCs, PCBs, metals, EPH, VPH, explosives	Sample from each boring from the greatest observed contamination (i.e., PID response, staining, etc.). In the absence of contamination, sample to be collected from groundwater interface or 2 foot interval above refusal.
Background	To evaluate background concentrations of arsenic for comparison to other areas of the Site in the risk assessment	Grab	BG-1 BG-2 BG-3 BG-4 BG-5 BG-6 BG-7 BG-8 BG-9 BG-10	Arsenic	Samples will be collected with hand tools from the top 1 foot

bgs - below ground surface
*Samples contingent on Site conditions. Conditions warranting collection indicated under Sample Decision Process.
^Sample depths will not be duplicated. If target intervals overlap, multiple samples will not be collected. Sample IDs will be adjusted based on required number of samples in each boring.
1 – Fingerprinting will be performed on two of the samples containing tar-like material.
2 – PCB specific (i.e., transformer) Sites have a sample design specific to requirements for assessment and delineation under TSCA.
"Metals" list is considered to include priority pollutant metals (Sb, As, Be, Cd, Cr, Cu, Hg, Pb, Ni. Se, Ag, Tl, Zn) and hexavalent chromium
All listed number of aliquots for ISM decision units are approximate and may be adjusted (±10) based on the grid overlay to ensure equal spacing throughout the DU.

GROUNDWATER

The following table summarizes the sample design and rationale for groundwater samples. Specific sample details for each sample ID listed below are provided in **Worksheet #18**. NED Sites that have no COPCs for groundwater are not listed below. Sample locations are depicted on **Figures 4A through 4C**. If proposed locations are not achievable due to field constraints, the field team will communicate these limitations according to **Worksheet #6** to evaluate changes or elimination to the design.

NED Site No.	Data Gap Justifying Sampling	Sample Type	Well ID	Proposed Analyses	Comments
4 Pump House	Assess groundwater impacts from release from drums	See NED Site No. 10, 10PZ-4			

NED Site No.	Data Gap Justifying Sampling	Sample Type	Well ID	Proposed Analyses	Comments
10 Disposal Area #14	To assess previously observed coal tar and possible free product on the beach area	Low flow grab	10PZ1	Explosives, VOCs, SVOCs, EPH, VPH, dissolved metals, pesticides	To be installed just above tidal zone, through black stained tar-like soil and across the groundwater interface
			10PZ2	Explosives, VOCs, SVOCs, EPH, VPH, dissolved metals, pesticides	
			10PZ3		
			10PZ4		
	To assess overburden groundwater impacts in the disposal area body of fill		10OBMW1		To be screened across the overburden groundwater interface and to refusal. Screen may exceed 10 feet.
			10OBMW2		
12 Maintenance Shop/ Garage/ Fire Station #39	To assess groundwater impacts associated with the maintenance pit	Low flow grab	12OBMW1	PCBs, EPH, VPH, VOCs, SVOCs, dissolved metals	To be screened across the groundwater interface
	To assess groundwater impacts in the vicinity of the suspected UST		12OBMW2		
21 Bunker #11	To assess overburden groundwater downgradient of Bunker #11	Low flow grab	21OBMW1	EPH, VPH, dissolved metals, PCBs, explosives, SVOCs, VOCs, pesticides	To be screened across the overburden groundwater interface and to refusal. Screen may exceed 10 feet.
22 Bunker #12	To assess overburden groundwater downgradient of Bunker #12	Low flow grab	22OBMW1		
27 & 31 Gas Pits	To assess the immediately downgradient location from the gas pits. Also intended to assess NED Sites 28, 30, 32, 33, 43 & 44, and includes those COPCs	Low flow grab	27OBMW1	VOCs, SVOCs, EPH, VPH, dissolved metals, PCBs, explosives	Screened at groundwater interface above high tide
			31OBMW1		
30 Pyrotechnics Storage	To assess the downgradient location of the concrete pad and NED Sites 28, 30, 32, 33, 43 & 44	Low flow grab	30OBMW1	VOCs, SVOCs, EPH, VPH, dissolved metals, PCBs, explosives	
37 Misc. Storage #28	To assess adjacent to suspected UST of unknown purpose	Low flow grab	37OBMW1	EPH, VPH, VOCs, SVOCs, PCBs, dissolved metals	Screened at the groundwater interface
42 5,000-gallon Tank (Av Gas)	To assess groundwater impacts in vicinity of former Av Gas tank	Low flow grab	42OBMW1	VOCs, SVOCs, EPH, VPH, dissolved metals, PCBs	Screened at the groundwater interface
Sitewide	To assess the bedrock aquifer in the bay ward sides of the island to intercept any possible contamination migrating into the bay at depth	Low flow grab	BRMW1	Explosives, VOCs, SVOCs, EPH, VPH, dissolved metals, PCBs	To be screened in a bedrock water bearing zone.
			BRMW2		
			BRMW3		
			BRMW4		
			BRMW5		

"Metals" list currently is considered to include priority pollutant metals (Sb, As, Be, Cd, Cr, Cu, Hg, Pb, Ni. Se, Ag, Tl, Zn) and hexavalent chromium

SEDIMENT

The following table summarizes the sample design and rationale for sediment samples. Specific sample details for each sample ID listed below are provided in **Worksheet #18**. NED Sites that have no COPCs for sediment are not listed below. Sample locations are depicted on **Figure 4B**. If proposed locations are not achievable due to field constraints, the field team will communicate these limitations according to **Worksheet #6** to evaluate changes or elimination to the design.

NED Site No.	Data Gap Justifying Sampling	Sample Type	Sample ID	Proposed Analyses	Comments
10 Disposal Area #14	Assess the extent of previously confirmed impacts to sediment	Grab	10SD1	Explosives, VOCs, SVOCs, PCBs, EPH, VPH, pH, metals, pesticides	To be collected during low tide from 0-6 inches
			10SD2		
			10SD3		
			10SD4S		S samples to be collected during low tide from 0-6 inches, D samples will be collected from the greatest observed contamination (e.g., black tar staining, PID response) within the top 4 feet
			10SD4D		
			10SD5S		
			10SD5D		
			10SD6S		
			10SD6D		
			10SD7S		
			10SD7D		
			10SD8		To be collected during low tide from 0-6 inches
			10SD9		To be collected with ponar sampler from boat
			10SD10		
			10SD11		
			10SD12		
			10SD13		
			10SD14		
			10SD15		

"Metals" list currently is considered to include priority pollutant metals (Sb, As, Be, Cd, Cr, Cu, Hg, Pb, Ni. Se, Ag, Tl, Zn) and hexavalent chromium

WORKSHEET #18 – SAMPLING LOCATIONS AND METHODS

SOIL/SOLIDS

The following table summarizes samples to be collected for soil.

Sample ID	Matrix	Depth Key*	Method	Analytical Method	Sampling SOP	Comments
01DU1-1	S	0-1'/30	ISM	PAHs (8270D), PP metals (6020A/7471B), Cr^{+6} (7196A), EPH (MADEP EPH-04-1.1),	CA-26	-
01DU1-2	S	0-1'/30	ISM			-
01DU1-3	S	0-1'/30	ISM			
01DU2-1	S	0-1'/30	ISM			-
01DU2-2	S	0-1'/30	ISM			-
01DU2-3	S	0-1'/30	ISM			
01DU3-1	S	0-1'/30	ISM			-
01DU3-2	S	0-1'/30	ISM			-
01DU3-3	S	0-1'/30	ISM			-
01SB1-1	S	B	Grab		CA-5	FD: 01SB1-10
01SB1-2*	S	B	Grab			Contingent on coal thickness
01SB2-1	S	B	Grab			MS/MSD
01SB2-2*	S	B	Grab			Contingent on coal thickness
01SB3-1	S	B	Grab			-
01SB3-2*	S	B	Grab			Contingent on coal thickness
01SB4-1	S	B	Grab			-
01SB4-2*	S	B	Grab			Contingent on coal thickness
01SB5-1	S	B	Grab			-
01SB5-2*	S	B	Grab			Contingent on coal thickness
01SB6-1	S	B	Grab			-
01SB6-2*	S	B	Grab			Contingent on coal thickness
01SB7-1	S	B	Grab			-
01SB7-2*	S	B	Grab			Contingent on coal thickness
01SB8-1	S	B	Grab			-
01SB8-2*	S	B	Grab			Contingent on coal thickness
01SB9-1	S	B	Grab			-
01SB9-2*	S	B	Grab			Contingent on coal thickness
01SB10-1	S	B	Grab			-
01SB10-2*	S	B	Grab			Contingent on coal thickness

Sample ID	Matrix	Depth Key*	Method	Analytical Method	Sampling SOP	Comments
04CP-1	S	0-0.5'/16	Composite	VOCs (8260C), SVOCs (8270D), EPH (MADEP EPH-04-1.1), VPH (MADEP VPH-04-1.1), PCBs (8082A), PP metals (6020A/7471B), Cr^{+6} (7196A)	CA-5	-
08W-1*	D	NA	Grab	PAHs (8270D), dioxins/furans (1613B), PP metals (6020A/7471B), Cr^{+6} (7196A), PCBs (8082A)	CA-5	FD: 08W-10
08W-2*	D	NA	Grab			-
08W-3*	D	NA	Grab			-
08W-4*	D	NA	Grab			-
08DU1-1	S	0-1'/30	ISM		CA-26	MS/MSD
08DU1-2	S	0-1'/30	ISM			-
08DU1-3		0-1'/30	ISM			-
08DU2-1	S	0-1'/30	ISM			-
08DU2-2	S	0-1'/30	ISM			-
08DU2-3		0-1'/30	ISM			-
08DU3-1	S	0-1'/30	ISM			-
08DU3-2	S	0-1'/30	ISM			-
08DU3-3		0-1'/30	ISM			-
08DU4-1	S	0-1'/30	ISM			-
08DU4-2	S	0-1'/30	ISM			-
08DU4-3		0-1'/30	ISM			-
08SB1-1	S	C	Grab		CA-5	FD: 08SB1-10
08SB2-1	S	C	Grab			-
08SB3-1	S	C	Grab			-
08SB4-1	S	C	Grab			-
08SB5-1	S	C	Grab			MS/MSD
08SB6-1	S	C	Grab			-
08SB7-1	S	C	Grab			-

Sample ID	Matrix	Depth Key*	Method	Analytical Method	Sampling SOP	Comments
10SB1-1	S	0-2’	Grab	VOC (8260C), SVOCs (8270D, EPH (MADEP EPH-04-1.1), VPH (MADEP VPH-04-1.1), PP metals (6020A/7471B), Cr^{+6} (7196A), explosives (8330B), PCBs (8082A), pH (9045C), pesticides (8081A)	CA-5	-
10SB1-2^	S	2-4’	Grab			-
10SB1-3 ^	S	4-6’	Grab			-
10SB1-4^	S	D	Grab			-
10SB1-5^	S	E	Grab			-
10SB2-1	S	0-2’	Grab			-
10SB2-2^	S	2-4’	Grab			FD: 10SB2-20
10SB2-3 ^	S	4-6’	Grab			-
10SB2-4^	S	D	Grab			-
10SB2-5^	S	E	Grab			-
10SB3-1	S	0-2’	Grab			-
10SB3-2^	S	2-4’	Grab			-
10SB3-3 ^	S	4-6’	Grab			-
10SB3-4^	S	D	Grab			-
10SB3-5^	S	E	Grab			-
10SB4-1	S	0-2’	Grab			-
10SB4-2^	S	2-4’	Grab			-
10SB4-3 ^	S	4-6’	Grab			-
10SB4-4^	S	D	Grab			-
10SB4-5^	S	E	Grab			-
10SB5-1	S	0-2’	Grab			MS/MSD
10SB5-2^	S	2-4’	Grab			-
10SB5-3 ^	S	4-6’	Grab			-
10SB5-4^	S	D	Grab			-
10SB5-5^	S	E	Grab			-
10TP1-1	S	F	Grab	VOC (8260C), SVOCs (8270D, EPH (MADEP EPH-04-1.1), VPH (MADEP VPH-04-1.1), PP metals (6020A /7471B), Cr^{+6} (7196A), explosives (8330B), PCBs (8082A), pH (9045C) , pesticides (8081A)	CA-5	MS/MSD
10TP2-1	S	F	Grab			-
10TP3-1	S	F	Grab			-
10TP4-1	S	F	Grab			FD: 10TP4-10
10TP5-1	S	F	Grab			-
10TP5-2	S	D	Grab			-
10TP6-1	S	F	Grab			-
10TP7-1	S	F	Grab			-
10TP8-1	S	F	Grab			-
10TP8-2	S	D	Grab			-
10TP9-1	S	F	Grab			-
10TP10-1	S	F	Grab			-
10TP11-1	S	F	Grab			-
10TP12-1	S	F	Grab			-
10TP13-1	S	F	Grab			-
11DU1-1	S	0-1’/50	ISM	SVOCs (8270D), EPH (MADEP EPH-04-1.1), Cr^{+6} (7196A), PP metals (6020A/7471B)	CA-26	-
11DU1-2	S	0-1’/50	ISM			-
11DU1-3	S	0-1’/50	ISM			-

Sample ID	Matrix	Depth Key*	Method	Analytical Method	Sampling SOP	Comments
11SS-1	S	0-1’	Grab	VOCs (8260C), SVOCs (8270D), EPH (MADEP EPH-04-1.1), VPH (MADEP VPH-04-1.1), Cr^{+6} (7196A), PP metals (6020A/7471B)	CA-5	-
11SS-2	S	0-1’	Grab			-
11SS-3	S	0-1’	Grab			-
12SS-1	S	0-0.5 ft	Grab	PCBs (8082A), and EPH (MADEP EPH-04-1.1) and VPH (MADEP VPH-04-1.1) for soil only	CA-5	FD: 12SS-10
12CC-1	C	0-0.5 in	Grab		CA-23	-
12SS-2	S	0-0.5 ft	Grab		CA-5	-
12CC-2	C	0-0.5 in	Grab		CA-23	-
12SS-3	S	0-0.5 ft	Grab		CA-5	-
12CC-3	C	0-0.5 in	Grab		CA-23	-
12SS-4	S	0-0.5 ft	Grab		CA-5	-
12CC-4	C	0-0.5 in	Grab		CA-23	-
12SS-5	S	0-0.5 ft	Grab		CA-5	-
12CC-5	C	0-0.5 in	Grab		CA-23	-
12SS-6	S	0-0.5 ft	Grab		CA-5	-
12CC-6	C	0-0.5 in	Grab		CA-23	-
12SS-7	S	0-0.5 ft	Grab		CA-5	-
12CC-7	C	0-0.5 in	Grab		CA-23	-
12SS-8	S	0-0.5 ft	Grab		CA-5	-
12CC-8	C	0-0.5 in	Grab		CA-23	-
12SS-9	S	0-0.5 ft	Grab		CA-5	MS/MSD
12CC-9	C	0-0.5 in	Grab		CA-23	-
12SB1-1	S	A	Grab	PCBs (8082A), EPH (MADEP EPH-04-1.1), VPH (MADEP VPH-04-1.1), VOCs (8260C), SVOCs (8270D), Cr^{+6} (7196A), PP metals (6020A/7471B)	CA-5	-
12W-1	SL/D	0-0.5 ft	Grab			-
12W-2	SL/D	0-0.5 ft	Grab			-
12W-3	SL/D	0-0.5 ft	Grab			-
12SB2-1	S	A	Grab			-
12SB3-1	S	A	Grab			-
12SB4-1	S	A	Grab			-
13CC-1	C	0-0.5 in	Grab	PCBs (8082A)	CA-23	-
13CC-2	C	0-0.5 in	Grab			-
13CC-3	C	0-0.5 in	Grab			-
13CC-4	C	0-0.5 in	Grab			-
13SS-3	S	0-6 in	Grab	PCBs (8082A), EPH (MADEP EPH-04-1.1), Cr^{+6} (7196A), PP metals (6020A/7471B)	CA-5	-
13SS-4	S	0-6 in	Grab			-
13SS-5	S	0-6 in	Grab			-
13SS-6	S	0-6 in	Grab			-

Sample ID	Matrix	Depth Key*	Method	Analytical Method	Sampling SOP	Comments
16T-1	S	0-6 in	Grab	PCBs (8082A), EPH (MADEP EPH-04-1.1), VPH (MADEP VPH-04-1.1)	CA-5	FD: 16T-10
16T-2	S	0-6 in	Grab			Contingent on number of transformer found
16T-3	S	0-6 in	Grab			
16SS-1	S	0-6 in	Grab			Contingent on release
16SS-2	S	0-6 in	Grab			Contingent on release
16SS-3	S	0-6 in	Grab			Contingent on release
16SS-4	S	0-6 in	Grab			Contingent on release
16SS-5	S	0-6 in	Grab			Contingent on release
16SS-6	S	0-6 in	Grab			Contingent on release
16SS-7	S	0-6 in	Grab			Contingent on release
16SS-8	S	0-6 in	Grab			Contingent on release
16SS-9	S	0-6 in	Grab			Contingent on release
16SS-10	S	0-6 in	Grab			Contingent on release
16SS-11	S	0-6 in	Grab			Contingent on release
16SS-12	S	0-6 in	Grab			MS/MSD, Contingent on release
16SS-13	S	0-6 in	Grab			Contingent on release
16SS-14	S	0-6 in	Grab			Contingent on release
16SS-15	S	0-6 in	Grab			Contingent on release
16SS-16	S	0-6 in	Grab			Contingent on release
19DU1-1	S	0-1’/30	ISM	EPH (MADEP EPH-04-1.1),	CA-26	FD: 19DU1-10
19DU1-2	S	0-1’/30	ISM			MS/MSD
19DU1-3	S	0-1’/30	ISM			-
21SS-3	S/D	0-2 ft or floor debris	Grab	PCBs (8082A), EPH (MADEP EPH-04-1.1), VPH (MADEP VPH-04-1.1), VOCs (8260C), SVOCs (8270D), Cr^{+6} (7196A), PP metals (6020A/7471B), pesticides (8081B), explosives (8330B)	CA-5	-
21SS-4	S/D		Grab			-
21SS-5	S/D		Grab			-
21SS-6	S/D		Grab			-
21SS-7	S/D		Grab			-
21SB1-1	S	G	Grab			-
21SB2-1	S	G	Grab			-
21SB3-1	S	G	Grab			-
21SB4-1	S	G	Grab			-
21SB-5-1	S	G	Grab			MS/MSD
21SB-6-1	S	G	Grab			-
22SS-3	S/D	0-2 ft or floor debris	Grab			FD: 22SS-30
22SS-4	S/D		Grab			-
22SS-5	S/D		Grab			-
22SB1-1	S	G	Grab			-
22SB2-1	S	G	Grab			-
22SB3-1	S	G	Grab			-
22SB4-1	S	G	Grab			-
22SB-5-1	S	G	Grab			-
22SB-6-1	S	G	Grab			MS/MSD
23DU1-1	S	0-1’/50	ISM	PAHs (8270D), PP metals (6020A/7471B), Cr^{+6} (7196A), EPH (MADEP EPH-04-1.1)	CA-26	-
23DU1-2	S	0-1’/50	ISM			-
23DU1-3	S	0-1’/50	ISM			-
23DU2-1	S	0-1’/50	ISM			MS/MSD
23DU2-2	S	0-1’/50	ISM			-
23DU2-3	S	0-1’/50	ISM			-

Sample ID	Matrix	Depth Key*	Method	Analytical Method	Sampling SOP	Comments
23SB1-1	S	B	Grab	PAHs (8270D), PP metals (6020A/7471B), Cr^{+6} (7196A), EPH (MADEP EPH-04-1.1)	CA-5	-
23SB1-2	S	B	Grab			Contingent on coal thickness
23SB2-1	S	B	Grab			FD: 23SB2-10
23SB2-2	S	B	Grab			Contingent on coal thickness
23SB3-1	S	B	Grab			-
23SB3-2	S	B	Grab			Contingent on coal thickness
23SB4-1	S	B	Grab			FD: 23SB4-10
23SB4-2	S	B	Grab			Contingent on coal thickness
23SB5-1	S	B	Grab			-
23SB5-2	S	B	Grab			Contingent on coal thickness
23SB6-1	S	B	Grab			-
23SB6-2	S	B	Grab			Contingent on coal thickness
23SB7-1	S	B	Grab			-
23SB7-2	S	B	Grab			Contingent on coal thickness
23SB8-1	S	B	Grab			-
23SB8-2	S	B	Grab			Contingent on coal thickness
24CC-1	C	0-0.5 in	Grab	PCBs (8082A)	CA-23	FD: 24CC-10
24SS-2	S	0-0.5 ft	Grab	PCBs (8082A), EPH (MADEP EPH-04-1.1), VPH (MADEP VPH-04-1.1)		-
24SS-3	S	0-0.5 ft	Grab			-
24SS-4	S	0-0.5 ft	Grab			-
24SS-5	S	0-0.5 ft	Grab			-
27SL-1	SL	0-0.5 ft	Grab	VOCs (8260C), SVOCs (8270D), EPH (MADEP EPH-04-1.1), VPH (MADEP VPH-04-1.1), PP metals (6020A /7471B), Cr^{+6} (7196A), PCBs (8082A)	CA-5	FD: 27SL-10
27SL-2	SL	0-0.5 ft	Grab			-
31SL-1	SL	0-0.5 ft	Grab			-
27SB1-1	S	G	Grab			-
27SB2-1	S	G	Grab			MS/MSD
31SB1-1	S	G	Grab			-

Sample ID	Matrix	Depth Key*	Method	Analytical Method	Sampling SOP	Comments
APSB1-1	S	H	Grab	VOCs (8260C), PCBs (8082A), EPH (MADEP EPH-04-1.1), VPH, MADEP VPH-04-1.1), SVOCs (8270D), PP metals (6020A/7471B), Cr^{+6} (7196A), explosives (8330B)	CA-5	-
APSB2-1	S	H	Grab			-
APSB3-1	S	H	Grab			-
APSB4-1	S	H	Grab			-
APSB5-1	S	H	Grab			-
APSB6-1	S	H	Grab			-
APSB7-1	S	H	Grab			-
APSB8-1	S	H	Grab			-
APSB9-1	S	H	Grab			-
APSB10-1	S	H	Grab			-
APSB11-1	S	H	Grab			-
APSB12-1	S	H	Grab			-
30SB1-1	S	G	Grab	PCBs (8082A), EPH (MADEP EPH-04-1.1), VPH (MADEP VPH-04-1.1), VOCs (8260C), SVOCs (8270D), PP metals (6020A/7471B), Cr^{+6} (7196A), explosives (8330B)	CA-5	-
32SB1-1	S	G	Grab		CA-5	-

Sample ID	Matrix	Depth Key*	Method	Analytical Method	Sampling SOP	Comments
35W-1	S/D	NA	Grab	PAHs (8270D), dioxin/furan (1613B), Cr^{+6} (7196A), PP metals (6020A/7471B)	CA-5	-
35W-2	S/D	NA	Grab			-
35W-3	S/D	NA	Grab			-
35W-4	S/D	NA	Grab			-
35DU1-1	S	0-1'/30	ISM		CA-26	FD: 35DU1-10
35DU1-2	S	0-1'/30	ISM			-
35DU1-3	S	0-1'/30	ISM			
35DU2-1	S	0-1'/30	ISM			-
35DU2-2	S	0-1'/30	ISM			-
35DU2-3	S	0-1'/30	ISM			
35DU3-1	S	0-1'/30	ISM			-
35DU3-2	S	0-1'/30	ISM			-
35DU3-3	S	0-1'/30	ISM			
35DU4-1	S	0-1'/30	ISM			MS/MSD
35DU4-2	S	0-1'/30	ISM			-
35DU4-3	S	0-1'/30	ISM			-
35SB1-1	S	B	Grab		CA-5	FD: 35SB1-10
35SB1-2	S	B	Grab			Contingent on coal/ash thickness
35SB2-1	S	B	Grab			-
35SB2-2	S	B	Grab			Contingent on coal/ash thickness
35SB3-1	S	B	Grab			-
35SB3-2	S	B	Grab			Contingent on coal/ash thickness
35SB4-1	S	B	Grab			-
35SB4-2	S	B	Grab			Contingent on coal/ash thickness
35SB5-1	S	B	Grab			-
35SB5-2	S	B	Grab			Contingent on coal/ash thickness
35SB6-1	S	B	Grab			-
35SB6-2	S	B	Grab			Contingent on coal/ash thickness
35SB7-1	S	B	Grab			MS/MSD
35SB7-2	S	B	Grab			Contingent on coal/ash thickness
35SB8-1	S	B	Grab			-
35SB8-2	S	B	Grab			Contingent on coal/ash thickness
35SB9-1	S	B	Grab			-
35SB9-2	S	B	Grab			Contingent on coal/ash thickness
35SB10-1	S	B	Grab			-
35SB10-2	S	B	Grab			Contingent on coal/ash thickness
35SB11-1	S	B	Grab			-
35SB11-2	S	B	Grab			Contingent on coal/ash thickness

Sample ID	Matrix	Depth Key*	Method	Analytical Method	Sampling SOP	Comments
37SB1-1	S	G	Grab	PCBs (8082A), EPH (MADEP EPH-04-1.1), VPH (MADEP VPH-04-1.1), VOCs (8260C), SVOCs (8270D), Cr^{+6} (7196A), PP metals (6020A/7471B)	CA-5	-
37SB2-1	S	G	Grab			-
38SS-1	S	0-0.5 ft	Grab	PCBs (8082A), and EPH (MADEP EPH-04-1.1) and VPH (MADEP VPH-04-1.1) for soil only	CA-5	FD: 38SS-10
38CC-1	C	0-0.5 in	Grab		CA-23	Contingent on presence of concrete
38SS-2	S	0-0.5 ft	Grab		CA-5	-
38CC-2	C	0-0.5 in	Grab		CA-23	Contingent on presence of concrete
38SS-3	S	0-0.5 ft	Grab		CA-5	-
38CC-3	C	0-0.5 in	Grab		CA-23	Contingent on presence of concrete
38SS-4	S	0-0.5 ft	Grab		CA-5	-
38CC-4	C	0-0.5 in	Grab		CA-23	Contingent on presence of concrete
38SS-5	S	0-0.5 ft	Grab		CA-5	-
38CC-5	C	0-0.5 in	Grab		CA-23	Contingent on presence of concrete
38SS-6	S	0-0.5 ft	Grab		CA-5	-
38CC-6	C	0-0.5 in	Grab		CA-23	Contingent on presence of concrete
38SS-7	S	0-0.5 ft	Grab		CA-5	-
38CC-7	C	0-0.5 in	Grab		CA-23	Contingent on presence of concrete
38SS-8	S	0-0.5 ft	Grab		CA-5	MS/MSD
38CC-8	C	0-0.5 in	Grab		CA-23	Contingent on presence of concrete
38SS-9	S	0-0.5 ft	Grab		CA-5	-
38CC-9	C	0-0.5 in	Grab		CA-23	Contingent on presence of concrete
39CC-1	C	0-0.5 in	Grab	PCBs (8082A)	CA-23	-
39SS-2	S	0-0.5 ft	Grab	PCBs (8082A), EPH (MADEP EPH-04-1.1) and VPH (MADEP VPH-04-1.1)	CA-5	-
39SS-3	S	0-0.5 ft	Grab			-
39SS-4	S	0-0.5 ft	Grab			-
39SS-5	S	0-0.5 ft	Grab			-

Sample ID	Matrix	Depth Key*	Method	Analytical Method	Sampling SOP	Comments
42SB1-1	S	0-2 ft	Grab	VOCs (8260C), SVOCs (8270D), EPH (MADEP EPH-04-1.1), VPH (MADEP VPH-04-1.1), Cr^{+6} (7196A), PP metals (6020A/7471B), PCBs (8082A)	CA-5	-
42SB1-2	S	G	Grab			-
42SB2-1	S	0-2 ft	Grab			-
42SB2-2	S	G	Grab			-
42SB3-1	S	0-2 ft	Grab			-
42SB3-2	S	G	Grab			-
42SB4-1	S	0-2 ft	Grab			-
42SB4-2	S	G	Grab			-
42SB5-1	S	0-2 ft	Grab			-
42SB5-2	S	G	Grab			-
48DU1-1	S	0-1'/50	ISM	SVOCs (8270D), EPH (MADEP EPH-04-1.1), Cr^{+6} (7196A), PP metals (6020A/7471B), PCBs (8082A), explosives (8330B)	CA-26	-
48DU1-2	S	0-1'/50	ISM			-
48DU1-3	S	0-1'/50	ISM			-
48SB1-1	S	G	Grab	VOCs (8260C), SVOCs (8270D), EPH (MADEP EPH-04-1.1), VPH (MADEP VPH-04-1.1), Cr^{+6} (7196A), PP metals (6020A/7471B), PCBs (8082A), explosives (8330B)	CA-5	-
48SB2-1	S	G	Grab			-
BG1	S	0-1	Grab	Arsenic (6020A)	CA-5	-
BG2	S	0-1	Grab			-
BG3	S	0-1	Grab			-
BG4	S	0-1	Grab			-
BG5	S	0-1	Grab			-
BG6	S	0-1	Grab			-
BG7	S	0-1	Grab			-
BG8	S	0-1	Grab			-
BG9	S	0-1	Grab			-
BG10	S	0-1	Grab			-

S-soil SL – sludge D – debris/ash C - concrete

***Depth Key:**

0-1/50 – 50 aliquots from 0 to 1 foot from respective decision unit, similarly 0-1/20, 0-1/30, etc.

A – Sample to be collected from depth of greatest observation contamination (i.e., PID response, staining, etc.) or the groundwater interface in the absence of contamination.

B – Sample to be collected from the entire thickness of observed coal up to 4-foot intervals.

C – Sample to be collected from observable contamination or 0-1 foot

D – Sample to be collected from greatest observed contamination. If no evidence of contamination, no sample to be collected.

E – Sample to be collected from 2-foot interval above refusal

F – Sample to be collected from greatest observed contamination. If no evidence of contamination, sample to be collected from 0 to 2 feet bgs

G – Sample to be collected from greatest observed contamination. If no evidence of contamination, sample to be collected from groundwater interface or 2 feet above refusal, whichever is shallower.

H – Sample to be collected from 2-foot interval below concrete.

^Sample depths will not be duplicated. If target intervals overlap, multiple samples will not be collected. Sample IDs will be adjusted based on required number of samples in each boring.

GROUNDWATER

The following table summarizes samples to be collected for groundwater.

Sample ID	Matrix	Depth (ft. bgs)	Method	Analytical Method	Sampling SOP	Comments
10PZ-1-mmyy	GW	Screen interval	Low flow/ peristaltic pump	Explosives (8330B), VOCs (8260C), SVOCs (8270D), PAHs (8270D SIM), EPH (MADEP EPH-04-1.1), VPH (MADEP VPH-04-1.1), Cr^{+6} (7196A), PP metals (6020A/7470A), pesticides (8081A)	CA-12	-
10PZ-2-mmyy	GW	Screen interval	Low flow/ peristaltic pump	Explosives (8330B), VOCs (8260C), SVOCs (8270D), PAHs (8270D SIM), EPH (MADEP EPH-04-1.1), VPH (MADEP VPH-04-1.1), Cr^{+6} (7196A), PP metals (6020A/7470A) , pesticides (8081A)	CA-12	FD:10PZ-20-mmyy
10PZ-3-mmyy	GW	Screen interval				-
10PZ-4-mmyy	GW	Screen interval				-
10OBMW1-mmyy	GW	Screen interval				-
10OBMW2-mmyy	GW	Screen interval				-
12OBMW1-mmyy	GW	Screen interval	Low flow/ peristaltic pump	VOCs (8260C), SVOCs (8270D), PAHs (8270D SIM), EPH (MADEP EPH-04-1.1), VPH (MADEP VPH-04-1.1), PCBs (8082A), Cr^{+6} (7196A), PP metals (6020A/7470A)	CA-12	-
12OBMW2-mmyy	GW	Screen interval				-

Sample ID	Matrix	Depth (ft. bgs)	Method	Analytical Method	Sampling SOP	Comments
21OBMW1-mmyy	GW	Screen interval	Low flow/ peristaltic pump	Explosives (8330B), VOCs (8260C), SVOCs (8270D), PAHs (8270D SIM), EPH (MADEP EPH-04-1.1), VPH (MADEP VPH-04-1.1), PCBs (8082A), Cr^{+6} (7196A), PP metals (6020A/7470A), pesticides (8081B)	CA-12	-
22OBMW1-mmyy	GW	Screen interval				-
27OBMW1-mmyy	GW	Screen interval	Low flow/ peristaltic pump	Explosives (8330B), VOCs (8260C), SVOCs (8270D), PAHs (8270D SIM), EPH (MADEP EPH-04-1.1), VPH (MADEP VPH-04-1.1), PCBs (8082A), Cr^{+6} (7196A), PP metals (6020A/7470A)	CA-12	MS/MSD
30OBMW1-mmyy	GW	Screen interval				-
31OBMW1-mmyy	GW	Screen interval				-
37OBMW1-mmyy	GW	Screen interval	Low flow/ peristaltic pump		CA-12	-
42OBMW1-mmyy	GW	Screen interval	Low flow/ peristaltic pump	VOCs (8260C), SVOCs (8270D), PAHs (8270D SIM), EPH (MADEP EPH-04-1.1), VPH (MADEP VPH-04-1.1), Cr^{+6} (7196A), PP metals (6020A/7470A), PCBs (8080A)	CA-12	-
BRMW-1-mmyy	GW	10 feet from bottom of well or within water column (if less than 10')	Low flow/ submersible pump	Explosives (8330B), VOCs (8260C), SVOCs (8270D), PAHs (8270D SIM), EPH (MADEP EPH-04-1.1), VPH (MADEP VPH-04-1.1), Cr^{+6} (7196A), PP metals (6020A/7470A), PCBs (8080A)	CA-12	FD: BRMW-10-mmyy
BRMW-2-mmyy	GW					-
BRMW-3-mmyy	GW					-
BRMW-4-mmyy	GW					MS/MSD
BRMW-5-mmyy	GW					-

GW-groundwater

SEDIMENT

The following table summarizes samples to be collected for sediment.

Sample ID	Matrix	Depth (ft. bgs)	Method	Analytical Method	Sampling SOP	Comments
10SD1	SD	0-0.5	Hand auger or handheld probe	Explosives (with nitroglycerin) (8330B), VOCs (8260C), SVOCs (8270D), PAHs (8270D SIM), Cr^{+6} (7196A), PP metals (6020A/7471B), pH (9045C), EPH (MADEP EPH-04-1.1), VPH (MADEP VPH-04-1.1) , pesticides (8081A), PCBs (8082A)	CA-13	-
10SD2	SD	0-0.5				MS/MSD
10SD3	SD	0-0.5				-
10SD4S	SD	0-0.5				-
10SD4D	SD	0-2				Depth interval to be adjusted based on observed evidence of contamination
10SD5S	SD	0-0.5				-
10SD5D	SD	0-2				Depth interval to be adjusted based on observed evidence of contamination
10SD6S	SD	0-0.5				-
10SD6D	SD	0-2				Depth interval to be adjusted based on observed evidence of contamination
10SD7S	SD	0-0.5				-
10SD7D	SD	0-2				FD: 10SD70D, Depth interval to be adjusted based on observed evidence of contamination
10SD8	SD	0-0.5				-
10SD9	SD	0-0.5	Ponar sampler	Explosives (8330B), VOCs (8260C), SVOCs (8270D), PAHs (8270D SIM), Cr^{+6} (7196A), PP metals (6020A/7471B), pH (9045C), EPH (MADEP EPH-04-1.1), VPH (MADEP VPH-04-1.1) , pesticides (8081A), PCBs (8082A)	CA-13	-
10SD10	SD	0-0.5				-
10SD11	SD	0-0.5				-
10SD12	SD	0-0.5				-
10SD13	SD	0-0.5				-
10SD14	SD	0-0.5				-
10SD15	SD	0-0.5				-

SD - sediment

WORKSHEET #19 & 30 – SAMPLE CONTAINERS, PRESERVATION, AND HOLD TIMES

SOIL & SEDIMENT

Laboratory Name: Absolute Resource Associates, Portsmouth New Hampshire or their subcontractor lab, Test America in Sacramento, California (indicated with an *)
Accreditation/Certification: DoD ELAP
Backup Laboratory: Test America, Sacramento, California
Matrix: Soil and sediment

Analysis	Method/SOP	Accreditation Expiration Date	Sample Container	Preservative	Holding Time	Data Package Turnaround
VOCs	EPA Method 8260C/QA-5120	7/31/2019	One 40 mL VOA vial	Methanol, cooled to less than 4°C	14 days	20 day
PAHs SIM	EPA Method 3550B and 8270D with SIM/WS-OP-0001, WS-MS-0008*	1/20/2018	One 8 oz. amber jar	Cooled to less than 4°C	14 days extraction, 40 days analysis	20 day
SVOCs (and PAHs)	EPA Method 8270D/QA-5515	7/31/2019	One 4 oz. amber jar	Cooled to less than 4°C	14 days extraction, 40 days analysis	20 day
Metals	EPA Method 6020A/ QA-5605	7/31/2019	One 4 oz. soil jar	Cooled to less than 4°C	180 days	20 day
Mercury	EPA Method 7471B/QA-5600	7/31/2019	One 4 oz. soil jar	Cooled to less than 4°C	28 days	20 day
Hexavalent Chromium	EPA Method 7196A/QA-5813	None	One 4 oz. soil jar	Cooled to less than 4°C	30 days digestion, 7 days after till analysis	20 day
EPH	MADEP EPH-04-1.1/QA-5313	7/31/2019	One 4 oz. amber jar	Cooled to less than 4°C	14 days extraction, 40 days analysis	20 day
VPH	MADEP VPH-04-1.1/QA-5130	7/31/2019	One 40 mL VOA vial	Methanol, cooled to less than 4°C	28 days	20 day
PCBs	EPA Method 8082A/QA-5303	7/31/2019	One 4 oz. soil jar	Cooled to less than 4°C	1 year to extraction, 40 day analysis	20 day
Explosives	EPA Method 8330B/WS-LC-0009*	1/20/2018	One 4 oz amber jar	Cooled to less than 4°C	14 days extraction, 40 days analysis	20 day
Dioxins/ furans	EPA Method 1613B/WS-ID-0007*	1/20/2018	One 4 oz amber jar	Cooled to less than 4°C	1 year	20 day

Analysis	Method/SOP	Accreditation Expiration Date	Sample Container	Preservative	Holding Time	Data Package Turnaround
Pesticides	EPA Method 8081B/QA-5304	7/31/2019	One 4 oz. soil jar	Cooled to less than 4°C	14 days extraction, 40 days analysis	20 day
pH	EPA Method 9045C/QA-5851	None	One 4 oz. soil jar	Cooled to less than 4°C	7 days	20 day

VOA – volatile organic analysis oz – ounce °C – degree Celsius mL – milliliter

GROUNDWATER

Laboratory Name: Absolute Resource Associates, Portsmouth New Hampshire or their subcontractor lab, Test America in Sacramento, California (indicated with an *)
Accreditation/Certification: DoD ELAP
Backup Laboratory: Test America, Sacramento, California
Matrix: Groundwater

Analysis	Method/SOP	Accreditation Expiration Date	Sample Container	Preservative	Holding Time	Data Package Turnaround
VOCs	EPA Method 8260C/QA-5120	7/31/2019	Two 40 mL VOA vial	HCl, cooled to less than 4°C	14 days	20 day
PAHs	EPA Method 3510C and 8270D with SIM/ WS-OP-0001, WS-MW-0008*	1/20/2018	One 1 liter amber	Cooled to less than 4°C	7 days to extraction, 40 days to analysis	20 day
SVOCs	EPA Method 8270D/QA-5515	7/31/2019	One 1 liter amber	Cooled to less than 4°C	7 days to extraction, 40 days to analysis	20 day
Metals	EPA Method 6020A/ QA-5605	7/31/2019	One 250 mL HDPE	HNO_3, cooled to less than 4°C	180 days	20 day
Mercury	EPA Method 7471B/QA-5600	7/31/2019	One 250 mL HDPE	HNO_3, cooled to less than 4°C	28 days	20 day
Hexavalent Chromium	SM 3500 Cr-B/QA-5813	None	One 250 mL HDPE	$(NH_4)_2SO_4$, cooled to less than 4°C	28 days	20 day
EPH	MADEP EPH-04-1.1/QA-5313	7/31/2019	One 1 liter amber	HCl, cooled to less than 4°C	14 days extraction, 40 days analysis	20 day
VPH	MADEP VPH-04-1.1/QA-5130	7/31/2019	Two 40 mL VOA vial	HCl, cooled to less than 4°C	14 days	20 day

Analysis	Method/SOP	Accreditation Expiration Date	Sample Container	Preservative	Holding Time	Data Package Turnaround
PCBs	EPA Method 8082A/QA-5303	7/31/2019	One 1 liter amber	Cooled to less than 4°C	1 year extraction, 40 days analysis	20 day
Explosives	EPA Method 8330B/WS-LC-0009*	1/20/2018	Two 1 liter amber	Cooled to less than 4°C	7 days extraction, 40 days analysis	20 day
Pesticides	EPA Method 8081B/QA-5304	7/31/2019	One 1 liter amber	Cooled to less than 4°C	7 days extraction, 40 days analysis	20 days

HCl – hydrochloric acid HNO_3 – nitric acid $(NH_4)_2SO_4$ – ammonia sulfate
HDPE – high density polyethylene

WORKSHEET #20 – FIELD QUALITY CONTROL SUMMARY

Matrix	Analyte/ Analyte Group	# Samples	# FD	# MS/MSD	# FB	#EB**	#TB***	Total # Analyses
Soil/sediment	VOCs	124	7	7/7	0	10	8	163
Soil/sediment	SVOCs	130	7	7/7	0	10	0	161
Soil/sediment	PAHs	112	6	6/6	0	10	0	140
Soil/sediment	PAHs SIM	19	1	1/1	0	1	0	23
Soil/sediment	Metals	246	13	13/13	0	10	0	295
Soil/sediment	Mercury	246	13	13/13	0	10	0	295
Soil/sediment	Chromium^{+6}	246	13	13/13	0	10	0	295
Soil/sediment	EPH	233	12	12/12	0	10	0	279
Soil/sediment	VPH	173	9	9/9	0	10	8	218
Soil/sediment	PCBs	224	12	12/12	0	10	0	270
Soil/sediment	Explosives	97	5	5/5	0	10	0	122
Soil/sediment	Dioxin/Furan	61	3	3/3	0	10	0	80
Soil/sediment	Pesticides	79	4	4/4	0	10	0	101
Soil/sediment	pH	58	3	3/3	0	10	0	77
Groundwater	VOCs	20	2	2/2	0	3	6	35
Groundwater	SVOCs	20	2	2/2	0	3	0	29
Groundwater	PAHs SIM	20	2	2/2	0	0	0	29
Groundwater	Metals	20	2	2/2	0	3	0	29
Groundwater	Mercury	20	2	2/2	0	3	0	29
Groundwater	Chromium^{+6}	20	2	2/2	0	3	0	29
Groundwater	EPH	20	2	2/2	0	3	0	29
Groundwater	VPH	20	2	2/2	0	3	6	35
Groundwater	PCBs	14	1	1/1	0	3	0	20
Groundwater	Explosives	16	1	1/1	0	3	0	22
Groundwater	Pesticides	8	1	1/1	0	3	0	14

FD – field duplicate FB – field blank EB – equipment blanks TB – trip blanks
** Equipment blanks will be submitted at a rate of 1 per day when non-disposal equipment is used.
***Trip blanks are submitted at a rate of one per cooler. Indicated quantity is estimated.

WORKSHEET #21 – FIELD SOPS

Field activities will be completed in accordance with Credere's SOPs for the respective activity. Credere's SOPs consider both EPA SOPs and RIDEM rules and are at least as comprehensive as the procedures described therein. Credere field SOPs to be used as part of field activities described herein are summarized below and are included in **Appendix E**.

SOP #	Title	Date/Revision	Modified for Project
CA-1	Field Activity Documentation	August 2, 2016, revision 1	No
CA-2	Equipment Decontamination Procedures	March 17, 2016, revision 0	No
CA-4	Soil Description	March 17, 2016, revision 0	No
CA-5	Environmental Soil Sampling	May 27, 2016, revision 0	No
CA-6	Test Pitting	March 17, 2016, revision 0	No
CA-7	Headspace Field Screening	May 20, 2016, revision 0	No
CA-8	Monitoring Well Installation	October 20, 2017, revision 1	No
CA-9	Monitoring Well Development	October 20, 2017, revision 1	No
CA-10	Monitoring Well Gauging	August 29, 2016, revision 0	No
CA-11	Water Quality Field Instrument Calibration	September 18, 2017, revision 0	No
CA-12	Low-Flow Groundwater Sampling	October 11, 2017, revision 2	No
CA-13	Surface Water and Sediment Sampling	September 9, 2016, revision 0	No
CA-16	Chain of Custody Procedure	November 11, 2017, revision 0	No
CA-17	Packaging and Shipping Samples	August 22, 2017, revision 0	No
CA-23	Collection of PCB-Containing Building Material and Substrate Samples	October 25, 2017, revision 0	No
CA-24	Bedrock Drilling, Well Installation, and Packer Operation	October 31, 2017, revision 0	No
CA-26	Incremental Sampling Methodology	October 31, 2017, revision 0	No

WORKSHEET #22 – FIELD EQUIPMENT CALIBRATION, MAINTENANCE, TESTING, AND INSPECTION

The following data collection equipment will be used during field activities:

- PID
- Water level meter/oil water interface probe
- Multi-parameter meter (Yellow Springs Instrument [YSI], or similar) with membrane electrode probe for dissolved oxygen (DO) capable of reading greater than 4.8 milligrams per liter (mg/L) and pH, temperature, conductivity, and oxidation reduction potential (ORP)
- Turbidity meter
- GPS
- Survey rod and level

Preventative Maintenance

Equipment, instruments, tools, gauges, and other items owned by Credere and requiring preventive maintenance will be serviced in accordance with the manufacturer's recommendations. It will be the responsibility of the operator to adhere to this maintenance schedule and to arrange for service as required. Service to the equipment, instruments, tools, gauges, etc. shall be performed by qualified personnel. Maintenance of field equipment from vendors is the responsibility of the vendors.

Maintenance records will be documented and traceable to the specific equipment, instruments, and tools. Critical spare parts will be stored for availability and use in order to reduce downtime. In the event that an instrument needs to be replaced during the field program, replacement equipment will be obtained either from Credere's equipment supply or from an equipment rental vendor, depending on availability.

Equipment	Maintenance	Responsible Person	Frequency	Acceptance Criteria	Corrective Action
PID	Visually inspect	Field Lead	Daily prior to use	No visual defect; conformance with manufacturer standards	Repair or use alternate equipment
	Check battery life	Field Lead	Close of each day		Charge equipment
	Inspection	Equipment Manager	Monthly and after each field job		Professional maintenance
Water level indicator/interface probe	Clean and visually inspect probe	Field Lead	Daily prior to use	No visual defect; conformance with manufacturer standards	Repair or use alternate equipment
	Inspection	Equipment Manager	Monthly and after each field job		Professional maintenance

Equipment	Maintenance	Responsible Person	Frequency	Acceptance Criteria	Corrective Action
Multi-parameter meter	Clean and visually inspect probe	Field Lead	Daily prior to use	No visual defect; conformance with manufacturer standards	Repair or use alternate equipment
	Check battery life	Field Lead	Close of each day		Charge equipment
	Inspection	Equipment Manager	Monthly and after each field job		Professional maintenance
Turbidity meter	Clean and visually inspect probe	Field Lead	Daily prior to use	No visual defect; conformance with manufacturer standards	Repair or use alternate equipment
	Check battery life	Field lead	Close of each day		Charge/replace batteries
	Inspection	Equipment Manager	Monthly and after each field job		Professional maintenance
GPS	Clean and visually inspect probe, check battery life	Field Lead	Daily prior to use	No visual defect; conformance with manufacturer standards	Repair or use alternate equipment
	Check battery life	Field lead	Close of each day		Charge GPS
	Inspection	Equipment Manager	Monthly and after each field job		Professional maintenance
Survey rod and level	Clean and visually inspect probe	Field Lead	Daily prior to use	No visual defect; conformance with manufacturer standards	Repair or use alternate equipment
	Inspection	Equipment Manager	Monthly and after each field job		Professional maintenance/replace

Calibration and Corrective Action

Field equipment will be checked and calibrated, if required, in accordance with the procedures and frequency summarized below. The calibration procedures will conform to manufacturer's standard instructions and equipment will be calibrated to within the allowable tolerances established by the manufacturer. Records of instrument calibration will be maintained by field personnel for each project/field mobilization. If proper calibration is not achieved, Credere will attempt to recalibrate the equipment until the allowable tolerances are met or the equipment will be repaired/replaced/substituted.

Equipment	Calibration	Frequency	Acceptance Criteria	Corrective Action
PID	Check calibration at site using span gas	Beginning and end of each field day. Bump checked as needed.	+/-10% of true value	Recalibrate; replace instrument if criteria exceeded*
	Factory calibration	As needed	Cannot calibrate daily to within 10% of span gas	Factory calibration
Multi-parameter meter	Calibrate DO, pH, Specific Conductivity, and ORP probes per Operators' Manual, check temperature	Beginning of each field day with a check at the end of each field day.	pH = +/- 10% of standard Spec. Cond. = +/- 10% of standard ORP = +/- 10% of standard DO = +/- 10% of temperature dependent DO saturation in deionized (DI) water Turbidity = +/- 10% of true value	Recalibrate; replace instrument if criteria exceeded*
	Factory calibration	As needed	Cannot calibrate daily to within above	Factory calibration
Water level indicator	Perform check of accuracy	As needed.	+/- 0.1 ft	Recalibrate; replace if criteria exceeded*
Turbidity meter	3 calibration standards	Beginning of each field day with a check at the end of each field day	Standards dependent on make and model	Recalibrate; replace instrument if criteria exceeded*
	Factory calibration	As needed	Cannot calibrate daily to within 10% of standards	Factory calibration
GPS	Benchmark check, if available	Prior to use in the field to check reference point	± 1 foot	Note discrepancy in field notes
	Software updates and setting reset	As needed	Known benchmark cannot be achieved at 3 sites	Attempt known benchmark

*Until the instruments are replaced, deviations from the acceptance criteria will be noted on the field sheet.

WORKSHEET #23 – ANALYTICAL SOPS

The following Credere field SOPs and analytical SOPs from ARA and their subcontracted lab, TAS, will be used during this project. Copies of these SOPs are included as **Appendix C** for screening SOPs and **Appendix D** for definitive analytical SOPs.

SOP #	Title, Date, Revision	SOP Type	Matrix/Analytical Group	Equipment	Modified for Project
CA-7	Headspace and Field Screening, May 20, 2016, revision 0	Screening	Soil, groundwater, and sediment/VOCs	PID	No
CA-12	Groundwater Sampling, May 2, 2017, revision 2	Screening	Groundwater/DO, ORP, conductivity, pH, temperature	Multi-parameter sonde	No
QA-801	Sample Readiness, March 2016, revision 3	Prepation	Soil/All for ISM	NA	No
QA-5120	Analysis of VOCs in Water and Solid Samples by EPA Method 8260C, July 2017, revision 14	Definitive	Soil, groundwater, and sediment/VOCs	GC/MS	No
QA-5125	Preparation of Solid Samples for Volatile Organic Analyses by 5035A, February 2017, revision 8	Preparation	Soil and sediment/VOCs, VPH	NA	No
WS-MS-0008	Determination of Polycyclic Aromatic Hydrocarbons (PAHs) by GC/MS-SIM Internal Standard Technique, June 28, 2016, revision 2.7	Definitive	Soil, groundwater, and sediment/PAHs	GC/MS	No
WS-OP-0001	Extraction of Semivolatile Organic Compounds by Method 8270C, Based on SW-846 3500 Series and 3600 Series, and PAH-SIM by Internal Standard and Isotope Dilution Procedures [Methods 3510C, 3550B/C, 3546 and 3580], Revision 5.1, Effective 07/27/2017	Extraction	Soil and Water/PAHs	NA	No
QA-5515	Method 8270D – PAHs, Base/Neutrals and Acids, July 2017, revision 11	Definitive	Soil, groundwater, and sediment/SVOCs	GC/MS	No
QA-5524	Seperatory Funnel Liquid-Liquid Extraction by EPA Method 3510C, July 2017, revision 4	Extraction	Groundwater/ SVOCs, EPH, PCBs, Pesticides	NA	No
QA-5520	Ultrasonic Extraction by EPA Method 3550C, May 2016, revision 4	Extraction	Soil and sediment/SVOCs	NA	No
QA-5522	Microwave Extraction by EPA Method 3546, May 2016, revision 5	Extraction	Soil and sediment/SVOCs, EPH, PCBs, pesticides	NA	No
QA-5605	Determination of Metals and Trace Elements in Water, Solids, and Wastes by Inductively Coupled Plasma-Mass Spectrometry by 200.8/6020A, July 2017, revision 4	Definitive/ Digestion	Soil, groundwater, and sediment/metals	ICP/MS	No

SOP #	Title, Date, Revision	SOP Type	Matrix/Analytical Group	Equipment	Modified for Project
QA-5813	Hexavalent Chromium SM 3500 Cr-B and SW-846 7196A, rev 7, 3/16	Definitive	Soil, groundwater, and sediment/ Hexavalent Chromium	Spectrophotometer	No
QA-5600	Mercury Analysis by Cold Vapor Methods 245.1, 7470A/7471B, July 2017, revision 16	Definitive	Soil, groundwater, and sediment/mercury	CVAA	No
QA-5313	Extractable Petroleum Hydrocarbons MADEP 2004-1.1, February 2017, revision 14	Definitive	Soil, groundwater and sediment/EPH	GC/FID, GC/MS	No
QA-5130	Volatile Petroleum Hydrocarbons by MADEP VPH-04-1.1, April 2016, revision 7	Definitive	Soil, groundwater and sediment/VPH	GC/PID-FID	No
QA-5303	Analysis of Polychlorinated Biphenyls in Soil and Water Extracts by EPA 8082A, July 2017, revision 19	Definitive	Soil, groundwater, and sediment/PCBs	GC/ECD	No
QA-5305	Soxhlet Extraction by EPA Method 3540C, February 2017, revision 8	Extraction	Soil and concrete/PCBs	NA	No
WS-LC-0009	Determination of Nitroaromatics, Nitramines, and Specialty Explosives Based on Method 8330, SW-846, July 15, 2016, revision 5.4	Definitive	Soil, groundwater, and sediment/explosives	HPLC/UV	No
WS-ID-0007	Analysis and Preparation of Tetra- through Octo-Chlorinated Dioxins and Furans by Isotope Dilution (HRGC/HRMS) by Method 1613B, June 15, 2017, revision 4.1	Definitive	Soil/dioxins, furans	HRGC/HRMS	No
WS-IDP-0007	Preparation of Samples for Tetra- through Octa CHlroinated Dioxin and Furan by Isotope Dilution HRGC/HRMS by Method 1613B, August 16, 2017, revision 3.3	Preparation	Soil/dioxin, furan	NA	No
WS-IDP-005	Preparation of Samples for Anlysis of Polychlorinated Dioxin and Furans for Analysis HRGC/HRMS, February 7, 2017, revision 2.4	Preparation	Soil/dioxin, furans	NA	No
QA-5304	Preparation & Analysis of Organo-Chlorine Pesticides in Soil and Water Samples by method 8081B, July 2017, revision 11	Definitive	Soil and groundwater/pesticides	GC-ECD	No
QA-5522	Microwave Extraction by EPA Method 3546, March 2016, revision 5	Extraction	Soil and sediment/all	NA	No
QA-5851	pH by Method SM 4500 H+B and SW-846 9045C, February 2017, revision 8	Definitive	Soil and sediment/pH	pH meter	No

GC – gas chromatography MS – mass spectrometry ICP – inductively coupled plasma
HPLC – high performance liquid chromatography LC – liquid chromatography
CVAA – cold vapor atomic absorption ECD – electron capture detector UV – ultra violet
FID – flame ionization detector

WORKSHEET #24 - ANALYTICAL INSTRUMENT CALIBRATION

Laboratory: Absolute Resource Associates, Portsmouth, New Hampshire
Instrument: GC/MS
Method/SOP: VOC/QA-5120
Responsible Person: Analyst, Department Manager
Typical Calibration Range: 2-500 µg/L

Calibration Procedure	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (ICAL). A minimum 5-point calibration is required for all VOCs	At instrument set-up, prior to sample analysis	Each analyte must meet one of the three options below: Option 1: Relative Standard Deviation (RSD) for each analyte ≤ 15%; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.997$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.997$.	Correct problem then repeat ICAL.
Establish Retention Time (RT) Window Position	During setup of method and if acceptance criteria is not met.	The position shall be set using the midpoint standard of the ICAL curve. 0.3 minutes is used to calculate a window size for each analyte.	NA.
Evaluation of Relative Retention Times (RRTs)	With each QC and sample.	RRT of each reported analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 20% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within ± 20% of true value. All reported analytes and surrogates within 50-150% for end of analytical batch CCV.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.
Continuing Calibration Blank (CCB)	Before beginning a sample sequence	No analytes detected > LOQ.	Correct the problem, then re-prepare and reanalyze.
BFB Tune	Every 12 hours	Criteria listed Table 1, current revision of SOP QA-5120	Retune and/or clean source.

BFB – bromofluorobenzene

Laboratory: Test America, Sacramento, California
Instrument: GC/MS
Method/SOP: PAHs/WS-MS-0008
Responsible Person: Analyst, Department Manager
Calibration Range: 10-5,000 µg/L

Calibration Procedure	Frequency	Acceptance Criteria	Corrective Action
Check of mass spectral ion intensities (tuning procedure) using DFTPP	Prior to ICAL and at the beginning of each 12-hour period.	Refer to method/SOP for specific ion criteria.	Retune instrument and verify.
Performance Check	Prior to ICAL and at the beginning of each 12-hour period.	DDT breakdown ≤ 20%	Perform instrument maintenance as needed, then repeat check.
Minimum five-point initial calibration for target analytes, lowest concentration standard at or near the reporting limit. (ICAL)	Initial calibration prior to sample analysis	RSD for each analyte ≤ 20% -or- linear least squares regression for each analyte: r2 ≥ 0.99; Min RRFs as stated in SOP.	Verify standard solutions still valid, perform instrument maintenance as needed, then repeat the ICAL.
Second-source calibration verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 20% of true value.	Correct problem, and verify second source standard. Rerun verification. If still fails, repeat initial calibration.
Retention Time Window Position Establishment	Once per ICAL, and at the beginning of the analytical sequence for each analyte and surrogate.	Set position using the mid-point standard of the ICAL when ICAL is performed. On days when ICAL is not performed, use initial CCV.	NA
Daily calibration verification	Daily, prior to sample analysis and after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within ± 20% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV.	Evaluate failure and impact on samples. If samples non-detect for analytes which have a high bias, report non-detect results with case narrative comment. For closing CCVs, if compounds are not identified as critical compounds of concern report results with qualifiers. For closing CCVs, if the compound is identified as a critical compound of concern, then recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.

Calibration Procedure	Frequency	Acceptance Criteria	Corrective Action
Internal Standards	During acquisition of calibration standard.	Areas within -50% to +100% of last ICAL mid-point for each CCV. ON days when the ICAL is not performed, the initial CCV is used.	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning

Laboratory: Absolute Resource Associates, Portsmouth, New Hampshire
Instrument: GC/MS
Method/SOP: SVOCs/QA-5515
Responsible Person: Analyst, Department Manager
Calibration Range: 5-160 µg/L

Calibration Procedure	Frequency	Acceptance Criteria	Corrective Action
Check of mass spectral ion intensities (tuning procedure) using DFTPP	Prior to ICAL and at the beginning of each 12-hour period.	Refer to method/SOP for specific ion criteria.	Retune instrument and verify.
Performance Check	Prior to ICAL and at the beginning of each 12-hour period.	DDT breakdown ≤ 20%	Perform instrument maintenance as needed, then repeat check.
Minimum five-point initial calibration for target analytes, lowest concentration standard at or near the reporting limit. (ICAL)	Initial calibration prior to sample analysis	RSD for each analyte ≤ 20% -or- linear least squares regression for each analyte: r2 ≥ 0.99; Min RRFs as stated in SOP.	Verify standard solutions still valid, perform instrument maintenance as needed, then repeat the ICAL.
Second-source calibration verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 20% of true value.	Correct problem, and verify second source standard. Rerun verification. If still fails, repeat initial calibration.
Retention Time Window Position Establishment	Once per ICAL, and at the beginning of the analytical sequence for each analyte and surrogate.	Set position using the mid-point standard of the ICAL when ICAL is performed. On days when ICAL is not performed, use initial CCV.	NA

Calibration Procedure	Frequency	Acceptance Criteria	Corrective Action
Daily calibration verification	Daily, prior to sample analysis and after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within ± 20% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV.	Evaluate failure and impact on samples. If samples non-detect for analytes which have a high bias, report non-detect results with case narrative comment. For closing CCVs, if compounds are not identified as critical compounds of concern report results with qualifiers. For closing CCVs, if the compound is identified as a critical compound of concern, then recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.
Internal Standards	During acquisition of calibration standard.	Areas within -50% to +100% of last ICAL mid-point for each CCV. ON days when the ICAL is not performed, the initial CCV is used.	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning

Laboratory: Absolute Resource Associates, Portsmouth, New Hampshire
Instrument: ICP/MS
Method/SOP: Metals/QA-5605
Responsible Person: Analyst, Department Manager
Typical Calibration Range: 5-5000 µg/L

Calibration Procedure	Frequency	Acceptance Criteria	Corrective Action
Tune	Daily prior to calibration.	Mass calibration must be within 0.1 atomic mass unit (amu) from the true value. Resolution must be <0.9 amu full width at 10% peak height. Four injections – percent relative standard deviation (%RSD) must be <5%.	Perform necessary equipment maintenance.
ICAL - 1 point calibration plus blank	Daily ICAL prior to sample analysis.	Minimum of one point calibration plus a blank per manufacturer's guidelines.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.
ICV	Once after each ICAL, and before beginning a sample run.	Percent recovery (%R) must be within 90-110% for all project compounds.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.
CCB	Before beginning a sample sequence, after every 10 samples and at end of the analysis sequence.	No analytes detected > LOQ.	Correct the problem, then re-prepare and reanalyze.
CCV	After every 10 samples and at the end of each run sequence.	%R must be within 90-110% for all project compounds.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.
Interference	Daily, before	ICS solution A (ICSA) recoveries must be	Correct the problem, then re-

Calibration Procedure	Frequency	Acceptance Criteria	Corrective Action
Check Sample (ICS) - ICSA & ICSAB	sample injections	less than the absolute value of the LOD and ICS solution AB (ICSAB) %R must be within 80-120%.	prepare checks and reanalyze all affected samples.

Laboratory: Absolute Resource Associates, Portsmouth, New Hampshire
Instrument: CVAA
Method/SOP: Mercury/QA-5600
Responsible Person: Analyst, Department Manager
Typical Calibration Range: 0.2-10 µg/L

Calibration Procedure	Frequency	Acceptance Criteria	Corrective Action
ICAL	Daily ICAL prior to sample analysis.	A 5 point calibration is required. The calibration curve must have a correlation coefficient (r^2) of 0.998 or greater	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.
ICV	Once after each ICAL, and before beginning a sample run.	Percent recovery (%R) must be within 90-110% for all project compounds.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.
CCB	Before beginning a sample sequence, after every 10 samples and at end of the analysis sequence.	No analytes detected > LOQ.	Correct the problem, then re-prepare and reanalyze.
CCV	After every 10 samples and at the end of each run sequence.	%R must be within 90-110% for all project compounds.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.

Laboratory: Absolute Resource Associates, Portsmouth, New Hampshire
Instrument: Spectrophotometer
Method/SOP: Hexavalent chromium/QA-5813
Responsible Person: Analyst, Department Manager
Typical Calibration Range: 0.01-0.5 µg/L

Calibration Procedure	Frequency	Acceptance Criteria	Corrective Action
ICAL	ICAL prior to sample analysis and as instrument QC performance requires.	A 5 point calibration is required. The calibration curve must have a correlation coefficient (r^2) of 0.995 or greater	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.
ICV	Once after each ICAL	Percent recovery (%R) must be within 85-115% for all project compounds.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.
CCB	Before beginning a sample sequence, after every 10 samples and at end of the analysis sequence	No analytes detected > LOQ.	Correct the problem, then re-prepare and reanalyze.

Calibration Procedure	Frequency	Acceptance Criteria	Corrective Action
CCV	After every 10 samples and at the end of each run sequence.	%R must be within 85-115% for all project compounds.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.

Laboratory: Absolute Resource Associates, Portsmouth, New Hampshire
Instrument: GC/FID- GC/MS
Method/SOP: EPH/QA-5313
Responsible Person: Analyst, Department Manager
Typical Calibration Range: 0.1-200 µg/L

Calibration Procedure	Frequency	Acceptance Criteria	Corrective Action
ICAL	ICAL prior to sample analysis and as instrument QC performance requires.	A 5 point calibration is required. The calibration curve must have a correlation coefficient (r^2) of 0.995 or greater or average response factor of <25%	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.
ICV	Once after each ICAL	Percent recovery (%R) must be within 80-120% for all project compounds.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.
CCB	Before beginning a sample sequence, after every 10 samples and at end of the analysis sequence	No analytes detected > LOQ.	Correct the problem, then re-prepare and reanalyze.
Check of mass spectral ion intensities (tuning procedure) using DFTPP	Prior to ICAL and at the beginning of each 12-hour period.	Refer to method/SOP for specific ion criteria.	Retune instrument and verify.
Retention Time Window Position Establishment	Once per ICAL, and at the beginning of the analytical sequence for each analyte and surrogate.	The position shall be set using the midpoint standard of the ICAL curve. 0.3 minutes is used to calculate a window size for each analyte.	NA
CCV	After every 10 samples and at the end of each run sequence.	%R must be within 80-120% for all project compounds.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.

Laboratory: Absolute Resource Associates, Portsmouth, New Hampshire
Instrument: GC/PID-FID
Method/SOP: VPH/QA-5130
Responsible Person: Analyst, Department Manager
Typical Calibration Range: 1-250 µg/)

Calibration Procedure	Frequency	Acceptance Criteria	Corrective Action
ICAL. A minimum 5-point calibration is required for targets and ranges	At instrument set-up, prior to sample analysis	Each analyte must meet one of the three options below: Option 1: Relative Standard Deviation (RSD) for each analyte ≤ 15%; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.	Correct problem then repeat ICAL.
Establish RT Window Position	During setup of method and if acceptance criteria is not met.	The position shall be set using the midpoint standard of the ICAL curve. 0.3 minutes is used to calculate a window size for each analyte.	NA.
Evaluation of RRTs	With each QC and sample.	RRT of each reported analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.
ICV	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 30% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.
CCV	Daily before sample analysis; and at the end of the analytical batch run.	All reported analytes and surrogates within ± 25% of true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.
CCB	Before beginning a sample sequence	No analytes detected > LOQ.	Correct the problem, then re-prepare and reanalyze.

Laboratory: Absolute Resource Associates, Portsmouth, New Hampshire
Instrument: GC/ECD
Method/SOP: PCBs/QA-5303
Responsible Person: Analyst, Department Manager
Typical Calibration Range: 0.2-4 µg/L

Calibration Procedure	Frequency	Acceptance Criteria	Corrective Action
ICAL	ICAL prior to sample analysis and as instrument QC performance requires.	A 5 point calibration is required. The calibration curve must have a correlation coefficient (r^2) of 0.995 or greater	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.
ICV	Once after each ICAL	Percent recovery (%R) must be within 85-115% for all project compounds.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.
CCB	Before beginning a sample sequence,	No analytes detected > LOQ.	Correct the problem, then re-prepare and reanalyze.

Calibration Procedure	Frequency	Acceptance Criteria	Corrective Action
	after every 10 samples and at end of the analysis sequence		
CCV	After every 10 samples and at the end of each run sequence.	%R must be within 85-115% for all project compounds.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.

Laboratory: Test America, Sacramento, California
Instrument: HPLC
Method/SOP: Explosives/WS-LC-0009
Responsible Person: Analyst, Department Manager
Calibration Range: 5-500µg/L

Calibration Procedure	Frequency	Acceptance Criteria	Corrective Action
Minimum five-point initial calibration for target analytes, lowest concentration standard at or below the reporting limit. (ICAL)	Initial calibration prior to sample analysis	Average RF: RSD </= 15%; Linear: R^2 >0.99, intercept < MDL.	Evaluate standards, chromatography, and detector response. If problem found with above, correct as appropriate, then repeat initial calibration
Second-source calibration verification	Immediately following ICAL.	Less than 20 % difference for target analytes and surrogates.	Evaluate data. If problem (e.g., concentrated standard, plugged injector needle) found, correct, then repeat second source verification. If still fails, repeat initial calibration.
Daily calibration verification	Before sample analysis; after every 10 field samples and at the end of the sequence.	Less than 20 % difference for reported analytes and surrogates	Evaluate standard - if response is elevated and associated samples are non-detect for that analyte, narrate. If ending CCV, and second analysis of samples due to non-compliant ending CCV, narrate. Otherwise, recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV. Corrective actions include: evaluate standard, chromatography, and detector response. Correct as appropriate (instrument maintenance, remake standards), then repeat initial calibration.

Laboratory: Test America, Sacramento, California
Instrument: HRGC/HRMS
Method/SOP: Dioxin and furans/WS-ID-0007
Responsible Person: Analyst, Department Manager
Calibration Range: 0.5/2.5/5 to 200/1000/500 pg/uL

Calibration Procedure	Frequency	Acceptance Criteria	Corrective Action
Tune / Mass Resolution Check (PFK)	At the beginning and the end of each 12-hour period of analysis.	Resolving power ≥ 10,000 at m/z=304.9842 & m/z=380.9760 + 5ppm of expected mass. Lock-mass ion between lowest and highest masses for each descriptor and level of reference ≤ 10% full-scale deflection.	Retune instrument & verify. Assess data for impact if end resolution is less than 10,000 narrate or reinject as necessary.
GC Column Performance Check (CPSM/WDM per method)	Prior to ICAL or calibration verification.	Peak separation between 2,3,7,8-TCDD and other TCDD isomers result in a valley of ≤ 25%; and identification of all first and last eluters of the eight homologue retention time windows and documentation by labeling (F/L) on the chromatogram; and absolute retention times for switching from one homologous series to the next ≥ 10 seconds for all components of the mixture.	1) Readjust windows. 2) Evaluate system. 3) Perform maintenance. 4) Reanalyze CPSM. 5) No corrective action is necessary if 2,3,7,8-TCDD is not detected and the % valley is greater than 25%.
Minimum five-point initial calibration for target analytes, lowest concentration standard at or near the reporting limit. (ICAL)	ICAL prior to sample analysis, as needed by the failure of calibration verification, and when a new lot is used as a standard source for calib verification, internal standard, or recovery standard solutions.	RSD ≤ 20% for response factors for 17 unlabeled isomers & ≤30% for labelled IS, and ion abundance ratios within limits specified in SOP; and S/N ≥ 10:1for target analytes.	Evaluate standard and instrument response. If problem with instrument (autosampler failure, response poor, etc.) or standards, correct as appropriate, then repeat initial calibration.
Second-source calibration verification	Immediately following ICAL.	Ion abundance ratios in accordance with SOP; and RF within ± 30%D of average RF from ICAL.	Evaluate standards and instrument response. If standard issue, repeat or remake then repeat standard as appropriate. If still fails, repeat initial calibration
CCV	At the beginning of each 12-hour period, and at the end of each analytical sequence.	Ion abundance ratios in accordance with SOP; and acceptance criteria listed in Method 1613B Table 6 "VER".	Correct problem, repeat calibration verification. If fails, repeat ICAL and reanalyze all samples analyzed since last successful CCV.

Laboratory: Absolute Resource Associates, Portsmouth, New Hampshire
Instrument: GC-ECD
Method/SOP: Pesticides/QC-5304
Responsible Person: Analyst, Department Manager
Typical Calibration Range: 1-100 ng/mL single pesticides, 50-1000 for toxaphene, and 10-1000 for technical chlordane

Calibration Procedure	Frequency	Acceptance Criteria	Corrective Action
ICAL	ICAL prior to sample analysis and as instrument QC performance requires.	A 5 point calibration is required. The calibration curve must have a correlation coefficient (r^2) of 0.99 or greater	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.
ICV	Once after each ICAL	Percent recovery (%R) must be within 85-115% for all project compounds.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.
CCB	Before beginning a sample sequence, after every 10 samples and at end of the analysis sequence	No analytes detected > LOQ.	Correct the problem, then re-prepare and reanalyze.
CCV	After every 10 samples and at the end of each run sequence.	%R must be within 85-115% for all project compounds.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.

Laboratory: Absolute Resource Associates, Portsmouth, New Hampshire
Instrument: pH meter
Method/SOP: pH/QA-5851
Responsible Person: Analyst, Department Manager
Calibration Range: 2-12 µg/L

Calibration Procedure	Frequency	Acceptance Criteria	Corrective Action
ICAL	Daily ICAL and every 4 hours prior to sample analysis.	Two point calibration.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.
ICV	Once after each ICAL	True value must be within +/- 0.06 pH units	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.
CCV	After every 10 samples and at the end of each run sequence.	True value must be within +/- 0.06 pH units	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.

WORKSHEET #25 - ANALYTICAL INSTRUMENT AND EQUIPMENT MAINTENANCE, TESTING, AND INSPECTION

Instrument	Maintenance Activity	Testing Activity/ Method/SOP	Inspection	Frequency	Acceptance Criteria	Corrective Action	Responsible Person
GC-ECD or FID/PID	Change septum, clean injection port, change or clip column, install new liner	Detector signals and chromatogram review	Instrument performance and sensitivity	As needed	CCV passes criteria	Reinspect injector port, cut additional column, reanalyze CCV, recalibrate instrument	ARA Chemist
GC/MS	Clean source, maintain vacuum pumps, replace filament	Tuning	Instrument performance and sensitivity	Service vacuum pumps, other maintenance as needed	Tune and CCV pass criteria	Recalibrate instrument	ARA Chemist
GC/MS	Change septum, clean injection port, change or clip column, install new liner, change trap	Sensitivity check	Instrument performance and sensitivity	Daily or as needed	Tune and CCV pass criteria	Reinspect injector port, cut additional column, reanalyze CCV, recalibrate instrument	ARA Chemist
LC/MS/ MS	Replace columns as needed, check eluent reservoirs	Sensitivity check	Instrument performance and sensitivity	Daily or as needed	CCV pass criteria	Recalibrate	TAS Chemist
HPLC/UV	Replace columns as needed, check eluent reservoirs	Sensitivity check	Instrument performance and sensitivity	Daily or as needed	CCV pass criteria	Recalibrate	TAS Chemist
GC/ HRMS	Parameter Setup	Physical check	Physical check	Initially; prior to DCC	Correct Parameters	Reset if incorrect	TAS Chemist
GC/ HRMS	Tune Check	Instrument Performance	Conformance to instrument tuning.	Initially; prior to DCC	Compliance to ion abundance criteria	Correct the problem and repeat tune check	TAS Chemist

WORKSHEET #26 & 27 - SAMPLE HANDLING, CUSTODY, AND DISPOSAL

Sampling Organization: Credere Associates, LLC
Laboratory: ARA and their subcontractor TAS
Method of Sample Delivery: Courier
Number of Days from Reporting Until Sample Disposal: 30 days or as directed

Activity	Organization and Title or Position of Person Responsible for the Activity	SOP Reference
Sample Labeling	Credere/Allison Drouin & Matthew Kennedy	CA-1
Chain of Custody Completion		CA-1
Packaging		CA-17
Shipping Coordination		CA-17
Sample Receipt, Inspection & Login	ARA/Jane Stratton	QA-400
Sample Custody and Storage		QA-400
Sample Disposal		QA-5001
Sample Readiness		QA-801
Sample Receipt, Inspection & Login	TAS/	WS-QA-0003
Sample Custody and Storage		WS-QA-0034
Sample Disposal		WS-EHS-0001

WORKSHEET #28 – ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION

Matrix: Soil, water, sediment
Analytical Group: VOCs
Analytical Method: 8260C
Laboratory SOP: QA-5120

QC Sample	Frequency	Acceptance Criteria	Corrective Action	Title of Corrective Action Responsibility	Project Specific MPCs
ICV	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 20% of true value.	Rerun ICV. If that fails, correct problem and repeat ICAL.	Analyst, Department Manager	Same as Method/SOP QC Acceptance Limits.
CCV	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within ± 20% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Analyst, Department Manager	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per sample delivery group (SDG) or every 20 samples as provided.	All reported analytes within ± 30% of true value. RPD of all analytes 20% for waters and 30% for solids (between MS and MSD)	Qualify data. Examine the project- specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Method blank	Per laboratory sample batch (once every 12 hour period)	No analytes detected > LOQ.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Internal standards (IS)	Three per sample: Fluorobenzene Chlorobenzene-D5 1,4-dichlorobenzene-D4	Retention time within ± 0.3 seconds from ICAL. IS area must be 50-200% of the initial calibration curve (avg).	Inspect mass spectrometer or gas chromatograph for malfunctions. Mandatory reanalysis of samples analyzed while system was malfunctioning.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Surrogates	3 per sample: dibromofluoromethane toluene-D8 4-bromofluorobenzene	78-114% 88-110% 86-115%	Correct problem, then reprep and reanalyze all samples with failing surrogates. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.

QC Sample	Frequency	Acceptance Criteria	Corrective Action	Title of Corrective Action Responsibility	Project Specific MPCs
LCS; blank spike/LCSD	One per preparation batch of twenty or fewer samples of similar matrix.	All reported analytes must be within 70% and 130% of true value, except for difficult compounds (acetone, MEK,MIBK, 2-Hexanone, dichlorodifluoromethane, bromomethane, chloromethane, 1,4-dioxane) which may exhibit recoveries between 40% and 160%. RPD of all analytes 20% for waters and 30% for solids (between LCS & LCSD)	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.

Matrix: Soil, water, and sediment
Analytical Group: SVOCs and PAHs
Analytical Method: 8270D
Laboratory SOP: QA-5515

QC Sample	Frequency	Acceptance Criteria	Corrective Action	Title of Corrective Action Responsibility	Project Specific MPCs
Internal standards	During acquisition of calibration standard, samples, and QC check samples: 1,4-Dichlorobenzene-d4, naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12, and perylene-d12	Retention time within ± 0.3 seconds from ICAL. IS area must be 50-200% of the initial calibration curve (avg). or daily CCV when curve not analyzed	Inspect mass spectrometer or gas chromatograph for malfunctions. Mandatory reanalysis of samples analyzed while system was malfunctioning.	Analyst, Laboratory Department Manager	Same as Method/SOP QC Acceptance Limits.

QC Sample	Frequency	Acceptance Criteria	Corrective Action	Title of Corrective Action Responsibility	Project Specific MPCs
Method blank	Per laboratory sample batch (once every 24 hour period)	No analytes detected > LOQ.	Correct problem. If required, reprep and reanalyze BLK and all samples processed with the contaminated blank or qualify if re-extraction isn't possible. No impact if samples are ND.	Analyst, Laboratory Department Manager	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One MS/MSD per analytical/preparation batch	All reported analytes within 30-130% of true value for acid compounds and 40-140% for base-neutral compounds. RPD of all analytes 20% for waters and 30% for solids (between MS and MSD)	Verify recovery in associated LCS/D and re-extract if failures are not associated with matrix interference. Qualify failures related to matrix interference.	Analyst, Laboratory Department Manager	Same as Method/SOP QC Acceptance Limits.
LCS/D	One LCS/D per analytical/preparation batch	All reported analytes within 30-130% of true value for acid compounds and 40-140% for base-neutral compounds. RPD of all analytes 20% for waters and 30% for solids (between LCS and LCSD)	If LCS/D has high bias, and samples non-detect, report with case narrative comment. If LCS/D has low bias for a non-chemical of concern and samples have no detection, report with case narrative comment. Recoveries outside the criteria for chemicals of concern should be re-extracted when possible.	Analyst, Laboratory Department Manager	Same as Method/SOP QC Acceptance Limits.

QC Sample	Frequency	Acceptance Criteria	Corrective Action	Title of Corrective Action Responsibility	Project Specific MPCs
Surrogate standards	All field and QC samples. ABN surrogates: 2-fluorophenol, phenol-d5, 2,4,6-tribromophenol, nitrobenzene-d5, 2-fluorobiphenyl, terphenyl-d14, and o-terphenyl.	21-100% 10-102% 10-123% 35-114% 43-116% 33-141% 33-141%	Evaluate data, if samples non-detect and surrogate recovery is above upper limits, report with case narrative comment. If obvious chromatographic interference is present, report with narrative. Otherwise, re-extract and reanalyze.	Analyst, Laboratory Department Manager	Same as Method/SOP QC Acceptance Limits.

Matrix: Water, and sediment
Analytical Group: PAHs with SIM
Analytical Method: 8270D with SIM
Laboratory SOP: WS-MS-0008

QC Sample	Frequency	Acceptance Criteria	Corrective Action	Title of Corrective Action Responsibility	Project Specific MPCs
Internal standards	During acquisition of calibration standard, samples, and QC check samples	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within - 50% to +100% of ICAL midpoint standard. On days with the ICAL is not analyzed, use the initial CCV.	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning in accordance with QSM requirements. If field samples still outside criteria, qualify data and explain in case narrative.	Lab Manager / Analyst	Precisions and Accuracy/Bias
Method blank	One per analytical batch	No target analytes ≥ ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). For common laboratory contaminants, no analytes detected >RL in accordance with QSM requirements	Verify instrument clean (evaluate calibration blank & samples prior to method blank), then reanalyze. Evaluate to determine if systematic issue within laboratory, correct, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank in accordance with QSM requirements.	Lab Manager / Analyst	Accuracy/Bias Contamination

QC Sample	Frequency	Acceptance Criteria	Corrective Action	Title of Corrective Action Responsibility	Project Specific MPCs
MS/MSD	One MS/MSD per analytical/preparation batch	Recovery: QSM limits (if available) or current in-house limits if no QSM limits published. RPD: RPD of all analytes ≤ 40% (between MS and MSD).	If not related to matrix interference, re-extract and reanalyze MS/MSD.	Lab Manager / Analyst	Precision and Accuracy/Bias
LCS	One LCS per analytical/preparation batch	QSM limits (if available) or current in-house limits if no QSM limits published.	Reanalyze LCS once. If acceptable, report. Otherwise, if exceedance is not a critical chemical of concern as identified by the project team, evaluate for sporadic marginal exceedance (SME). If acceptable, report with case narrative comment. If not acceptable for SME, evaluate samples for detections, and LCS for high bias. If LCS has high bias, and samples non-detect, report with case narrative comment. If LCS has low bias, or if there are detections for critical chemicals of concern, evaluate and reprep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.	Lab Manager / Analyst	Precision and Accuracy/Bias
Surrogate standards (including Fluoranthene-D10 and 2-methylnaphthalene-d10)	All field and QC samples.	QSM limits (if available) or current in-house limits if no QSM limits published.	Evaluate data, if samples non-detect and surrogate recovery is above upper limits, report with case narrative comment. If obvious chromatographic interference is present, report with narrative comment. Otherwise, re-extract and reanalyze.	Lab Manager / Analyst	Accuracy/Bias

Matrix: Soil, water, sediment
Analytical Group: Metals
Analytical Method: 6020A
Laboratory SOP: QA-5605

QC Sample	Frequency	Acceptance Criteria	Corrective Action	Title of Corrective Action Responsibility	Project Specific MPCs
ICV	Once after each ICAL, before beginning a sample run.	All reported analytes must be within ± 10% of true value for ICV and ± 30% of true value for Low Level ICV	Rerun ICV. If that fails, correct problem and repeat ICAL.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.

QC Sample	Frequency	Acceptance Criteria	Corrective Action	Title of Corrective Action Responsibility	Project Specific MPCs
IS	One used per element. IS mix includes: Li, Sc, Tb, Y, In, Bi, and Ho	Recovery between 30% and 120%.	Dilute and re-analyze sample. Flush the instrument. If the problem persists, investigate possible partially blocked sampling cone, unfavorable mixing in the spray chamber or mixing T, or change in tuning conditions of the instrument.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
CCV	After every 10 samples and at the end of each run sequence.	All reported analytes must be within ± 10% of true value	Rerun calibration verification. If that fails, correct problem then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per preparatory batch.	All reported analytes within ± 25% of true value. RPD of all analytes 20% for waters and solids (between MS and MSD)	Qualify data. Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Method blank	One per preparatory batch.	No analytes detected > LOQ	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
LCS/LCSD	One per preparatory batch.	All reported analytes within ± 20% of true value for waters and 95% confidence limit for solid CRM. RPD of all reported analytes 20% for waters and solids (between LCS and LCSD).	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.

Matrix: Soil, water, sediment
Analytical Group: Mercury
Analytical Method: 7471B/7470A
Laboratory SOP: QA-5600

QC Sample	Frequency	Acceptance Criteria	Corrective Action	Title of Corrective Action Responsibility	Project Specific MPCs
ICV	Once after each ICAL, before beginning a sample run.	Percent recovery (%R) must be within 90-110%.	Rerun ICV. If that fails, correct problem and repeat ICAL.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.

QC Sample	Frequency	Acceptance Criteria	Corrective Action	Title of Corrective Action Responsibility	Project Specific MPCs
CCV	After every 10 samples and at the end of each run sequence.	Percent recovery (%R) must be within 90-110%.	Rerun calibration verification. If that fails, correct problem then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
MS	1 per 10 samples.	Percent recovery (%R) must be within 80-120%.	Qualify data. Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Duplicate	1 per 20 samples.	Relative percent difference must be ±20% for waters and ±30% for solids	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Method blank	One per preparatory batch.	No analytes detected > LOQ.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
LCS/LCSD	One per preparatory batch.	Percent recovery (%R) must be within 80-120%. Relative percent difference must be ±20% for waters and ±30% for solids (between LCS and LCSD)	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.

Matrix: Soil, water, sediment
Analytical Group: Hexavalent chromium
Analytical Method: SM 3500 Cr-B and SW-846 7196A
Laboratory SOP: QA-5813

QC Sample	Frequency	Acceptance Criteria	Corrective Action	Title of Corrective Action Responsibility	Project Specific MPCs
ICV	Once after each ICAL, before beginning a sample run.	Percent recovery (%R) must be within 85-115%.	Rerun ICV. If that fails, correct problem and repeat ICAL.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
CCV	After every 10 samples and at the end of each run sequence.	Percent recovery (%R) must be within 85-115%.	Rerun calibration verification. If that fails, correct problem then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
MS	1 per 10 samples.	Percent recovery (%R) must be within 75-125%.	Qualify data. Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.

QC Sample	Frequency	Acceptance Criteria	Corrective Action	Title of Corrective Action Responsibility	Project Specific MPCs
Duplicate	1 per 20 samples.	Relative percent difference must be ±20%	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Method blank	One per preparatory batch.	No analytes detected > LOQ.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
LCS/LCSD	One per preparatory batch.	Percent recovery (%R) must be within 85-115%. 95% confidence interval for CRM. Relative percent difference must be ±20. (between LCS and LCSD)	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.

Matrix: Soil, water, sediment
Analytical Group: EPH
Analytical Method: MADEP EPH-04-1.1
Laboratory SOP: QA-5313

QC Sample	Frequency	Acceptance Criteria	Corrective Action	Title of Corrective Action Responsibility	Project Specific MPCs
ICV	Once after each ICAL.	Percent recovery (%R) must be within 80-120%.	Rerun ICV. If that fails, correct problem and repeat ICAL.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
CCV	After every 10 samples and at the end of each run sequence.	Percent recovery (%R) must be within 80-120%.	Rerun calibration verification. If that fails, correct problem then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per preparatory batch.	Percent recovery (%R) must be within 40-140%. Relative percent difference must be ±50%.	Qualify data. Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Method blank	One per preparatory batch.	No analytes detected > LOQ	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.

QC Sample	Frequency	Acceptance Criteria	Corrective Action	Title of Corrective Action Responsibility	Project Specific MPCs
LCS/LCSD	One per preparatory batch.	Percent recovery (%R) must be within 40-140%. Relative percent difference must be ±25%.	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Surrogates	4 per sample: chloro-octadecane 2-fluorobiphenyl 2-bromonapthalene O-terphenyl	40-140% For each	Correct problem, then reprep and reanalyze all samples with failing surrogates. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.

Matrix: Soil, water, sediment
Analytical Group: VPH
Analytical Method: MADEP VPH-04-1.1
Laboratory SOP: QA-5130

QC Sample	Frequency	Acceptance Criteria	Corrective Action	Title of Corrective Action Responsibility	Project Specific MPCs
ICV	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 25% of true value.	Rerun ICV. If that fails, correct problem and repeat ICAL.	Analyst, Department Manager	Same as Method/SOP QC Acceptance Limits.
CCV	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within ± 25% of true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Analyst, Department Manager	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per sample delivery group (SDG) or every 20 samples as provided.	All reported analytes within ± 30% of true value. RPD of all analytes 50% (between MS and MSD)	Qualify data. Examine the project- specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Method blank	Per laboratory sample batch (once every 12 hour period)	No analytes detected > LOQ.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.

QC Sample	Frequency	Acceptance Criteria	Corrective Action	Title of Corrective Action Responsibility	Project Specific MPCs
Surrogates	3 per sample: 2,5-dibromotoluene as aliphatic (FID) 2,5-dibromotoluene as aromatic (PID) ααα-trifluorotoluene (PID- Solids only)	70-130% for each	Correct problem, then reprep and reanalyze all samples with failing surrogates. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
LCS/LCSD	One per preparation batch of twenty or fewer samples of similar matrix.	All reported analytes must be within 70% and 130% of true value. RPD of all analytes 25% . (between LCS & LCSD)	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.

Matrix: Soil, water, sediment, concrete
Analytical Group: PCBs
Analytical Method: 8082A
Laboratory SOP: QA-5303

QC Sample	Frequency	Acceptance Criteria	Corrective Action	Title of Corrective Action Responsibility	Project Specific MPCs
ICV	Once after each ICAL.	Percent recovery (%R) must be within 85-115%.	Rerun ICV. If that fails, correct problem and repeat ICAL.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
CCV	After every 10 samples and at the end of each run sequence.	Percent recovery (%R) must be within 85-115%.	Rerun calibration verification. If that fails, correct problem then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per preparatory batch, as requested	Percent recovery (%R) must be within 40-140%. The RPD limits are +/- 50 % for Aroclors.	Qualify data. Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Method blank	One per preparatory batch.	No analytes detected > LOQ.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.

QC Sample	Frequency	Acceptance Criteria	Corrective Action	Title of Corrective Action Responsibility	Project Specific MPCs
LCS/ LCSD	One per preparatory batch.	Percent recovery (%R) must be within 40-140%. Relative percent difference must be ±20% for waters and ±30% for solids.	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Surrogates	2 per sample: tetrachlorometaxylene (TCMX) and decachlorobiphenyl (DCBP)	30-150%	Correct problem, then reprep and reanalyze all samples with failing surrogates. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.

Matrix: Soil, water, sediment
Analytical Group: Explosives
Analytical Method: 8330B
Laboratory SOP: WS-LC-0009

QC Sample	Frequency	Acceptance Criteria	Corrective Action	Title of Corrective Action Responsibility	Project Specific MPCs
Method Blank	One per preparation batch	No target analytes ≥ ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).	Verify instrument clean (evaluate calibration blank & samples prior to method blank), then reanalyze. Evaluate to determine if systematic issue within laboratory, correct, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank in accordance with QSM requirements	Lab Manager / Analyst	Accuracy/Bias Contamination
Surrogates	Every sample, spiked sample, standard, and method blank	Laboratory statistically derived control limits in accordance with QSM requirements.	Evaluate data, if samples non-detect and surrogate recovery is above upper limits, report with case narrative comment. If obvious chromatographic interference is present, report with narrative comment. Otherwise, re-extract and reanalyze.	Lab Manager / Analyst	Accuracy/Bias

QC Sample	Frequency	Acceptance Criteria	Corrective Action	Title of Corrective Action Responsibility	Project Specific MPCs
LCS	One LCS per analytical/preparation batch, spiked with all analytes to be reported.	QSM limits (if available) or current in-house limits if no QSM limits published.	Reanalyze LCS once. If acceptable, report. Otherwise, if exceedance is not a critical chemical of concern as identified by the project team, evaluate for sporadic marginal exceedance (SME). If acceptable, report with case narrative comment. If not acceptable for SME, evaluate samples for detections, and LCS for high bias. If LCS has high bias, and samples non-detect, report with case narrative comment. If LCS has low bias, or if there are detections for critical chemicals of concern, evaluate and reprep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.	Lab Manager / Analyst	Accuracy/Bias
MS/MSD for all analytes	One MS/MSD pair per preparation batch	Recovery: QSM limits (if available) or current in-house limits if no QSM limits published. RPD: RPD of all analytes ≤ 20% (between MS and MSD).	If not related to matrix interference, re-extract and reanalyze MS/MSD.	Lab Manager / Analyst	Precision and Accuracy/Bias
Confirmation Analysis	All positive results must be confirmed.	Calibration and QC criteria the same as for initial/primary analysis. Results between primary and secondary column RPD ≤ 40%	Evaluate data, then report with flag to denote RPD > 40%. Narrate obvious matrix issues.	Lab Manager / Analyst	Accuracy/Bias
Soil Grinding Blank	At a minimum, prior to grinding samples, after every 10 samples, and at the end of the batch.	Blank material ground and subsampled as with the samples. Composited prior to analysis. No target analytes detected at > than 1/2 the RL (LOQ)	All blanks are reported, and sample results associated with a blank/composite of blanks are flagged/narrated if the criteria are exceeded.	Lab Manager / Analyst	Accuracy/Bias

QC Sample	Frequency	Acceptance Criteria	Corrective Action	Title of Corrective Action Responsibility	Project Specific MPCs
Grinding Standard Certified Material (SRM)	When puck mill grinding is performed, SRM prepared by outside vendor is ground for every 20 samples ground and extracted with those field samples.	QSM LCS Limits (if available) or current in-house limits if no QSM limits published.	Evaluate the data, narrate any outliers and report.	Lab Manager / Analyst	Accuracy/Bias
Soil Sample Triplicate	When incremental subsampling is performed, one sample per batch.	Three 10g subsamples are taken from a sample expected to have the highest levels of explosives. RSD < 20% for results > RL (LOQ)	Evaluate grinding and subsampling process. If sufficient material, repeat triplicate sample. If insufficient material remains, flag data.	Lab Manager / Analyst	Precision and Accuracy/Bias
[1]Recovery and precision limits are not available in the QSM for surrogates and some analytes in this analysis. When no limit is available in the QSM, the laboratory historical control limits are used (as per the QSM). Laboratory historical control limits are subject to change as a result of periodic re-evaluation. Limits in use at the time of sample analysis are available from the laboratory.					

Matrix: Soil
Analytical Group: Dioxins/furans
Analytical Method: 1613B
Laboratory SOP: WS-ID-0007

QC Sample	Frequency	Acceptance Criteria	Corrective Action	Title of Corrective Action Responsibility	Project Specific MPCs
Method Blank	One per preparation batch	Project specific criteria, if available. Otherwise, no target analytes detected ≥ 1/2 LOQ or ≥ 10% of the associated regulatory limit or ≥ 10% of the sample result for the analyte, whichever is greater. (OCDD is considered a common laboratory contaminant and treated accordingly).	Verify instrument clean (evaluate calibration blank & samples prior to method blank), then reanalyze. Evaluate to determine if systematic issue within laboratory, correct, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank in accordance with DoD QSM requirements. "Totals" are not considered "target analytes" – no corrective action or flagging is necessary for "totals".	Chemist	Accuracy/Bias Contamination

QC Sample	Frequency	Acceptance Criteria	Corrective Action	Title of Corrective Action Responsibility	Project Specific MPCs
Internal Standard Spike	Every field sample, standard and QC sample	% recovery for each IDA in the original sample (prior to dilutions) must be within acceptance criteria in Table 6/6A (blank spikes) and Table 7/7A Method 1613B (field samples).	Correct problem, then reprep and reanalyze the samples with failed IS.	Lab Manager / Analyst	Precisions and Accuracy/Bias
Laboratory Control Sample (LCS)	One per sample preparation batch	Method 1613B OPR acceptance criteria in Table 6	Reanalyze LCS once. If acceptable, report. Otherwise, if exceedance is not a critical chemical of concern as identified by the project team, evaluate for sporadic marginal exceedance (SME). If acceptable, report with case narrative comment. If not acceptable for SME, evaluate samples for detections, and LCS for high bias. If LCS has high bias, and samples non-detect, report with case narrative comment. If LCS has low bias, or if there are detections for critical chemicals of concern, evaluate and reprep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.	Lab Manager / Analyst	Accuracy/Bias Contamination
MS/MSD	One MS/MSD per analytical/preparation batch	Method 1613B OPR acceptance criteria (Table 6/6A of method), RPD ≤ 20%.	Evaluate data, if samples non-detect and surrogate recovery is above upper limits, report with case narrative comment. If obvious chromatographic interference is present, report with narrative comment. Otherwise, re-extract and reanalyze.	Lab Manager / Analyst	Precision and Accuracy/Bias

Matrix: Soil, sediment, water
Analytical Group: Pesticides
Analytical Method: 8081B
Laboratory SOP: QA-5304

QC Sample	Frequency	Acceptance Criteria	Corrective Action	Title of Corrective Action Responsibility	Project Specific MPCs
ICV	Once after each ICAL.	Percent recovery (%R) must be within 85-115%.	Rerun ICV. If that fails, correct problem and repeat ICAL.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
CCV	After every 10 samples and at the end of each run sequence.	Percent recovery (%R) must be within 85-115%.	Rerun calibration verification. If that fails, correct problem then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per preparatory batch, as requested	Percent recovery (%R) must be within 30-150%. The RPD limits are 30% for single component compounds and 50% for multi-component compounds.	Qualify data. Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Method blank	One per preparatory batch.	No analytes detected > LOQ.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
LCS/ LCSD	One per preparatory batch.	Percent recovery (%R) must be within 40-140%. Relative percent difference must be ±20% for waters and ±30% for solids.	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Surrogates	2 per sample: tetrachlorometaxylene (TCMX) and decachlorobiphenyl (DCBP)	30-150%	Correct problem, then reprep and reanalyze all samples with failing surrogates. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.

Matrix: Soil, sediment
Analytical Group: pH
Analytical Method: 9045C
Laboratory SOP: QA-5851

QC Sample	Frequency	Acceptance Criteria	Corrective Action	Title of Corrective Action Responsibility	Project Specific MPCs
ICV	Once after each ICAL	True value must be within +/- 0.06 pH units	Rerun ICV. If that fails, correct problem and repeat ICAL.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
CCV or LCS	After every 10 samples and at the end of each run sequence.	True value must be within +/- 0.06 pH units	Rerun calibration verification. If that fails, correct problem then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Duplicate	One per 10 samples and with each preparatory batch.	Result of duplicate must be within 0.2 pH units of original sample.	Qualify the result. Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.

WORKSHEET #29 – PROJECT DOCUMENTATION AND RECORDS

Record	Generation	Verification	Storage Location
Field logbook and field forms	Field Manager/Field Lead	Field Manager/Project Manager	Project File
COC			
Correspondence	Project Manager	Program Manager	
Field audit	Field Manager	Project Manager	
Data validation report	Third party validator	Field Manager/Project Manager	
Data tabulation	Field lead	Field Manager/Project Manager/Program Manager	
Laboratory reports	Analytical laboratory	Third party validator/Field Manager/Project Manager	
Chain of custody (3 days after demobilization)	Project Manager	USACE Project Manager/QC Manager	FUDSChem
Laboratory reports			
Data validation report			
SEDD File			

Data will be provided by the laboratory as a Staged Electronic Data Deliverable (SEDD) Stage 2a deliverable to also include a .pdf file of the laboratory's final data report that includes applicable chromatographs and instrument calibration QC documentation. The following table summarizes the requirements for the laboratory deliverable.

Record	VOCs	SVOCs/ PAHs	Metals	EPH	VPH	Dioxins/ furans	Pesticides	pH	PCBs	Explosives
Narrative	X	X	X	X	X	X	X	X	X	X
Summary Results	X	X	X	X	X	X	X	X	X	X
QC Results	X	X	X	X	X	X	X	X	X	X
Chromatograms	X	X		X	X		X		X	
Tentatively Identified Compounds (TICs)	X	X								
Chain of Custody	X	X	X	X	X	X	X	X	X	X

WORKSHEETS # 31, 32 & 33 – ASSESSMENTS AND CORRECTIVE ACTION

Credere quality control is ensured through compliance with Credere's Corporate Quality Control Plan dated February 12, 2016 (Credere, 2016).

Assessment				
Assessment	**Responsible Person**	**Frequency**	**Dates**	**Deliverable and Due Date**
Readiness review	Project manager/Field manager	1 week prior to mobilization	February 2018	Answers and clarifications that arise during readiness review meeting
Field sampling procedures technical systems audit (TSA)	QC Manager/Field Manager	Annually	January	Review of SOPs with updates by March
Site-specific field sampling TSA	Field Manager/Project Manager	Once per job and as needed	March 2018	Review of field documentation and submittal of feedback to field sampler
On-site analytical TSA	Project Manager	None	NA	Reliance on ELAP Certification, check ELAP certification expirations
Field documentation	Field Manager	At completion of job task	Within 3 days of demobilization	Provide feedback and notes regarding completeness and corrections
Management review	QC Manager/Program Manager	All deliverables	As needed	Edits and feedback on deliverables within 1 week of submittal

Corrective Action				
Assessment	**Responsible Person to Respond**	**Response Documentation**	**Timeframe**	**Responsible Person to Confirm Response**
Readiness review	Field Lead	NA	NA	Field Manager/Project Manager
Field sampling TSA	Field Lead	Acknowledgement of re-review of SOPs and discussion of understanding	Same day of TSA non-compliance finding and within 1 week of demobilization	Field Manager
Field Documentation	Field Lead	Field document corrections and improvements	1 week of receipt of comments on documentation	Field Manager
On-site analytical TSA	Laboratory	Updated ELAP certification	Upon receipt	Project Manager/Field Manager
Management review	Author	Updated deliverable	As needed	QC Manager/Program Manager/Project Manager as alternate

WORKSHEET #34 – DATA VERIFICATION AND VALIDATION INPUTS

Description	Verification (Completeness)	Validation (Conformance to Specifications)
Planning Documents/Records		
Contract	X	
Approved QAPP & Lab SOPs	X	
Approved Field Sampling Plan & Field SOPs	X	
Field Record		
Field Logbook and Field Forms	X	
Equipment calibration record	X	
Health and safety documentation forms	X	
Chain of custody	X	
Field sketches	X	
Analytical Data Package		
Cover Sheet	X	X
Narrative	X	X
Internal laboratory chain of custody	X	X
Sample receipt record	X	X
Sample chronology	X	X
Communication record (if any)	X	X
LOD/LOQ review	X	X
Standards traceability	X	X
Instrument calibration records	X	X
Definition of qualifiers	X	X
Results summary	X	X
QC summary with corrective actions	X	X
Raw data	X	X
Electronic data deliverable (EDD)		X

WORKSHEET #35 – DATA VERIFICATION PROCEDURE

Credere implements a multi-level review process for reports conveying results, work completed, field and analytical data, findings, conclusions, recommendations, and Credere's professional opinion. Data verification procedures are more thoroughly summarized in Credere's Corporate QC Plan (Credere, 2016) in Section 4.2 for report QC.

Records reviewed	Requirement Documents	Process Description	Responsible Person
Field Log Book, Field Forms, and Chain of Custody	QAPP, CA-1 SOP	The Field Lead is responsible to review their own documents for completeness prior to submittal for review to Field Manager/Project Manager	Field Lead
		The Project Manager or Field Manager will review for completeness, compliance with SOPs, consistency between the three, and that work is documented to have occurred in accordance with the QAPP/FSP. The event planning tools in FUDSChem will also be used to confirm completeness of a sampling program compared to the FSP.	Project Manager/Field Manager
Laboratory deliverables	QAPP	Verify that the laboratory deliverable contains all records specified in the QAPP. Check sample receipt records to ensure sample condition upon receipt was noted, and any missing/broken sample containers were noted and reported according to plan. Compare the data package with the COCs to verify that results were provided for all collected samples. Review the narrative to ensure all QC exceptions are described. Check for evidence that any required notifications were provided to project personnel as specified in the QAPP. Verify that necessary signatures and dates are present. Ensure the SEDD file is accepted by FUDSChem and require the laboratory make any applicable changes as needed.	Field Manager/Project Manager/Third party validator (LDC)
Reporting Deliverable	PWS, Corporate QC Plan, QAPP	The document author is responsible for drafting reports of the best quality of their ability.	Author (Field Lead or Field Manager)
		The author presents findings and data at a Challenge Session to be review and discussed with all Credere technical and project management staff so the report content with well vetted.	Credere personnel
		The author then facilitates the deliverable through a three-person review including a review by a technical staff, the Project Manager, Program Manager, and a final QC Manager review.	Technical Lead, Project Manager, Program Manager, and QC Manager

WORKSHEET #36 - DATA VALIDATION PROCEDURES

Data Validator: LDC of Carlsbad, California

Analytical Group:	All
Data deliverable requirement:	SEDD Stage 2a
Analytical specifications:	See worksheets #23 and #28
Measurement performance criteria:	See worksheet #12
Percent of data packages to be validated:	100%
Percent of raw data reviewed:	100%
Percent of results to be recalculated:	0%
Validation procedures:	QAPP, DoD Quality System Manual (QSM), EPA National Functional Guidelines (NFG)
Validation code:	S2aVEM
Electronic validation program/version:	ADR.net Version 1.9.0.330

Based on evaluation results, qualifiers will be added to reported analyte concentrations to indicate uncertainty, potential bias, or interferences. Specific data qualifiers which will be applied to sample concentration include the following:

- U - The analyte was not detected above the reporting limit (RL) or is considered not detected (i.e., false positive) due to blank contamination.
- J - The analyte was detected above the RL but the reported concentration is approximate and is considered estimated. (J+: associated numerical value indicates a positive bias, J-: associated numerical value indicates a negative bias).
- UJ - The analyte was not detected above the RL. However, due to quality control results that did not meet acceptance criteria, the RL is uncertain and may not accurately represent the actual limit.
- NJ – The analyte has been "tentatively identified" and is an estimated value.
- R - The reported analyte is rejected due to serious deficiencies with associated quality control results. The presence or absence of the analyte cannot be confirmed.
- EB, TB or BB – Analyte detected in equipment blank (EB), trip blank (TB) or bottle blank (BB).

WORKSHEET #37 – DATA USABILITY ASSESSMENT

Personnel: Field Manager, Project Manager, Program Manager, and QC Manager
Usability Documentation: Usability will be incorporated into an appropriate section of the RI

The following table summarizes the steps to Credere's Data Usability Assessment (DUA):

Step 1	Review field program and results relative to the projects objectives and sample design
Step 2	Review analytical results relative to expectations and the existing CSM
Step 3	After preparation of tabulated data, figures, and other presentation materials. Review data verification and data validation to assess impacts to trends and results. Are field or laboratory non-compliances affecting results?
Step 4	Review validation qualifiers to assess need for assumptions or additional data.
Step 5	Assess if the data can be used as intended or if limitations are significant to have an effect on conclusions to be drawn from the data.

Data will be reviewed according to the above as a preliminary step in preparing to complete the HHRA/ERA to ensure adequate data quality for use in quantifying risk according to the methodology in **Appendix F**.

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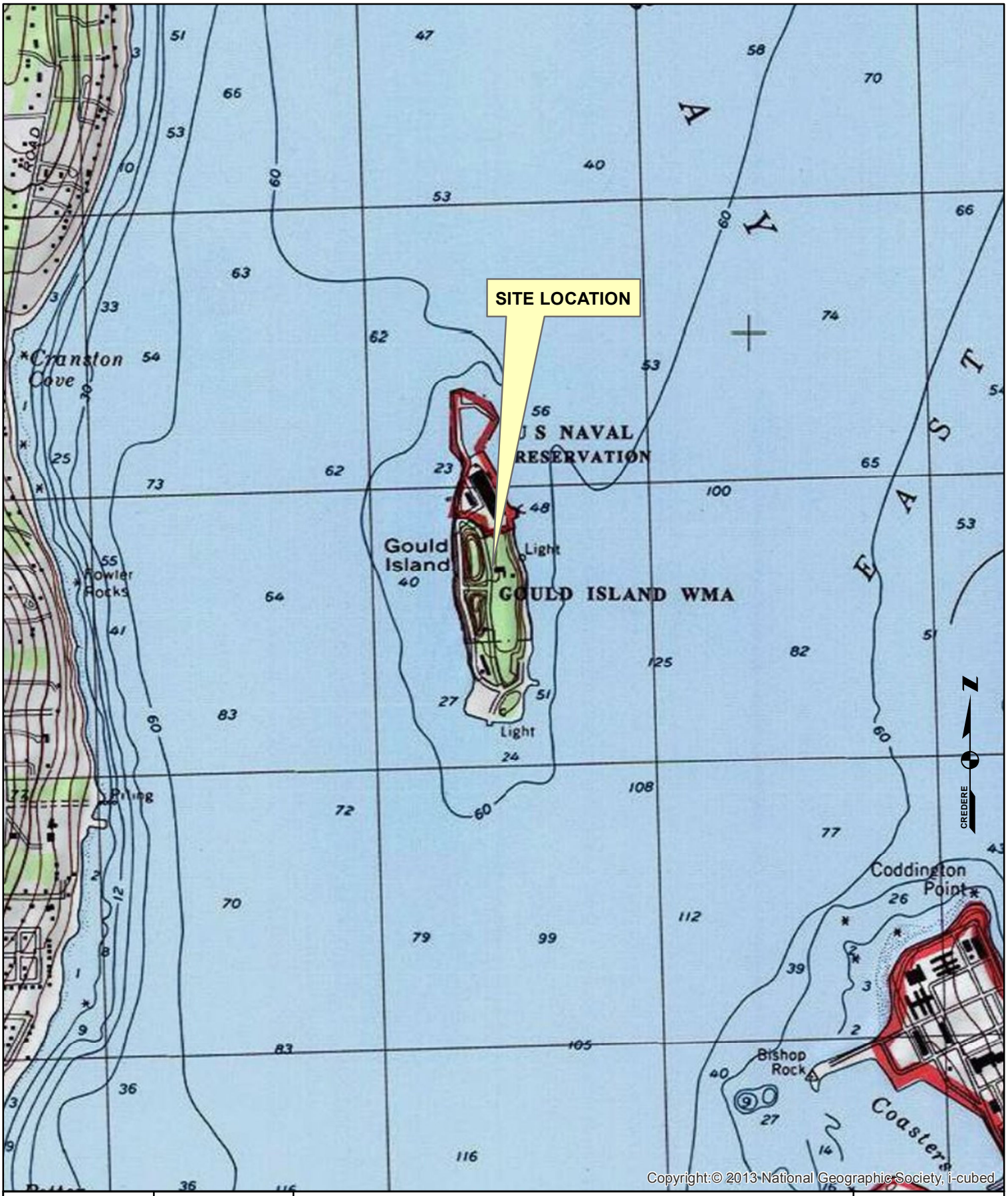
FIGURES



DRAWN BY: **MAK**	DATE: **7/13/2017**
CHECKED BY: **SWM**	PROJECT: **16001327**

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FIGURE 1
SITE LOCATION PLAN

GOULD ISLAND
NARRAGANSETT BAY
JAMESTOWN, RHODE ISLAND

1,000 0 2,000 Feet

1 INCH = 2,000 FEET

T:\Data\NH\Town\Franklin\14001247-Ferrari Mill

LEGEND

NED SITE NUMBER	LOCATION	NED SITE NAME
1	D-4	COAL STORAGE
2	B-5	MARINE BARRACKS (FOUNDATION REMAINING)
3	C-5	FORMER CARPENTERS SHOP
4	E-9	PUMP HOUSE
5	D-7	FORMER UNKNOWN BUILDING #2
6	E-10	UNKNOWN BUILDING #3
7	D-5	FORMER UNKNOWN BUILDING #4 (FOOTINGS REMAINING)
8	E-7	INCINERATOR
9	D-6	MAGAZINE IGNITOR STORAGE
10	E-8	DISPOSAL AREA #14
11	C-6	FORMER QUONSET HUTS/MAINTENACE SHOPS
12	C-7	MAINTENANCE SHOPS/GARAGE/FIRE STATION
13	B-8	ELECTRIC SUBSTATION TRANSFOMER PEN
14	C-8	FORMER MARINE BARRACKS
15	B-9	FORMER RECREATION BUILDING
16	C-9	FORMER BARRACKS
17	C-9	FORMER TRANSFORMER VAULT
18	D-10	WATER TREATMENT PLANT
19	C-9	TORCH POT STORAGE
20	B-6	FORMER CABLE TERMINAL BUILDING
21	B-11	BUNKER #11
22	B-12	BUNKER #12
23	C-12	COAL STORAGE
24	B-12	CABLE TERMINAL BUILDING
25	B-12	UNDERGROUND STORAGE TANKS

NED SITE NUMBER	LOCATION	NED SITE NAME
26	C-13	GAS PUMP HOUSE/GAS TANKS (REMOVED)
27	C-13	TWO GAS PITS
28	C-13	FORMER ORDNANCE TEST FACILITY, HANGER
29	D-13	FORMER UNKNOWN BUILDING #1 (HANGAR 5496-61)
30	E-13	FORMER PYROTECHNIC STORAGE
31	E-13	GAS PIT
32	E-12	FORMER DRUM STORAGE AREA
33	D-12	FORMER PAINT AND OIL STORAGE
34	D-12	THEATER/RESEARCH BUILDING (FOUNDATION REMAINING, CONCRETE PLATFORM)
35	E-12	BOILER HOUSE
36	D-11	DEGAUSSING BUILDING
37	D-11	MISCELLANEOUS STORAGE
38	D-11/12	STORAGE
39	D-6	WELLHOUSE #81
40	C-9	WELLHOUSE #78
41	D-8	AA GUNS ELECTRICAL SUPPLY (UTILITY POLE REMAINING)
42	C-13	5,000 GALLON AVGAS TANK
43	D-13	FORMER PAINT SHED
44	C-14	EMPTY DRUMS
45	D-8	FORMER FIRE APPARATUS HOUSES (2)
46	D-7, D-8, E-7, E-8	FORMER AA GUN EMPLACEMENTS (4)
47	C-10	UNDERGROUND WATER TANK
48	D-6	DEBRIS STOCKPILE
N/A	C-13	FORMER BACKGROUND SAMPLE #1 LOCATION (BG-48SS-001)
N/A	D-7	FORMER BACKGROUND SAMPLE #2 LOCATION (BG-48SS-002)

CREDERE

250 125 0 250 Feet

1 INCH = 120 FEET

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FIGURE 2
DETAILED SITE PLAN

GOULD ISLAND
NARRAGANSETT BAY
JAMESTOWN, RHODE ISLAND

- 10 NED SITE NUMBER
- PREVIOUS SEDIMENT SAMPLE LOCATION (STONE AND WEBSTER, 1997)
- PREVIOUS SOIL SAMPLE LOCATION (STONE AND WEBSTER, 1997)
- HISTORICAL EDGE OF ROADWAY
- HISTORICAL UNPAVED ROAD
- HISTORICAL RAIL LINE
- 5 FOOT TOPOGRAPHIC CONTOUR LINE
- US NAVY OR COAST GUARD EASEMENTS / NON-FUDS ELIGIBLE
- HISTORICAL STRUCTURES
- DISPOSAL AREA
- APPROXIMATE SITE BOUNDARY
- APPROXIMATE PARCEL BOUNDARY

NOTES:
EXISTING CONDITIONS FEATURES SHOWN ON THIS PLAN ARE APPROXIMATE AND ARE BASED ON INFORMATION OBTAINED FROM 2014 USDA NAIP DIGITAL TRUE COLOR ORTHOPHOTOGRAPHY, US COAST GUARD, AND ENGINEERING EVALUATION OF CONTAMINATION FORMER NAVY TORPEDO TESTING FACILITY BY STONE & WEBSTER ENVIRONMENTAL TECHNOLOGY & SERVICES.

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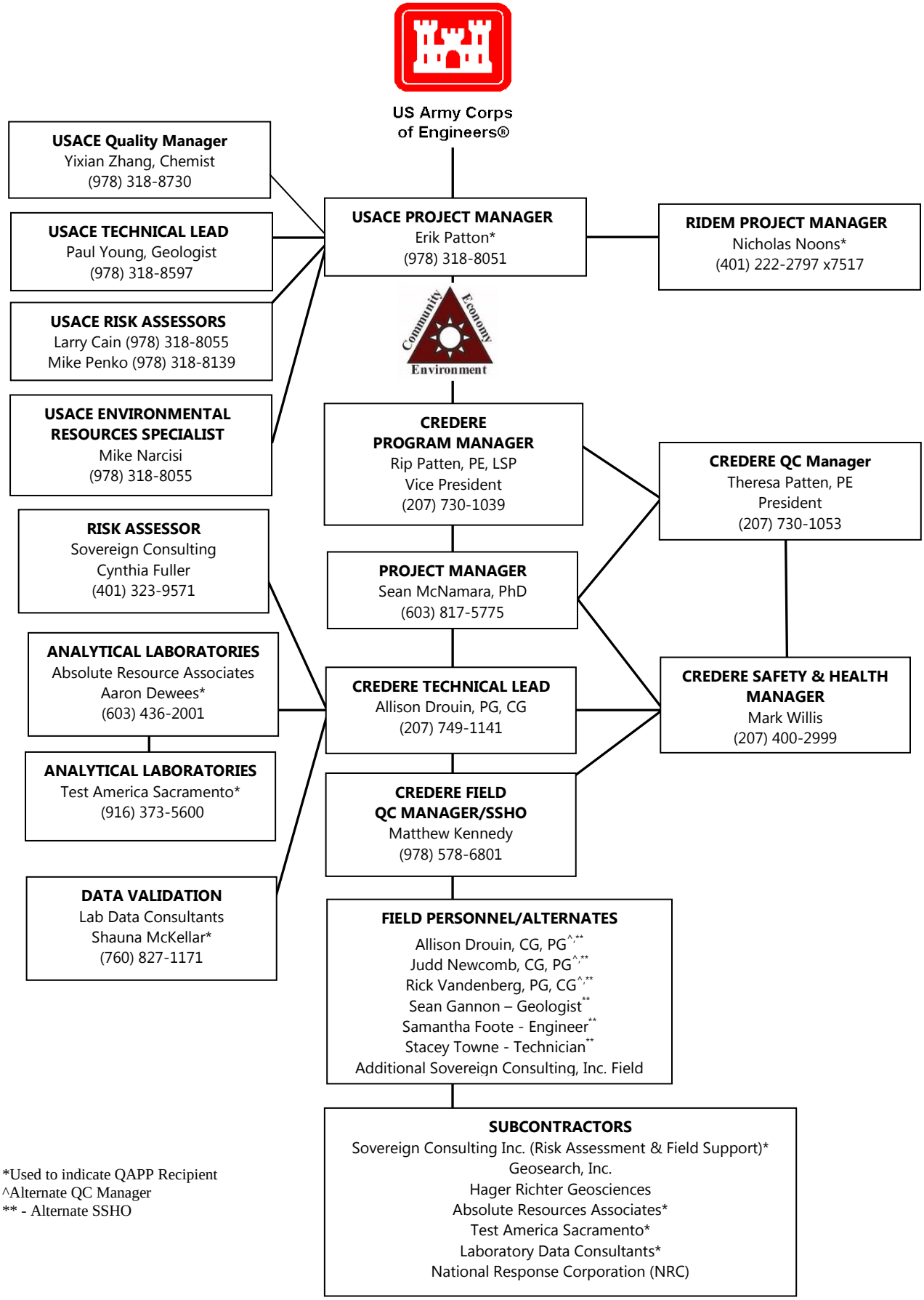
Figure 3 – Project Organization Flow Chart



*Used to indicate QAPP Recipient
^Alternate QC Manager
** - Alternate SSHO

AREA OWNED BY US NAVY

KEEPER'S HOUSE
BG-4
BG-7
BG-6
13CC-1 - 13CC-4
WELLHOUSE
BG-5
BG-8
13SS-3
13SS-6
13
13SS-4
13SS-001
13SS-002
13SS-5
PARTIALLY BURIED TRANSFORMERS
20
BG-3
BRMW-4
11SS-1 - 11SS-3 FROM 3 ALIQUOTS IN 11DU1
POSSIBLE UST VENT PIPE/ PRESUMED LOCATION OF UST, IF PRESENT
16T-1 THROUGH 16T-3 AND 16SS1- THROUGH 16SS-16 AREA
2
BG-2
BG-1
SPILLED TRANSFORMER OIL
FORMER COAL SHED
15
TENNIS COURT
3
11DU1
11
12SB2/ 12OBMW2
12SB3
12SB4
12
14
12SS-1 THROUGH 12SS-9 AND 12CC-1
16
17
12SB1/ 12OBMW1
DRUM
MAINTENANCE PIT (12W-1 THROUGH 12W-3)
40SS-001
40
47
01DU1
01DU3
39 (39CC-1 AND 39SS-1 THROUGH 39SS-4)
UST REMOVED
MAIN ROAD
PUMPHOUSE
01SB2
39SS-001
WATER TOWER (TOPPLED)
BUNKER
19DU1
18
01SB1
1
01SB3
01SB7
01SB10
PUMPHOUSE
FORMER BACKGROUND (48SS-002)
19
19SS-001
EXISTING MONITORING WELLS
01SB6
1
01SS-001
01SB9
48SB2
46
QUONSET HUT
QUONSET HUT
01SB4
7
01SB5
01SB8
48
NORTH HILL
48SB1
45
41
45
41SS-001
48DU1
5
FOR PROPOSED SAMPLE LOCATIONS AND DETAIL OF INSET SEE FIGURE 4B
6
9
01DU2
46
46
GI-DA-SS-02-01
GI-DA-SS-02-02
WEST ROAD
BRMW-5
8
GI-DA-SS-02-03
4
10SS-002
10SS-001
10
GI-DA-SB-04-01
SMALL BOAT LANDING (DETERIOTATED)
GI-DA-SD-02-02
GI-DA-SD-02-01
S-5
S-4
S-1
S-2
S-3
S-6
S-7
S-8
S-9
S-10

CREDERE

240 120 0 240 Feet

1 INCH = 120 FEET

LEGEND

NED SITE NUMBER	NED SITE NAME
1	COAL STORAGE
2	MARINE BARRACKS (FOUNDATION REMAINING)
3	FORMER CARPENTERS SHOP
4	PUMP HOUSE
5	FORMER UNKNOWN BUILDING #2
6	UNKNOWN BUILDING #3
7	FORMER UNKNOWN BUILDING #4 (FOOTINGS REMAINING)
8	INCINERATOR
9	MAGAZINE IGNITOR STORAGE
10	DISPOSAL AREA #14
11	FORMER QUONSET HUTS/MAINTENACE SHOPS
12	MAINTENANCE SHOPS/GARAGE/FIRE STATION
13	ELECTRIC SUBSTATION TRANSFOMER PEN
14	FORMER MARINE BARRACKS
15	FORMER RECREATION BUILDING
16	FORMER BARRACKS
17	FORMER TRANSFORMER VAULT
18	WATER TREATMENT PLANT
19	TORCH POT STORAGE
20	FORMER CABLE TERMINAL BUILDING
21	BUNKER #11
22	BUNKER #12
23	COAL STORAGE
24	CABLE TERMINAL BUILDING
25	UNDERGROUND STORAGE TANKS
26	GAS PUMP HOUSE/GAS TANKS (REMOVED)
27	TWO GAS PITS
28	FORMER ORDNANCE TEST FACILITY, HANGER
29	FORMER UNKNOWN BUILDING #1 (HANGAR 5496-61)
30	FORMER PYROTECHNIC STORAGE
31	GAS PIT
32	FORMER DRUM STORAGE AREA
33	FORMER PAINT AND OIL STORAGE
34	THEATER/RESEARCH BUILDING (FOUNDATION REMAINING, CONCRETE PLATFORM)
35	BOILER HOUSE
36	DEGAUSSING BUILDING
37	MISCELLANEOUS STORAGE
38	STORAGE
39	WELLHOUSE #81
40	WELLHOUSE #78
41	AA GUNS ELECTRICAL SUPPLY (UTILITY POLE REMAINING)
42	5,000 GALLON AVGAS TANK
43	FORMER PAINT SHED
44	EMPTY DRUMS
45	FORMER FIRE APPARATUS HOUSES (2)
46	FORMER AA GUN EMPLACEMENTS (4)
47	UNDERGROUND WATER TANK
48	DEBRIS STOCKPILE
N/A	FORMER BACKGROUND SAMPLE #1 LOCATION (BG-48SS-001)
N/A	FORMER BACKGROUND SAMPLE #2 LOCATION (BG-48SS-002)

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FIGURE 4A
SAMPLE LOCATION PLAN
NORTHERN AREAS OF CONCERN

GOULD ISLAND
NARRAGANSETT BAY
JAMESTOWN, RHODE ISLAND

- PROPOSED SOIL BORING/OVERBURDEN MONITORING WELL LOCATION
- PROPOSED SURFACE SOIL SAMPLE LOCATION
- PROPOSED BEDROCK MONITORING WELL LOCATION
- PROPOSED SOIL BORING SAMPLE LOCATION
- EXISTING MONITORING WELL
- PREVIOUS SEDIMENT SAMPLE LOCATION (STONE AND WEBSTER, 1997)
- PREVIOUS SOIL SAMPLE LOCATION (STONE AND WEBSTER, 1997)
- 5 FOOT TOPOGRAPHIC CONTOUR LINE
- DU BOUNDARY
- HISTORICAL EDGE OF ROADWAY
- HISTORICAL RAIL LINE
- SAMPLING BOUNDARY
- HISTORICAL UNPAVED ROAD
- (10) NED SITE NUMBER
- US NAVY OR COAST GUARD EASEMENTS / NON-FUDS ELIGIBLE
- HISTORICAL STRUCTURES
- DISPOSAL AREA
- APPROXIMATE SITE BOUNDARY
- APPROXIMATE PARCEL BOUNDARY

NOTES:
EXISTING CONDITIONS FEATURES SHOWN ON THIS PLAN ARE APPROXIMATE AND ARE BASED ON INFORMATION OBTAINED FROM 2014 USDA NAIP DIGITAL TRUE COLOR ORTHOPHOTOGRAPHY, US COAST GUARD, AND ENGINEERING EVALUATION OF CONTAMINATION FORMER NAVY TORPEDO TESTING FACILITY BY STONE & WEBSTER ENVIRONMENTAL TECHNOLOGY & SERVICES.

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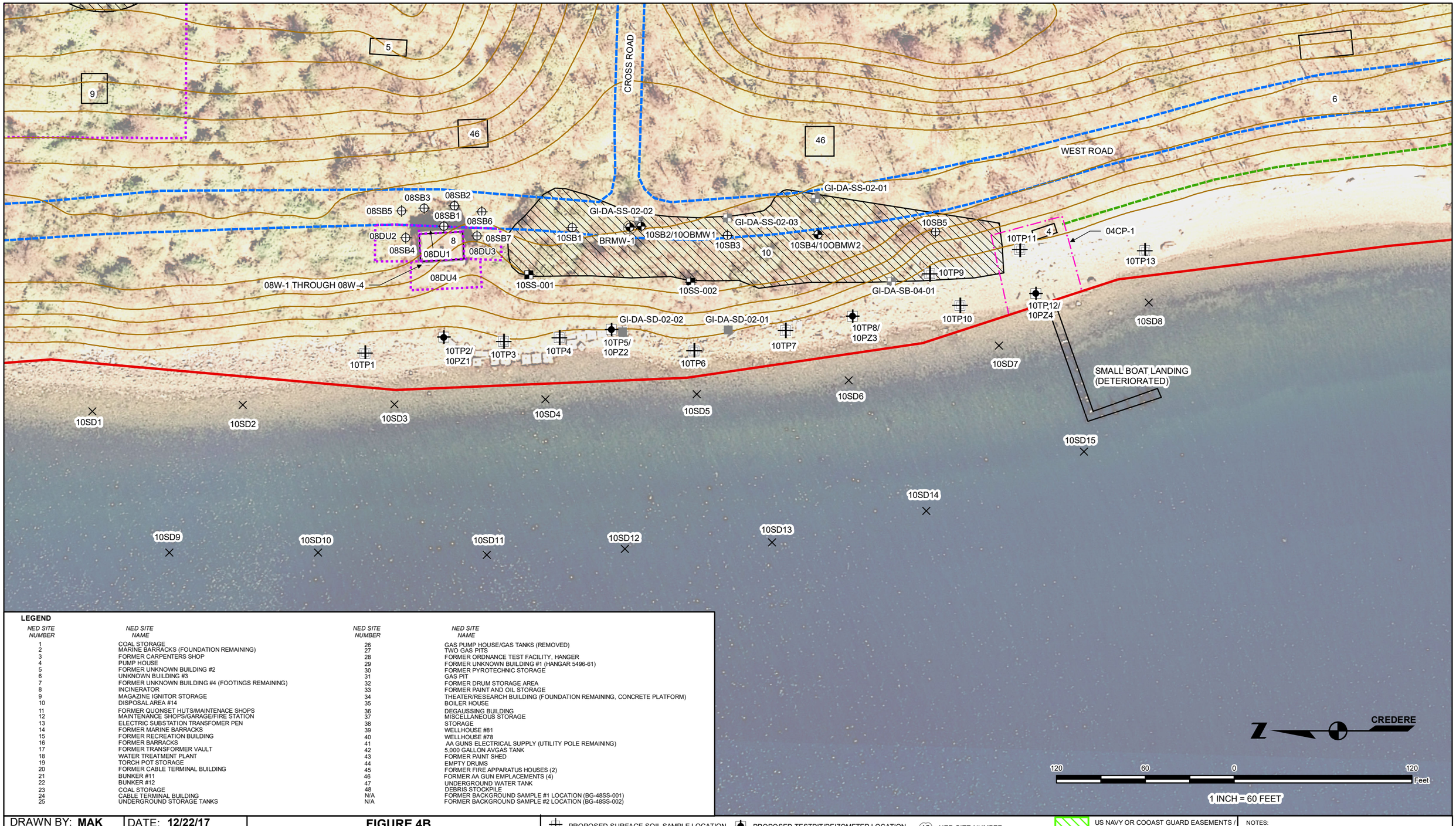

LEGEND

NED SITE NUMBER	*NED SITE NAME*	*NED SITE NUMBER*	*NED SITE NAME*
1	COAL STORAGE	26	GAS PUMP HOUSE/GAS TANKS (REMOVED)
2	MARINE BARRACKS (FOUNDATION REMAINING)	27	TWO GAS PITS
3	FORMER CARPENTERS SHOP	28	FORMER ORDNANCE TEST FACILITY, HANGER
4	PUMP HOUSE	29	FORMER UNKNOWN BUILDING #1 (HANGAR 5496-61)
5	FORMER UNKNOWN BUILDING #2	30	FORMER PYROTECHNIC STORAGE
6	UNKNOWN BUILDING #3	31	GAS PIT
7	FORMER UNKNOWN BUILDING #4 (FOOTINGS REMAINING)	32	FORMER DRUM STORAGE AREA
8	INCINERATOR	33	FORMER PAINT AND OIL STORAGE
9	MAGAZINE IGNITOR STORAGE	34	THEATER/RESEARCH BUILDING (FOUNDATION REMAINING, CONCRETE PLATFORM)
10	DISPOSAL AREA #14	35	BOILER HOUSE
11	FORMER QUONSET HUTS/MAINTENACE SHOPS	36	DEGAUSSING BUILDING
12	MAINTENANCE SHOPS/GARAGE/FIRE STATION	37	MISCELLANEOUS STORAGE
13	ELECTRIC SUBSTATION TRANSFOMER PEN	38	STORAGE
14	FORMER MARINE BARRACKS	39	WELLHOUSE #81
15	FORMER RECREATION BUILDING	40	WELLHOUSE #78
16	FORMER BARRACKS	41	AA GUNS ELECTRICAL SUPPLY (UTILITY POLE REMAINING)
17	FORMER TRANSFORMER VAULT	42	5,000 GALLON AVGAS TANK
18	WATER TREATMENT PLANT	43	FORMER PAINT SHED
19	TORCH POT STORAGE	44	EMPTY DRUMS
20	FORMER CABLE TERMINAL BUILDING	45	FORMER FIRE APPARATUS HOUSES (2)
21	BUNKER #11	46	FORMER AA GUN EMPLACEMENTS (4)
22	BUNKER #12	47	UNDERGROUND WATER TANK
23	COAL STORAGE	48	DEBRIS STOCKPILE
24	CABLE TERMINAL BUILDING	N/A	FORMER BACKGROUND SAMPLE #1 LOCATION (BG-48SS-001)
25	UNDERGROUND STORAGE TANKS	N/A	FORMER BACKGROUND SAMPLE #2 LOCATION (BG-48SS-002)

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FIGURE 4B
SAMPLE LOCATION PLAN
WESTERN AREAS OF CONCERN

GOULD ISLAND
NARRAGANSETT BAY
JAMESTOWN, RHODE ISLAND

- PROPOSED SURFACE SOIL SAMPLE LOCATION
- PROPOSED SOIL BORING SAMPLE LOCATION
- PROPOSED SOIL BORING/OVERBURDEN MONITORING WELL LOCATION
- PROPOSED BEDROCK MONITORING WELL LOCATION
- PREVIOUS SOIL SAMPLE LOCATION (STONE AND WEBSETER, 1997)
- PREVIOUS SEDIMENT SAMPLE LOCATION (STONE AND WEBSTER, 1997)
- PROPOSED TESTPIT/PEIZOMETER LOCATION
- PROPOSED SEDIMENT SAMPLE LOCATION
- PROPOSED TESTPIT LOCATION
- (10) NED SITE NUMBER
- 5 FOOT TOPOGRAPHIC CONTOUR LINE
- HISTORICAL EDGE OF ROADWAY
- HISTORICAL RAIL LINE
- SAMPLING BOUNDARY
- HISTORICAL UNPAVED ROAD
- DU BOUNDARY
- US NAVY OR COOAST GUARD EASEMENTS / NON-FUDS ELIGIBLE
- HISTORICAL STRUCTURES
- DISPOSAL AREA 14
- APPROXIMATE SITE BOUNDARY
- APPROXIMATE PARCEL BOUNDARY

NOTES:
1. EXISTING CONDITIONS FEATURES SHOWN ON THIS PLAN ARE APPROXIMATE AND ARE BASED ON INFORMATION OBTAINED FROM 2014 USDA NAIP DIGITAL TRUE COLOR ORTHOPHOTOGRAPHY, US COAST GUARD, AND ENGINEERING EVALUATION OF CONTAMINATION FORMER NAVY TORPEDO TESTING FACILITY BY STONE & WEBSTER ENVIRONMENTAL TECHNOLOGY & SERVICES.
2. SEE FIGURE 2 FOR FULL VIEW OF THE SITE.
3. PREVIOUS SEDIMENT SAMPLE LOCATIONS FROM THE 1984 AND 1985 INVESTIGATIONS BY LOUREIRO ENGINEERING ASSOCIATES ARE NOT SHOWN ON THIS FIGURE. SEE FIGURE 2 FOR THE APPROXIMATE LOCATION OF THESE SAMPLES.

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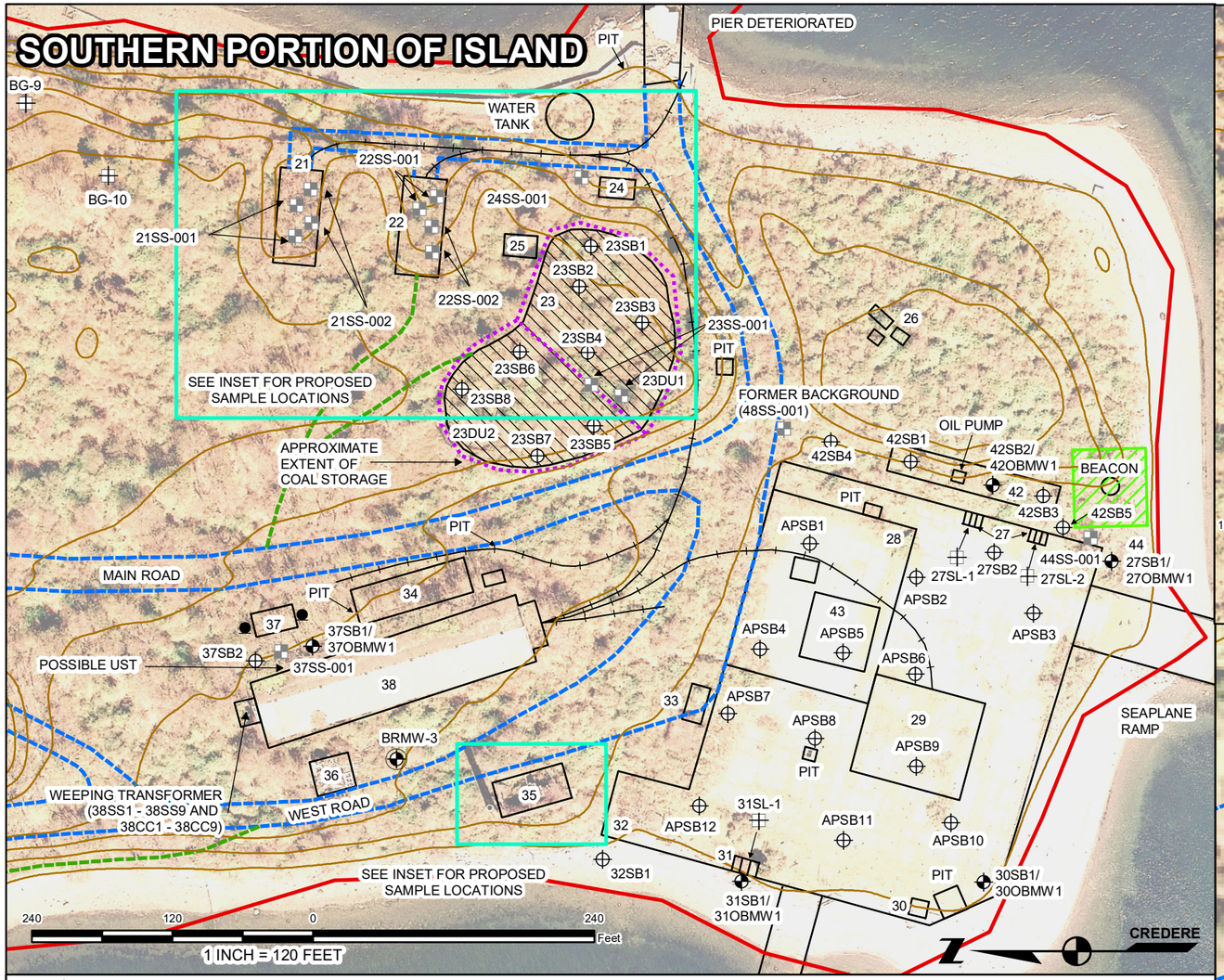

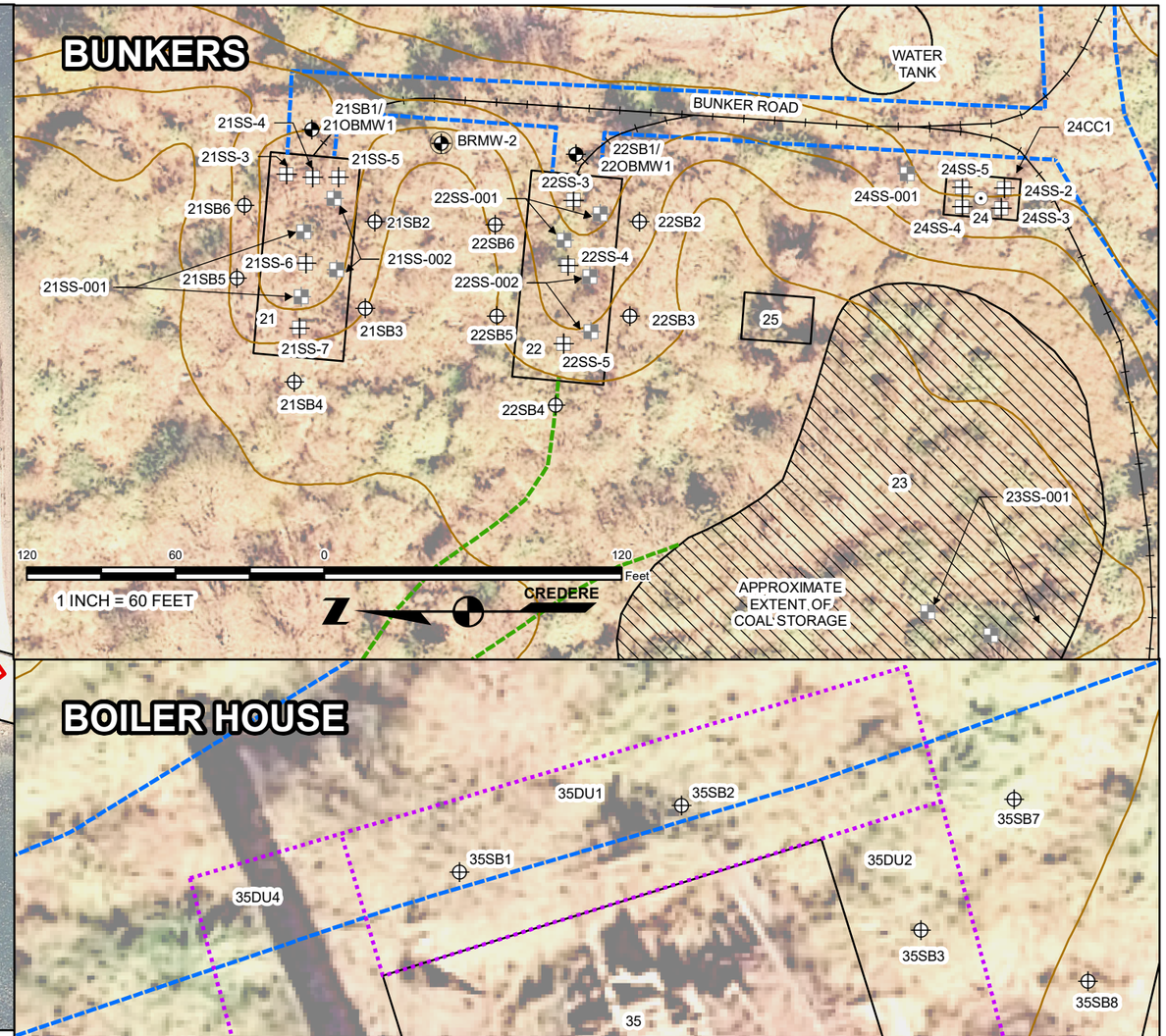

LEGEND

NED SITE NUMBER	NED SITE NAME	NED SITE NUMBER	NED SITE NAME
1	COAL STORAGE	26	GAS PUMP HOUSE/GAS TANKS (REMOVED)
2	MARINE BARRACKS (FOUNDATION REMAINING)	27	TWO GAS PITS
3	FORMER CARPENTERS SHOP	28	FORMER ORDNANCE TEST FACILITY, HANGER
4	PUMP HOUSE	29	FORMER UNKNOWN BUILDING #1 (HANGAR 5496-61)
5	FORMER UNKNOWN BUILDING #2	30	FORMER PYROTECHNIC STORAGE
6	UNKNOWN BUILDING #3	31	GAS PIT
7	FORMER UNKNOWN BUILDING #4 (FOOTINGS REMAINING)	32	FORMER DRUM STORAGE AREA
8	INCINERATOR	33	FORMER PAINT AND OIL STORAGE
9	MAGAZINE IGNITOR STORAGE	34	THEATER/RESEARCH BUILDING (FOUNDATION REMAINING, CONCRETE PLATFORM)
10	DISPOSAL AREA #14	35	BOILER HOUSE
11	FORMER QUONSET HUTS/MAINTENACE SHOPS	36	DEGAUSSING BUILDING
12	MAINTENANCE SHOPS/GARAGE/FIRE STATION	37	MISCELLANEOUS STORAGE
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FIGURE 4C
SAMPLE LOCATION PLAN
SOUTHERN AREAS OF CONCERN

GOULD ISLAND
NARRAGANSETT BAY
JAMESTOWN, RHODE ISLAND

- PROPOSED SURFACE SOIL SAMPLE LOCATION
- PREVIOUS SOIL SAMPLE LOCATION (STONE AND WEBSTER, 1997)
- PROPOSED SOIL BORING SAMPLE LOCATION
- PROPOSED SOIL BORING/OVERBURDEN MONITORING WELL LOCATION
- PROPOSED BEDROCK MONITORING WELL LOCATION
- PREVIOUS SEDIMENT SAMPLE LOCATION (STONE AND WEBSTER, 1997)
- PROPOSED CONCRETE SAMPLE LOCATION
- 5 FOOT TOPOGRAPHIC CONTOUR LINE
- DU BOUNDARY
- HISTORICAL EDGE OF ROADWAY
- HISTORICAL RAIL LINE
- HISTORICAL UNPAVED ROAD
- NED SITE NUMBER
- ABOVEGROUND STORAGE TANK
- US NAVY OR COAST GUARD EASEMENTS / NON-FUDS ELIGIBLE
- HISTORICAL STRUCTURES
- DISPOSAL AREA
- APPROXIMATE SITE BOUNDARY
- APPROXIMATE PARCEL BOUNDARY

NOTES:
1. EXISTING CONDITIONS FEATURES SHOWN ON THIS PLAN ARE APPROXIMATE AND ARE BASED ON INFORMATION OBTAINED FROM 2014 USDA NAIP DIGITAL TRUE COLOR ORTHOPHOTOGRAPHY, US COAST GUARD, AND ENGINEERING EVALUATION OF CONTAMINATION FORMER NAVY TORPEDO TESTING FACILITY BY STONE & WEBSTER ENVIRONMENTAL TECHNOLOGY & SERVICES.
2. SEE FIGURE 2 FOR FULL VIEW OF THE SITE.
3. PREVIOUS SEDIMENT SAMPLE LOCATIONS FROM THE 1984 AND 1985 INVESTIGATIONS BY LOUREIRO ENGINEERING ASSOCIATES ARE NOT SHOWN ON THIS FIGURE. SEE FIGURE 2 FOR THE APPROXIMATE LOCATION OF THESE SAMPLES.

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APPENDIX A

FIELD SAMPLING PLAN

Remedial Investigation Field Sampling Plan

Version: Final

Gould Island Site (D01RI033800)
Narragansett Bay
Jamestown, Rhode Island

Prepared for:
U.S. Army Corps of Engineers
New England District
696 Virginia Road
Concord, MA 01742

US Army Corps of Engineers®

April 16, 2018

Contract W912WJ-16-D-007
Task Order W912WJ-17-F-0039

Prepared by:
Credere Associates, LLC
776 Main Street
Westbrook, Maine 04092

TABLE OF CONTENTS

FIGURES

TABLE

APPENDICES

LIST OF ACRONYMS

AOC – area of concern
APP – Accident Prevention Plan
ARA – Absolute Resource Associates
Alion – Alion Science and Technology
bgs – below ground surface
BRMW – bedrock monitoring well
CENAE – Corps of Engineers New England District
CG – Certified Geologist
COC – chain of custody
COPC – contaminant of potential concern
Credere – Credere Associates, LLC
CSM – conceptual site model
DERP – Defense Environmental Restoration Program
DI - deionized
DNAPL – dense non-aqueous phase liquid
DO – dissolved oxygen
DU – decision unit
EIT – Engineer in Training
EPA – U.S. Environmental Protection Agency
EPH – extractable petroleum hydrocarbons
FSP – Field Sampling Plan
FUDS – Formerly Used Defense Sites
FUDSCHEM – Formerly Used Defense Sites Online Chemical Database
GPS – global positioning system
LDC – Laboratory Data Consultants
LNAPL – light non-aqueous phase liquid
LSP – Licensed Site Professional
MEC – munitions and explosives of concern
MPS – multiparameter sonde
NED – New England District
NETC – Naval Education and Training Center
NTU – nephelometric turbidity units
OBMW – overburden monitoring well
ORP – oxidation reduction potential
PAH – polycyclic aromatic hydrocarbon
PCB – polychlorinated biphenyls
PE – Professional Engineer
PG – Professional Geologist
PID – photoionization detector
PPE – personal protective equipment
PP – priority pollutant
ppm_v – parts per million by volume
PWS – Performance Work Statement
PZ – piezometer
QA – quality assurance
QAPP – Quality Assurance Project Plan

QC – quality control
RAB – Restoration Advisory Board
RI – Remedial Investigation
RIDEM – Rhode Island Department of Environmental Management
RTC – response to comments
SOP – standard operating procedure
Sovereign – Sovereign Consulting, Inc.
SSHO – Site Safety and Health Officer
SSHP – Site Safety and Health Plan
SPT – standard penetration test
SVOC – semi-volatile organic compound
USACE – U.S. Army Corps of Engineers
UST – underground storage tank
VOC – volatile organic compound
VPH – volatile petroleum hydrocarbons
YSI – Yellow Springs Instrument

1. INTRODUCTION

Credere Associates, LLC (Credere) was retained by the U.S. Army Corps of Engineers (USACE) New England District (CENAE) to prepare this Field Sampling Plan (FSP) for the Remedial Investigation (RI) at the Gould Island Site located in Narragansett Bay in Jamestown, Newport County, Rhode Island (Site). A Site Location Plan is provided as **Figure 1** and a Detailed Site Plan is provided as **Figure 2**. This FSP has been prepared to serve as a field sampling guide to be used in concert with the associated Quality Assurance Project Plan (QAPP). The scope in this FSP was developed in accordance with the Performance Work Statement (PWS) prepared by CENAE dated June 13, 2017 (CENAE, 2017).

1.1 PROJECT DESCRIPTION

The project includes assessment of potential source areas across the Formerly Used Defense Site (FUDS) eligible portion of the island. The potential source areas were identified based on review of prior environmental reports, munitions response reports, and historical information. Generally, historical data is limited and this assessment will be used to support a Human Health and Ecological Risk Assessment (HHRA/ERA) to be completed as part of the RI.

1.2 SITE DESCRIPTION

The 39.15-acre Site is the central and southern portion of Gould Island that has been assessed under the Defense Environmental Restoration Program (DERP) and is eligible for funding as a FUDS. The Site is currently owned by the Rhode Island Department of Environmental Management (RIDEM) Division of Fish & Wildlife and is designated as a bird sanctuary. The island is inaccessible during bird nesting between April 1st and August 15th. The 16.9-acre remainder of the island to the north is owned by the U.S. Navy. Additionally, within the FUDS Site boundary, four areas are retained by the Navy or Coast Guard, including the Keeper's House and lighthouse, water treatment plant #42 (New England District [NED] Site No. 18), the water tank #31 (NED Site No. 47), the transformer vault #25A (NED Site No. 17), and the 100-foot x 100-foot area around the beacon at the southern end of the island. These areas are not FUDS eligible and are not considered part of the Site.

The Site is currently heavily overgrown with remnants of dilapidated buildings, relict foundations, and bunkers that were formerly used in the storage, testing, and repair of torpedoes, research, housing, and infrastructure (e.g., power, heating, waste disposal). The southernmost point of the island has a large concrete former aviation pad and two dilapidated seaplane ramps (Alion Science and Technology [Alion], 2009). The Site is reportedly accessible from these ramps with use of a ramp barge or shallow landing craft.

1.3 PHYSICAL SETTING

The Site is densely vegetated with young trees and abundant vines and shrub. Substantial clearing is required prior to implementation of the RI. Prior aerial photographs and surveyed

topography indicate large portions of the western side of the island were built up with steeply sloping areas down to the historical roads.

Generally, overburden at the Site was previously described to be sand with varying amounts of silt and gravel. Overburden was reported to be thin along the westernmost side of the island, particularly in areas of bedrock outcropping, and as thick as 10 feet elsewhere on the eastern side of the island. Investigation was limited in the central built up portion of the island; therefore, thickness in this area is not yet known. Groundwater recharge is considered minimal due to the island nature with most precipitation discharging to the bay. Overburden groundwater is present only in the thickest areas of overburden on the eastern side of the island and possibly in only perched lenses and in the bedrock in the western portion of the island (SWETS, 1997).

According to drilling reports from prior investigations at the Navy northern portion of the island, bedrock is relatively soft and was easily cored with diamond bit coring equipment and required screens for well construction to avoid cave-in. Water bearing fractures or specific zones were not encountered during drilling of bedrock monitoring wells at the Navy portion of the island; however, bedrock wells were installed with 30 feet of screen 30 feet into bedrock and yielded groundwater.

1.4 SITE HISTORY

Beginning in 1887, a lighthouse was constructed for Narragansett Bay on the eastern side of the island (RIHPHC, 1995), and the island was otherwise used privately for residential purposes. Between 1918 and 1920, the island was seized by the United States for use by the U.S. Navy and Marines. Initial construction included adaptation of the existing residence for marine barracks, air hangars, a water tower and infrastructure, a personnel pier at the northern end of the island, a concrete torpedo pier at the southeast point of the island, a torpedo storage building, two warhead bunkers (later converted to a firing range and storage), the south powerhouse, and a railroad network for moving heavy equipment throughout the island. Torpedoes were test fired from seaplanes staged at the southern end of the Site and from an off-island barge. Test torpedoes were unarmed and used to test guidance and launching techniques only.

In the 1940s, additional buildings were rapidly constructed to support World War II, including additional hangars, a second southern seaplane ramp, torpedo overhaul building, power station, additional barracks, Quonset huts, degaussing buildings, and the firing pier in the northern offsite portion of the island. Warheads were transferred to Prudence Island in 1941 due to safe distance limitations, and a pistol range was constructed at former Bunker 11. By 1943, the firing pier was in operation and approximately 65,000 unarmed torpedoes were tested from the firing pier, barges, and submarines by the end of World War II.

In the 1950s, testing and production of torpedoes was outsourced to private contractors (Envirodyne, 1983), and in 1975 and 1989, parcels were transferred to the State of Rhode Island to form the current 39.15-acre Site. The Site has been generally abandoned since and is heavily overgrown with vegetation. Public access to the Site is by permit only, and the Site has been designated as a bird sanctuary.

Beginning in 1983, the Site was assessed as part of the larger Naval Education and Training Center (NETC). Several areas of concern (AOCs) were identified on the island and numerous drums, containers, and underground storage tanks (USTs) were removed before 1990. Subsequent Site visits in 1997 and later 2009 and current records review identified 48 AOCs (i.e., NED Sites). Limited sampling confirmed several contaminants of potential concern (COPCs), including metals, polycyclic aromatic hydrocarbons (PAHs), nitroglycerin, and petroleum hydrocarbons among other COPCs that cannot be dismissed based on the limited data set. Munitions investigations have previously concluded no ordnance and munitions and explosives of concern (MEC) remains on the island.

1.5 CONTAMINANTS OF POTENTIAL CONCERN

Based on the Site history and previous investigations, the following COPCs are identified for the Site for the indicated media and will be screened for during the investigation:

Soil	Groundwater	Sediment
VOCs SVOCs PP metals Hexavalent chromium EPH/VPH PCBs Explosives Dioxins/furans Pesticides Acids (via pH)	VOCs SVOCs PP metals Hexavalent chromium EPH/VPH PCBs Explosives Pesticides Acids (via pH)	VOCs SVOCs EPH/VPH PCBs Explosives PP metals Hexavalent chromium Pesticides Acids (via pH)

VOCs – volatile organic compounds
SVOCs – semi-volatile organic compounds
PP – priority pollutant
EPH – extractable petroleum hydrocarbons
VPH – volatile petroleum hydrocarbons
PCBs – polychlorinated biphenyl

1.6 NATURE AND EXTENT OF CONTAMINATION

As a limited number of samples were previously collected from some specific NED Sites, the extent of contamination at the Site is not known. The inferred source, specific COPCs for each media, expected extent based on currently available information, and current data gaps are summarized in Appendix B of the QAPP.

1.7 EXPOSURE PATHWAYS & POTENTIAL RECEPTORS

As the Site is to be assessed for the need for remedial actions under an unrestricted use scenario, potential receptors are conservatively assumed to be future residents and recreational users, as well as terrestrial and aquatic biota. Exposure pathways to these receptors based on the current conceptual site model (CSM) summarized in Appendix B of the QAPP, would be through direct contact or incidental uptake of contaminated soil, inhalation of VOCs, or active ingestion through drinking contaminated water.

2. PROJECT TEAM AND CONTACT INFORMATION

The following table summarizes project personnel and project stakeholders contact information for field reference. A Project Organizational Flow Chart is included as **Figure 3**.

Name	Role/Title	Organization	Contact Information
Rip Patten, PE, LSP	Program Manager, Vice President, Engineer	Credere	(207) 730-1039 rpatten@crederellc.com
Theresa Patten, PE	Quality Control (QC) Manager, President, Engineer	Credere	(207) 730-1053 tpatten@crederellc.com
Sean McNamara, Ph.D.	Project Manager, Project Engineer	Credere	(603) 817-5775 smcnamara@crederellc.com
Allison Drouin, PG, CG	Technical Lead, Geologist Alternate Field QC Manager, alternate Site Safety and Health Officer (SSHO)	Credere	(207) 749-1141 adrouin@crederellc.com
Rick Vandenberg, PG, CG	Alternate Field QC Manager, alternate SSHO, Hydrologist	Credere	(603) 312-8824 rickv@crederellc.com
Judd Newcomb, PG, CG	Alternate Field QC Manager, alternate SSHO, Geologist	Credere	(207) 232-5387 jnewcomb@crederellc.com
Mark Willis	Environmental Safety & Health; Industrial Hygienist, Safety & Health Manager (SHM)	Credere	(207) 400-2999 mwillis@crederellc.com
Matthew Kennedy	Field QC Manager, SSHO	Credere	(978) 578-6801 mkennedy@crederellc.com
Sean Gannon	Alternate SSHO, Geologist	Credere	(860) 733-2510 sgannon@crederellc.com
Stacy Towne	Alternate SSHO, Field Technician	Credere	(207) 284-3056 stowne@crederellc.com
Samantha Foote, EIT	Alternate SSHO,, Engineer	Credere	(207)449-2739 sfoote@crederellc.com
Aidan E. Desrosier	Alternate SSHO,, Field Scientist	Sovereign Consulting, Inc. (Sovereign)	(919) 520-3754
Chris Terra	Alternate field staff, Field Geologist	Sovereign	(781) 724-3632
Marissa Bovie	Alternate field staff, Environmental Scientist	Sovereign	(207) 312-1779
Cynthia Fuller	Lead for Risk Assessment	Sovereign	(401) 323-9571 cfuller@sovcon.com
Aaron Dewees	Laboratory QC Officer, Chemist	Absolute Resource Associates (ARA)	(603) 436-2001 aarond@absoluteresourceassociates.com
Shauna McKellar	Data Validator, Chemist	Laboratory Data Consultants (LDC)	smckellar@lab-data.com

CG – Certified Geologist
EIT – Engineer in Training
LSP – Licensed Site Professional
PE – Professional Engineer
PG – Professional Geologist

3. WORK PLAN OBJECTIVES AND SCHEDULE

3.1 STATEMENT OF OBJECTIVES

The primary objective of this FSP/RI is to collect sufficient analytical data to support a human health and ecological risk assessment and assess the need for remedial action. The following Site-specific objectives are established:

- Assess each NED Site requiring further investigation and collect sufficient soil, groundwater, and sediment data to confirm or dismiss the need for remedial action through risk assessment

3.2 SCHEDULE

The following table summarizes the schedule for project activities from completion of review of this FSP and associated QAPP through implementation of field activities by Credere.

Date	Task
October 2017	Submit Draft QAPP/FSP and Accident Prevention Plan (APP)/Site-Specific Safety and Health Plan (SSHP)
November 2017	Receive QAPP/FSP and APP/SSHP comments from CENAE
November 2017	Incorporate comments and submit response to comment (RTC) and Stakeholder-Draft version of QAPP/FSP and Final version of APP/SSHP
November – December 2017	Site clearing and initial site visit
December 2017 – February 2018	Submit QAPP/FSP to RIDEM
February 2018	Incorporate RIDEM comments and submit RTC and Final version of QAPP/FSP
February 2018	Complete electronic QAPP (eQAPP) and event planning in FUDSChem
February - March 2018	Perform RI field work
April 1st – August 15th (RIDEM, 2010)	ISLAND INACCESSIBLE TO HEAVY EQUIPMENT DUE TO BIRD NESTING (low impact hand tools may be allowed)
March –April 2018	Chain of custody reconciliation (within 3 business days of field demobilization) and field record upload to FUDSCHEM within 7 days of demobilization (CENAE, 2016)
May 2018	RI Field Report and Project Team Meeting
July 2018	Submit Draft RI Report
July - August 2018	CENAE review and RTC Period for Draft RI Report
August - October 2018	RIDEM and RAB review and RTC period for Stakeholder-Draft RI Report
November 2018	Final RI Report
Fall 2018	Alternatively, additional remedial investigation activities after August 15th

FUDSCHEM - Formerly Used Defense Sites Online Chemical Database

4. SCOPE OF WORK

Credere has prepared the following field sampling plan to meet the objectives outlined in **Section 3.1** and in accordance with the PWS (CENAE, 2017). A Detailed Site Plan showing pertinent Site features and sample locations is provided as **Figure 2**, and Sample Location Plans are provided as **Figures 4A** through **4C**.

4.1 GEOPHYSICAL AND PRIVATE UTILITY CLEARANCE

As the Site is in the middle of Narragansett Bay, DigSafe will not likely be required to perform utility clearance, but they will be notified nonetheless to allow member utilities to mark the Site. Credere will also contact non-member local utilities of the work. The Navy will also be notified of the work and schedule so as to allow marking of the water line maintained by the Navy.

Geophysics will be used to clear each exploration location (e.g., borings, test pits, and hand sampling locations) for utilities and other possible anomalies (e.g., unexploded ordnance, etc.).

Any identified anomalies will be marked in the field and avoided during sampling activities unless field conditions (e.g., availability of an excavator associated with Site clearing that can penetrate frozen ground during the geophysical mobilization) allow for exploration of anomalies. If suspected MC are identified, an UXO specialist will be contacted to identify the suspect material.

4.2 CONTAINER CONSOLIDATION

During RI activities, any encountered containers containing liquid or solid will be overpacked, if needed, and consolidated by NRC. Where empty containers are encountered, their locations will be marked for further evaluation/sampling. Empty containers will also be consolidated to a single area on the island to be removed and recycled as scrap to reduce the debris present on the island and facilitate future investigation activities. Any solid and liquid waste will be characterized for disposal according to disposal facility requirements and in accordance with RIDEM's Rules and Regulations for Hazardous Waste Management (RIDEM, 2016).

4.3 SOIL BORING ADVANCEMENT, TEST PITTING, & SOIL SAMPLING

Table 1 summarizes the sample design and rationale for soil samples. Sample locations or decision units (DUs) are depicted on **Figures 4A** through **4C**. If proposed locations are not achievable due to field constraints, the field team will communicate these limitations in accordance with the QAPP to assess the need to alter the design.

Incremental sampling DUs are also depicted on **Figures 4A** through **4C**. These units were designated based on review of historical aerial photographs. The number of aliquots per DU per replicate were determined based on the level of likelihood of associated contamination, expected heterogeneity of the unit, and size. The number of aliquots may vary slightly based on the layout

of the grid, particularly from oddly shaped DUs. DU and aliquot design are summarized in **Table 1**.

4.4 MONITORING WELL, PIEZOMETER INSTALLATION, & GROUNDWATER SAMPLING

Table 1 summarizes the groundwater sample design and well location rationale. Well and piezometer locations are depicted on **Figures 4A** through **4C**. If proposed locations are not achievable due to field constraints, the field team will communicate limitations in accordance with the QAPP to assess the need to alter the design. Particularly, if no overburden groundwater is encountered prior to refusal in a planned overburden well location, no overburden well will be installed as the groundwater table will be considered to be within bedrock and will be assessed through the nearest bedrock monitoring well.

Generally, based on the Navy account of Site conditions, overburden groundwater is likely temporarily perched stormwater that infiltrates to a bedrock aquifer. Therefore, overburden wells would be primarily assessing impacts associated with recent stormwater percolating through possibly contaminated source soil so if impacts to bedrock wells are identified, a possible source may be identified.

4.5 SEDIMENT SAMPLING

Table 1 summarizes the sediment sample design and location rationale. Sample locations are depicted on **Figures 4B**. If proposed locations are not achievable due to field constraints, the field team will communicate limitations in accordance with the QAPP to assess the need to alter the design.

4.6 CONCRETE SAMPLING

Table 1 summarizes the concrete sample design and location rationale. Sample locations are depicted on **Figures 4A** through **4C**. The sample design was based on assessment of the extent of a release per 40 CFR 761.265. If proposed locations are not achievable due to field constraints, the field team will communicate limitations in accordance with the QAPP to assess the need to alter the design.

5. METHODOLOGY

Field activities will be completed in accordance with Credere's Standard Operating Procedures (SOPs) for each respective activity. Credere's SOPs consider both US Environmental Protection Agency (EPA) and RIDEM SOPs and are at least as comprehensive as the procedures described therein. Credere field SOPs to be used as part of field activities described herein are summarized in the following table and are included as Appendix E of the QAPP:

SOP #	Title	Date/Revision	Modified for Project
CA-1	Field Activity Documentation	August 2, 2016, revision 1	No
CA-2	Equipment Decontamination Procedures	March 17, 2016, revision 0	No
CA-4	Soil Description	March 17, 2016, revision 0	No
CA-5	Environmental Soil Sampling	May 27, 2016, revision 0	No
CA-6	Test Pitting	March 17, 2016, revision 0	No
CA-7	Headspace Field Screening	May 20, 2016, revision 0	No
CA-8	Monitoring Well Installation	October 20, 2017, revision 1	No
CA-9	Monitoring Well Development	October 20, 2017, revision 1	No
CA-10	Monitoring Well Gauging	August 29, 2016, revision 0	No
CA-11	Water Quality Field Instrument Calibration	September 18, 2017, revision 0	No
CA-12	Low-Flow Groundwater Sampling	October 11, 2017, revision 2	No
CA-13	Surface Water and Sediment Sampling	September 9, 2016, revision 0	No
CA-16	Chain of Custody Procedure	November 11, 2017, revision 0	No
CA-17	Packaging and Shipping Samples	August 22, 2017, revision 0	No
CA-23	Collection of PCB-Containing Building Material and Substrate Samples	October 25, 2017, revision 0	No
CA-24	Bedrock Drilling, Well Installation, and Packer Operation	October 31, 2017, revision 0	No
CA-26	Incremental Sampling Methodology	October 31, 2017, revision 0	No

5.1 GEOPHYSICS AND DIGSAFE UTILITY CLEARANCE

The Site will be pre-marked and DigSafe will be notified of the planned drilling work. Member utilities will be notified by DigSafe to mark utilities. Credere will also contact non-member local utilities including the Jamestown Public Works Department Stormwater Management Division and Water and Sewer Department. The Navy will also be notified of the work to allow marking of the onsite water line currently maintained by the Navy.

The previously surveyed edges of the Disposal Area #14 (NED Site No. 10) will be surveyed to confirm the locations if it is not currently marked and discernable in the field. The edges will be surveyed with a global positioning system (GPS) to allow for more accurate mapping relative to other new Site features.

Boring locations will be cleared in the field using GPR and/or magnetometry in a 20-foot diameter around each location. Hand sample locations will also be cleared per decision unit.

Clearance will assure health and safety with regard to possible buried munitions, despite previous clearance, and for other unknown buried anomalies.

5.2 SOIL BORING ADVANCEMENT, TEST PITTING, & SOIL SAMPLING

Overburden soil borings will be advanced to refusal on the upland part of the island, and through evidence of contamination or to a minimum depth of 12 feet below ground surface (bgs; see Credere SOP CA-5). Soil borings (or test pits if soil borings aren't feasible) along the beach will be conducted at low tide for safety considerations and to best evaluate depth to groundwater. A copy of tide charts for February and March 2018 are included as **Appendix A**. If refusal is encountered, an additional 3 attempts will be made to advance the boring deeper or confirm the refusal depth to ensure refusal on possible bedrock and not other interferences (e.g., large cobbles, boulders, etc.). Overburden borings will be advanced using direct-push drilling methods with a Geoprobe® macrocore sampling device, or equivalent. Soil cores will be collected continuously using dedicated, disposable polyethylene liners. Macrocores will be individually logged according to a modified Burmister and USCS method in accordance with Credere SOP CA-4, evidence of contamination will be noted, and soil will be field screened according to Credere SOP CA-7 for total VOCs using a ppbRAE 3000 (or similar) photoionization detector (PID) calibrated with a 10 parts per million by volume (ppm_v) isobutylene gas standard and an instrument response factor of 1.0.

At bedrock monitoring well locations, soil borings will be advanced with hollow stem augers to bedrock refusal and continuously sampled using a 24-inch split spoon sampler using standard penetration test (SPT) techniques (see Credere SOP CA-5). Soil will be field screened per above; however, as bedrock wells are located on the Site perimeter or coupled with overburden wells in source areas, soil samples will not be collected from bedrock well soil borings. In the case of well couplets (BRMW-1 and 10OBMW-1), the bedrock boring/well will be completed first.

At test pit soil sample locations, soil will be removed from the test pits to refusal or to a maximum depth of 10 feet bgs and stockpiled adjacent to the test pit according to Credere SOP CA-6. A Credere geologist will log the changes in strata, observed contamination, and depth to groundwater (if encountered). Soil will be field screened above. After sampling, soil will be returned to the excavation in the approximate order it was removed. Soil will be compacted with the excavator bucket in 1-foot lifts. The surface will be finished such that no hazards are protruding from the ground (e.g., large buried debris, chunks of asphalt or metal, etc.).

In each instance, drill rigs or excavators are required to have spill kits and fire extinguishers available in the event of a hydraulic oil or fuel release or fire.

Sample target depths are summarized in **Table 1**, which is to be used as a field guide.

Grab/Discrete Sampling

Grab/discrete samples will be collected according to Credere SOP CA-5. Visible asphalt and base materials, landscaping materials, and other organic detritus will be removed prior to

sampling. In all soil samples, representative soil will be collected in accordance with Table 1 while wearing clean nitrile gloves and using decontaminated hand tools (e.g., stainless steel spoon or spade). VPH/VOC samples will be collected directly from the GeoProbe® macrocore using a dedicated soil syringe immediately after exposure to the atmosphere and determination of the appropriate sample collection depth to prevent loss of volatiles and degradation. The remaining representative soil will be placed in a decontaminated stainless steel bowl (see decontamination SOP CA-2), homogenized, and placed in laboratory provided glassware. Proposed sample analysis, the analytical method, container type, and preservative are summarized in **Table 1**.

Samples will be submitted on ice to ARA of Portsmouth, New Hampshire, whom will subcontract certain analyses to Test America of Sacramento, California, for analysis according to **Table 1**.

Excess soil at each location will be returned to its place of origin. Petroleum or free-product saturated soil will be collected into drums and left onsite at an accessible staging area at the southern end of the island to be managed according to **Section 5.8**. The staging area will be near the paint shed, on level concrete, and beyond the Site line from the shoreline.

Incremental Sampling

Incremental sampling will be completed according to Credere SOP CA-26. Each DU has three replicates to be collected. The grid will be overlaid on the figures electronically and the aliquot locations will be located in the field with a sub-foot accuracy GPS. Aliquot locations will be marked in the field with pin flags for later reference. One aliquot will be collected with a tile sampler from the top foot of soil and extruded into a decontaminated stainless steel bowl or plastic bucket. Once the respective number of aliquots has been collected, the soil will be homogenized and transferred to the 1-liter wide mouth laboratory provided sample container. The bowl/bucket will be decontaminated according to Credere SOP CA-2 and the process will be completed for the respective DU for the second and third replicates. Samples will be placed on ice and submitted to ARA for analysis according to **Table 1**. ARA will perform the ISM laboratory compositing in accordance with their SOP, QA-801 Sample Readiness, and appropriate analytical volume will be produced from this method for analysis. It will be important to provide the soil samples to the laboratory promptly to allow for their ISM processing and transfer of subcontracted analyses to the sub-lab within the respective sample holding time. Excess soil will be returned to the surface in the respective DU.

5.3 MONITORING WELL INSTALLATION & GROUNDWATER SAMPLING

Overburden Well Installation

Overburden monitoring wells will be installed according to Credere SOP CA-8. Each overburden monitoring well (OBMW) will be constructed in a new borings adjacent to initial direct push boring by advancing casing to allow for installation of adequate annulus materials. Wells will be constructed of 10 feet of 2-inch diameter 0.010-inch slotted PVC screen with at least 7 feet of screen below the depth of the water table to allow for groundwater table

fluctuations and enough solid PVC riser to reach the ground surface. The well annulus will be filled with No. 2 washed silica sand, and a 2-foot bentonite seal will be installed 1 foot above the screen. Each well will be finished with a standpipe, locking cap, and lock. Each standpipe will be cemented in-place. A well construction diagram will be completed to supplement the well construction log.

Piezometer Installation

Temporary piezometers (PZs) will be installed along the beach due to the tidal action and likely limited lifetime of a permanent monitoring well. Piezometers will be installed after low tide to ensure the piezometer is screened across the low water depth. Sufficient 1-inch diameter 0.010-inch slotted PVC screen will be installed in test pits to the low water depth up to 1 foot bgs with solid PVC to the surface and a 3-foot PVC stickup with a well plug. Test pits will be backfilled around the piezometers.

Bedrock Well Installation

Bedrock monitoring wells will be installed according to Credere SOP CA-24. Overburden soil will be removed to the bedrock interface per **Section 4.3** and 4-inch steel casing will be installed 5 to 10 feet into the bedrock surface. Grout will be allowed to set for 24 hours prior to continuing with bedrock drilling. Based on the design of successfully completed monitoring wells at the adjoining Navy property, bedrock monitoring wells (BRMWs) will be advanced 30 feet into reportedly soft bedrock with an NX core sampler or further if groundwater is not encountered within the top 30 feet of bedrock. Depending on Site conditions and bedrock coring success at the first location, air hammer may be substituted for subsequent wells. The hole will then be reamed to a 4-inch diameter hole for well construction. Bedrock is reportedly weathered with poor rock quality; therefore, bedrock wells will be screened with 30 feet of schedule 40 2-inch diameter 0.01-inch slotted PVC screen. The well annulus will be filled with No. 2 washed silica sand, and a 2-foot bentonite seal will be installed 1 foot above the screen. Each well will be finished with a standpipe and locking cap. A well construction diagram will be completed to supplement the well construction log.

Well Development

Monitoring well development will be completed according to Credere SOP CA-9. Each overburden well will be developed by over pumping and agitation methods. A surge block and check valve will be used during bedrock well development. The wells will be purged until the discharge is relative free of sediment and a total of at least three well volumes have been removed. The turbidity target is less than 10 nephelometric turbidity units (NTUs). Discharge water will also be monitored for stability using a multi-parameter sonde (MPS) such as a Yellow Springs Instrument (YSI), or similar. Development will be considered complete when the following parameters are stabilized according to the following criteria:

- pH: ±1 standard unit
- Temperature: ±3%
- Oxidation reduction potential (ORP): ±10 millivolts (mV)

- Dissolved oxygen (DO): ±0.5 mg/L for values less than 2 milligrams per liter (mg/L) or ±10% for values greater than 2 mg/L
- Specific conductivity: ±3%

Purge water will be discharged to a vegetated surface near the well to allow for re-infiltration. If well development results in purging free-product, purge water will be containerized in a drum and stored on the aviation pad at the southern end of the Site. This waste will be managed according to **Section 5.8**.

Groundwater Sampling

Credere will allow at least 7 days for the monitoring wells to equilibrate with the surrounding aquifer prior to sampling.

Monitoring well top of well/casing elevations as well as ground surface will be surveyed by a Rhode Island licensed surveyor. Depth to groundwater and light non-aqueous phase liquid (LNAPL)/dense non-aqueous phase liquid (DNAPL) thicknesses, if present, will then be measured with an interface probe relative to the top of casing elevations to allow for the calculation of relative groundwater elevations and the determination of groundwater flow direction at the Site. Gauging will be completed according to Credere SOP CA-10.

Credere will then sample overburden wells according to Credere SOP CA-12 with a peristaltic pump and the bedrock wells with a bladder pump using low-flow sampling methodologies or no-purge methodologies where appropriate per below. Wells will be purged at a stable flow rate between 100 and 400 mL per minute to avoid drawdown of the water level. Purging will occur by one of the following methods:

1. If a stable flow rate is achieved, groundwater will be monitored with an appropriately calibrated MPS (see Credere SOP CA-11) after filling of the flow through cell for temperature, pH, ORP, specific conductivity, DO, and turbidity using a MPS and an in-line flow-through cell until parameters have stabilized (see stabilization criteria in Well Development section) over a period of three readings, spaced 5 minutes apart or at a spacing to allow for a complete exchange of flow through the flow-through cell based on the flow-through cell volume and flow rate. If parameters do not stabilize within a period of 2 hours or before a maximum purge volume of 5 well volumes, samples will be collected with field note justification of attempts to achieve stabilization and data will be reviewed for evidence of bias.
2. If a stable flow rate greater than 100 mL/min cannot be achieved and drawdown continues beyond 0.3 feet, purging will be ceased and the no-purge sampling method will be implemented. The pump will be placed at the desired pump intake, one tubing volume will be purged, and samples will be collected. The wells will not be permitted to be pumped dry.

Groundwater samples will be collected immediately after the pump and directly into the appropriate bottle ware in order of decreasing volatility. Samples will be placed on ice and

submitted to ARA for analysis. Proposed sample analysis, analytical method, required container, and preservative are summarized below in **Table 1**.

Well sampling purge water will be discharged to a vegetated surface near the respective well. If sampling results in purging of free product, purge water will be containerized in drums and stored on the aviation pad at the southern end of the island and will be managed according to **Section 5.8**.

5.4 SEDIMENT SAMPLING

Sediment sampling will be completed according to Credere SOP CA-13.

Tidal Zone Sediment Samples

Tidal zone sediment samples will be collected during low tide via soil borings or test-pitting for the deeper samples intended to assess the extent of tar and with hand tools for the shallow risk-based samples. If soil borings are possible, GeoSearch will advance 24-inch cores through evidence of contamination or to a minimum depth of 6 feet unless sloughing saturated sands prevent such a depth. Hand tools may also be used to supplement coring if significant sloughing occurs. Sediment cores will be collected continuously using dedicated, disposable polyethylene liners. Macrocores will be individually logged, evidence of contamination will be noted, and soil will be field screened for total VOCs using a ppbRAE 3000 (or similar) PID calibrated with a 10 ppm_v isobutylene gas standard and an instrument response factor of 1.0. Sample target depths are summarized in **Table 1**, which is to be used as a field guide.

If field conditions limit the use of soil boring equipment, a small excavator will be used to dig test pits and sediments will be composited with hand tools from each target interval.

Representative sediment will be collected while wearing clean nitrile gloves and using decontaminated hand tools (e.g., stainless steel spoon or spade). VPH/VOC samples will be collected directly from the GeoProbe® macrocore using a dedicated soil syringe immediately after exposure to the atmosphere and determination of the appropriate sample depth to prevent loss of volatiles and degradation. The remaining representative sediment will be placed in a decontaminated stainless steel bowl, decanted, homogenized, and placed in laboratory provided glassware. Samples will be submitted on ice to ARA, whom will subcontract certain analyses to TAS, for analysis according to **Table 1**.

Excess sediment will be returned to its place of origin.

Ponar Sediment Sampling

Deeper water sediment samples will be collected from a boat and will require a team of four personnel: the boat driver, GPS locator, sampler, and onshore spotter (may be performing other activities onshore as long as eye contact can be made with the boat crew). Samples should be collected during favorable weather conditions with relatively calm waters. The boat driver will be responsible for maintaining the position of boat over the sampling locations that is to be

tracked by the GPR locator. The sampler will quickly collect the sediment to minimize drift from the location.

The sampler will be retrieved into the boat and inspected for evidence of contamination, odor, staining, or otherwise. Representative sediment will be collected while wearing clean nitrile gloves and using decontaminated hand tools (e.g., stainless steel spoon or spade). VPH/VOC samples will be collected directly from the Ponar sampler using a dedicated soil syringe immediately after exposure to the atmosphere and determination of the appropriate sample depth to prevent loss of volatiles and degradation. The remaining representative sediment will be placed in a decontaminated stainless (see decontamination SOP CA-2) steel bowl, decanted, homogenized, and placed in laboratory provided glassware. Samples will be submitted on ice to ARA, whom will subcontract certain analyses to TAS, for analysis according to **Table 1**.

Excess sediment will be discharged overboard to allow for resettling in the location of collection.

5.5 CONCRETE SAMPLING

Concrete sampling will be completed according to Credere SOP CA-23. Concrete samples will be collected from depths of 0.0 to 0.5 inches in a 3 meter grid surrounding the transformer release. A 0.5-inch masonry drill bit will be advanced 0.5 inches into the concrete floor with a rotary hammer drill. This method will be duplicated in adjoining holes to collect adequate sample volume (20 grams) to be measured using a scale. Concrete dust will be collect using a dedicated scoopula into laboratory provided glassware. Samples will be submitted to ARA for analysis of PCBs with soxhlet extraction.

5.6 SAMPLE HANDLING

Sample Identification

Sample nomenclature will be consistent with the USACE New England District Data Management Plan (CENAE, 2016). Sample identification will be NED Site specific (e.g., 10SB, 35SB) consistent with prior sample IDs for the Site.

Soil sample IDs will consist of the NED Site identifier, sample type designation (e.g., SB for soil boring, SS for surface soil, DU for decision unit incremental sample, SL for sludge, W for various wastes or debris, CC for concrete, SD for sediment, OBMW for overburden monitoring well, BRMW for bedrock monitoring wells, PZ for piezometers, CP for composite), and the # for the specific location and will be followed by the specific sample designation. Sample IDs are provided with their applicable location in **Table 1**.

Duplicate sample IDs will be designated as blind duplicates by adding a zero to the end of the ID. For example, a duplicate of 10SB1-1 would have an ID of 10SB1-10.

Sample Labeling

Each sample container will be affixed with a self-sticking, waterproof, adhesive label. Each label shall be completed with a pen of indelible ink and contain the following information:

- Client Name: Credere Associates, LLC
- Site Name: Gould Island
- Client Sample ID: 10SB1-1, for example
- Date Collected: (month/day/year)
- Sample Time: given in 24-hour format (for example: 1400)
- Initials of Collector: Credere field sampler
- Analytical method/analysis requested
- Preservative: (for example – None)

Sample Chain of Custody

Credere will follow procedures outlined in Credere SOP CA-16. A sample chain of custody (COC) will be designed to assure each sample is accounted for at all times. A chain of custody form must be completed by the appropriate sampling and laboratory personnel for each sample. The objective of the sample custody identification and control system will be to assure the following:

- Samples scheduled for collection are uniquely identified.
- The correct samples are analyzed and are traceable to their records.
- Samples are protected from loss, damage, or tampering.
- Alteration of samples (e.g., filtration, preservation) is documented.
- A forensic record of sample integrity is established.

The COC form includes the following:

- Sample number and sample bottle identification number, where applicable
- Names of the sampler(s) and the person shipping the samples
- Purchase order number and/or project number
- Name, telephone number, and fax number or email address of the contact person from Credere
- Project name (including specific portion of the Site if a large project)
- Signature of the sampler
- Date and time that the samples were collected

- Names of those responsible for receiving the samples and the date and time received at the laboratory
- Matrix of the sample
- The number of containers for a particular sample
- Analysis, container type, and preservative information

Corrections to a chain of custody will be made by putting one line through the incorrect entry and initialing and dating it. The chain of custody record will accompany the samples to the laboratory and a carbon copy of the chain of custody will be retained by the sampler. The chain of custody forms will be supplied by ARA with the data package and will be included with the laboratory analytical reports.

Sample Handling

Samples will be stored onsite in coolers packed with ice until they are sent to the laboratory for analysis. Glass bottles will be packed snugly with packing materials to protect the containers from breakage. Ice will be added to the cooler, and the chain of custody form will be placed in the cooler in a waterproof bag. Samples will be placed in the coolers directly after sampling to prevent overexposure to sunlight and to keep them cool for preservation. Field personnel will be responsible for the security of the samples before they are shipped/transferred to the laboratory. Coolers and samples will be stored in a secure or monitored area onsite until they are shipped/transported to the laboratory. During times of excessive heat or prolonged holding of samples, the field personnel are responsible to maintain the ice preservation and avoid the submersion of sample containers in water and obscuring the sample labels. Alternatively, during periods of cold, field personnel are responsible to prevent certain samples from freezing (e.g., groundwater samples with no headspace).

Sample coolers will be sealed with a custody seal and duct/packing tape, if using a courier service, and then transported to the laboratory for analysis.

5.7 DECONTAMINATION

Decontamination will be completed according to Credere SOP CA-2. Miscellaneous tools and equipment used for environmental sampling will be cleaned of bulk material, rinsed (outside, if possible), washed with a detergent solution, rinsed with deionized (DI) water, rinsed with nitric acid and finally rinsed with DI water. Clean tools will be placed in a clean container or covered with aluminum foil to prevent cross contamination. Methanol may be used for cleaning of tar-like material in NED Site No. 10.

5.8 INVESTIGATION DERIVED WASTE

Per **Section 5.2** and **5.3**, saturated excess soil or liquid free-product containing purge water will be containerized in buckets/drums and will be managed according to RIDEM's Policy Memo 95-01 Guidelines for the Management of Investigation Derived Wastes (RIDEM, 2011). Any solid

and liquid waste will be characterized for disposal according to disposal facility requirements and in accordance with RIDEM's Rules and Regulations for Hazardous Waste Management (RIDEM, 2016).

5.9 FIELD DOCUMENTATION

Field documentation will be completed according to Credere SOP CA-1.

Field Logbook

Field activities will have a dedicated field logbook, which will be reviewed for accuracy and completeness in accordance with Credere's SOP CA-1 by the Field Manager/Project Manager and scanned into the electronic project file with an appropriate file designation that includes the field activity and date at a minimum.

The daily entry will begin at the top of a new page and be printed legibly. Entries will be recorded using a 24-hour time clock and entered in the order that they occur. A new entry for each day will begin with the following:

- Date and project at the top of the page
- First and last name of Credere employee followed by initials and time of arrival onsite
- Full names and initials of additional team members and the time of their arrival onsite
- Scope of work for the day
- Weather (e.g., temperature, precipitation, wind directions) and tides
- Subcontractors and duties (e.g., driller, make and model of drilling equipment, foreman)
- Documentation of a safety acknowledgement or tool box meeting

If field activities extend beyond one page, each successive page shall have the date, project and initial of person doing logbook entries written at the top. If the logbook changes hands during a daily entry, the initial writer shall sign after their last entry and the full name and initials of the new writer shall be entered consecutively.

The final page of a daily entry shall have open lines marked diagonally with a strike and shall be signed and dated.

The following is a partial list of information typically recorded during field tasks:

- Level of personal protective equipment (PPE)
- Changes to PPE level
- Changes in personnel
- Sampling equipment serial numbers
- Equipment calibration details
- Decontamination procedures

- Field screening results
- Observations
- Unusual circumstances
- Problems encountered
- Deviations from the work plan
- Problem resolutions
- Safety issues/accidents/near misses/discussions with contractors
- Safety resolutions
- Name and time onsite/offsite of anyone who enters the Site
- Correspondence with project managers
- Sample IDs
- Sample depths
- Method of collection
- Sample times
- Sample analysis requested
- Sample preservation
- Sample volume collected
- Quality Assurance (QA)/QC samples
- Sample duplicate locations
- Site sketch
- Changes in weather

Field Forms

Credere utilizes dedicated soil boring and groundwater sampling field forms to facilitate field data collection. Field forms will be referenced in the daily logbook entry. Procedures for data entry and error correction will be followed when using field forms. Field forms will be thoroughly completed and self-checked at the time of the field task to avoid missing information. **Use of field forms does not exclude the need for field notes**. Field notes may not require sampling information that is included on the field forms; however, the general timeline of the day (e.g., the time a boring was started and complete, or the time a well began purging and was completed) should still be included in the field notes. Additionally, any deviations from the work plan or scope should also be documented in the field notes in addition to on the field forms.

6. REFERENCES

Alion, 2009. *Final Site Inspection (SI) Report for Gould Island*. Prepared by Alion Science and Technology, Prepared for USACE: Dated August 2009.

CENAE, 2016. *Formerly Used Defense Sites Online Chemical Database Data Management Plan*: Dated April 2016.

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RIDEM, 2011. *Rules and Regulations for the Investigation and Remediation of Hazardous Material Releases.* Prepared by Rhode Island Department of Environmental Management: Dated November 2011.

RIDEM, 2016. *Rules and Regulations for Hazardous Waste Management*. Prepared by Rhode Island Department of Environmental Management: Dated September 30, 2016.

RIHPHC, 1995. *Historic and Architectural Resources of Jamestown, Rhode Island*. Prepared by the Rhode Island Historical Preservation & Heritage Commission (RIHPHC): Dated 1995.

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USACE, 2001. EM 200-1-3 Requirements for the Preparation of Sampling and Analysis Plans.

FIGURES

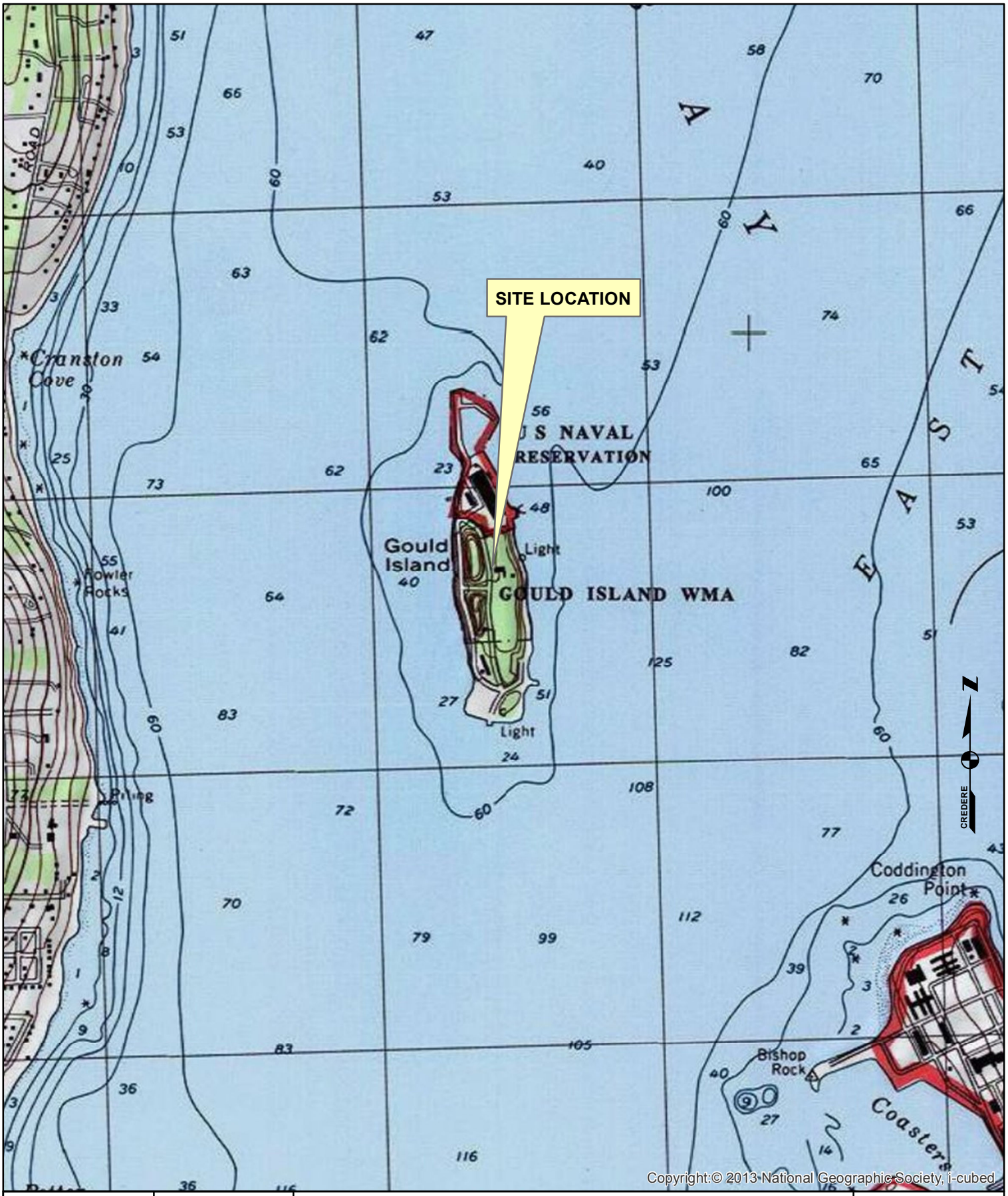

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FIGURE 1
SITE LOCATION PLAN

GOULD ISLAND
NARRAGANSETT BAY
JAMESTOWN, RHODE ISLAND

1,000 0 2,000 Feet

1 INCH = 2,000 FEET

T:\Data\NH\Town\Franklin\14001247-Ferrari Mill

LEGEND

NED SITE NUMBER	LOCATION	NED SITE NAME
1	D-4	COAL STORAGE
2	B-5	MARINE BARRACKS (FOUNDATION REMAINING)
3	C-5	FORMER CARPENTERS SHOP
4	E-9	PUMP HOUSE
5	D-7	FORMER UNKNOWN BUILDING #2
6	E-10	UNKNOWN BUILDING #3
7	D-5	FORMER UNKNOWN BUILDING #4 (FOOTINGS REMAINING)
8	E-7	INCINERATOR
9	D-6	MAGAZINE IGNITOR STORAGE
10	E-8	DISPOSAL AREA #14
11	C-6	FORMER QUONSET HUTS/MAINTENACE SHOPS
12	C-7	MAINTENANCE SHOPS/GARAGE/FIRE STATION
13	B-8	ELECTRIC SUBSTATION TRANSFOMER PEN
14	C-8	FORMER MARINE BARRACKS
15	B-9	FORMER RECREATION BUILDING
16	C-9	FORMER BARRACKS
17	C-9	FORMER TRANSFORMER VAULT
18	D-10	WATER TREATMENT PLANT
19	C-9	TORCH POT STORAGE
20	B-6	FORMER CABLE TERMINAL BUILDING
21	B-11	BUNKER #11
22	B-12	BUNKER #12
23	C-12	COAL STORAGE
24	B-12	CABLE TERMINAL BUILDING
25	B-12	UNDERGROUND STORAGE TANKS

NED SITE NUMBER	LOCATION	NED SITE NAME
26	C-13	GAS PUMP HOUSE/GAS TANKS (REMOVED)
27	C-13	TWO GAS PITS
28	C-13	FORMER ORDNANCE TEST FACILITY, HANGER
29	D-13	FORMER UNKNOWN BUILDING #1 (HANGAR 5496-61)
30	E-13	FORMER PYROTECHNIC STORAGE
31	E-13	GAS PIT
32	E-12	FORMER DRUM STORAGE AREA
33	D-12	FORMER PAINT AND OIL STORAGE
34	D-12	THEATER/RESEARCH BUILDING (FOUNDATION REMAINING, CONCRETE PLATFORM)
35	E-12	BOILER HOUSE
36	D-11	DEGAUSSING BUILDING
37	D-11	MISCELLANEOUS STORAGE
38	D-11/12	STORAGE
39	D-6	WELLHOUSE #81
40	C-9	WELLHOUSE #78
41	D-8	AA GUNS ELECTRICAL SUPPLY (UTILITY POLE REMAINING)
42	C-13	5,000 GALLON AVGAS TANK
43	D-13	FORMER PAINT SHED
44	C-14	EMPTY DRUMS
45	D-8	FORMER FIRE APPARATUS HOUSES (2)
46	D-7, D-8, E-7, E-8	FORMER AA GUN EMPLACEMENTS (4)
47	C-10	UNDERGROUND WATER TANK
48	D-6	DEBRIS STOCKPILE
N/A	C-13	FORMER BACKGROUND SAMPLE #1 LOCATION (BG-48SS-001)
N/A	D-7	FORMER BACKGROUND SAMPLE #2 LOCATION (BG-48SS-002)

CREDERE

250 125 0 250 Feet

1 INCH = 120 FEET

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FIGURE 2
DETAILED SITE PLAN

GOULD ISLAND
NARRAGANSETT BAY
JAMESTOWN, RHODE ISLAND

- 10 NED SITE NUMBER
- PREVIOUS SEDIMENT SAMPLE LOCATION (STONE AND WEBSTER, 1997)
- PREVIOUS SOIL SAMPLE LOCATION (STONE AND WEBSTER, 1997)
- HISTORICAL EDGE OF ROADWAY
- HISTORICAL UNPAVED ROAD
- HISTORICAL RAIL LINE
- 5 FOOT TOPOGRAPHIC CONTOUR LINE
- US NAVY OR COAST GUARD EASEMENTS / NON-FUDS ELIGIBLE
- HISTORICAL STRUCTURES
- DISPOSAL AREA
- APPROXIMATE SITE BOUNDARY
- APPROXIMATE PARCEL BOUNDARY

NOTES:
EXISTING CONDITIONS FEATURES SHOWN ON THIS PLAN ARE APPROXIMATE AND ARE BASED ON INFORMATION OBTAINED FROM 2014 USDA NAIP DIGITAL TRUE COLOR ORTHOPHOTOGRAPHY, US COAST GUARD, AND ENGINEERING EVALUATION OF CONTAMINATION FORMER NAVY TORPEDO TESTING FACILITY BY STONE & WEBSTER ENVIRONMENTAL TECHNOLOGY & SERVICES.

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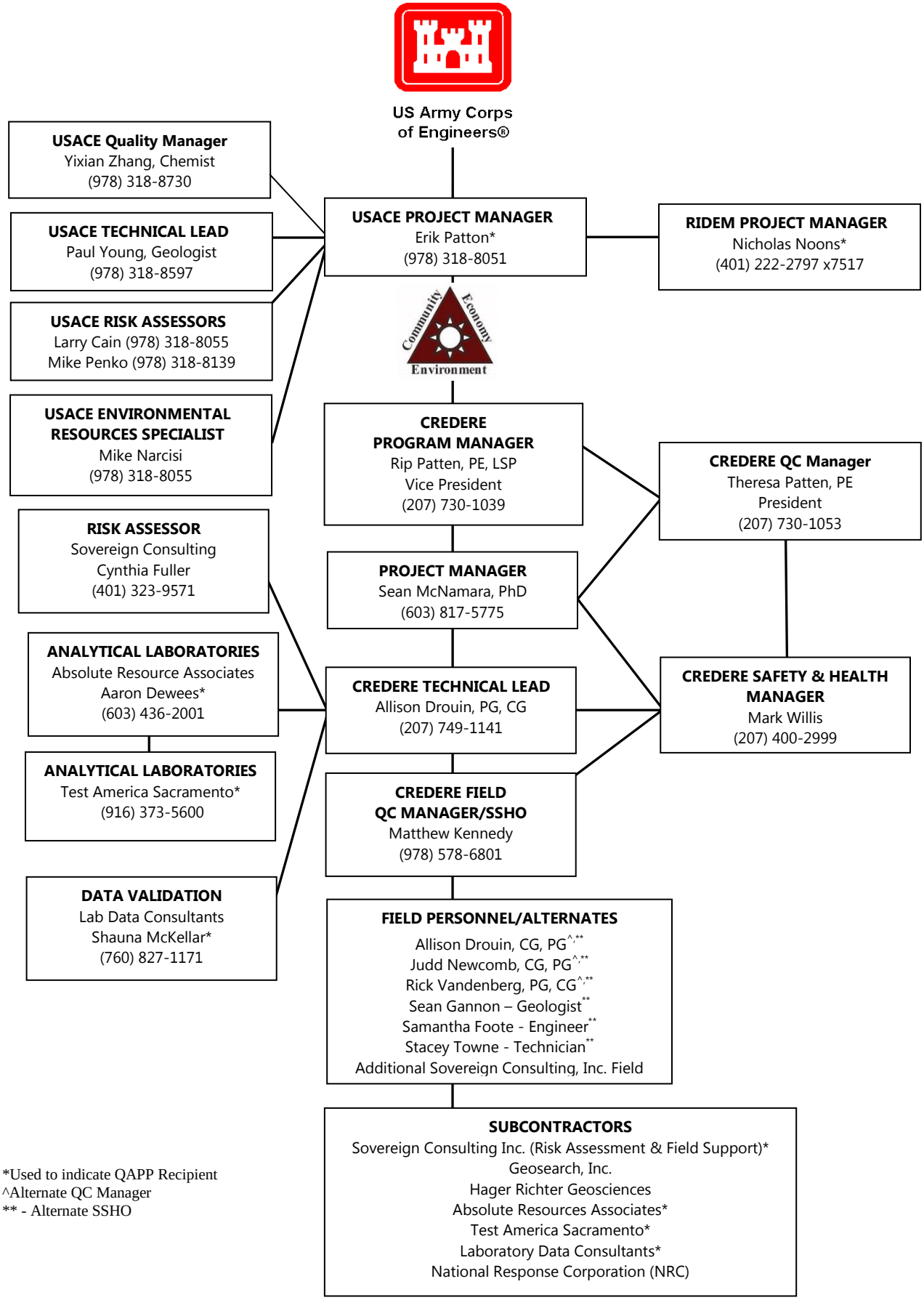
Figure 3 – Project Organization Flow Chart



*Used to indicate QAPP Recipient
^Alternate QC Manager
** - Alternate SSHO

AREA OWNED BY US NAVY

KEEPER'S HOUSE
BG-4
BG-7
BG-6
13CC-1 - 13CC-4
WELLHOUSE
BG-5
BG-8
13SS-3
13SS-6
13
13SS-4
13SS-001
13SS-002
13SS-5
PARTIALLY BURIED TRANSFORMERS
20
BG-3
BRMW-4
11SS-1 - 11SS-3 FROM 3 ALIQUOTS IN 11DU1
POSSIBLE UST VENT PIPE/ PRESUMED LOCATION OF UST, IF PRESENT
16T-1 THROUGH 16T-3 AND 16SS1- THROUGH 16SS-16 AREA
2
BG-2
BG-1
SPILLED TRANSFORMER OIL
FORMER COAL SHED
15
TENNIS COURT
3
11DU1
11
12SB2/ 12OBMW2
12SB3
12SB4
12
14
12SS-1 THROUGH 12SS-9 AND 12CC-1
16
17
12SB1/ 12OBMW1
DRUM
MAINTENANCE PIT (12W-1 THROUGH 12W-3)
40SS-001
40
47
01DU1
01DU3
39 (39CC-1 AND 39SS-1 THROUGH 39SS-4)
UST REMOVED
MAIN ROAD
PUMPHOUSE
01SB2
39SS-001
WATER TOWER (TOPPLED)
BUNKER
19DU1
18
01SB1
1
01SB3
01SB7
01SB10
PUMPHOUSE
FORMER BACKGROUND (48SS-002)
19
19SS-001
EXISTING MONITORING WELLS
01SB6
1
01SS-001
01SB9
48SB2
46
QUONSET HUT
QUONSET HUT
01SB4
7
01SB5
01SB8
48
NORTH HILL
48SB1
45
41
45
41SS-001
48DU1
5
FOR PROPOSED SAMPLE LOCATIONS AND DETAIL OF INSET SEE FIGURE 4B
6
9
01DU2
46
46
GI-DA-SS-02-01
GI-DA-SS-02-02
WEST ROAD
BRMW-5
8
GI-DA-SS-02-03
4
10SS-002
10SS-001
10
GI-DA-SB-04-01
SMALL BOAT LANDING (DETERIOTATED)
GI-DA-SD-02-02
GI-DA-SD-02-01
S-5
S-4
S-1
S-2
S-3
S-6
S-7
S-8
S-9
S-10

CREDERE

240 120 0 240 Feet

1 INCH = 120 FEET

LEGEND

NED SITE NUMBER	NED SITE NAME
1	COAL STORAGE
2	MARINE BARRACKS (FOUNDATION REMAINING)
3	FORMER CARPENTERS SHOP
4	PUMP HOUSE
5	FORMER UNKNOWN BUILDING #2
6	UNKNOWN BUILDING #3
7	FORMER UNKNOWN BUILDING #4 (FOOTINGS REMAINING)
8	INCINERATOR
9	MAGAZINE IGNITOR STORAGE
10	DISPOSAL AREA #14
11	FORMER QUONSET HUTS/MAINTENACE SHOPS
12	MAINTENANCE SHOPS/GARAGE/FIRE STATION
13	ELECTRIC SUBSTATION TRANSFOMER PEN
14	FORMER MARINE BARRACKS
15	FORMER RECREATION BUILDING
16	FORMER BARRACKS
17	FORMER TRANSFORMER VAULT
18	WATER TREATMENT PLANT
19	TORCH POT STORAGE
20	FORMER CABLE TERMINAL BUILDING
21	BUNKER #11
22	BUNKER #12
23	COAL STORAGE
24	CABLE TERMINAL BUILDING
25	UNDERGROUND STORAGE TANKS
26	GAS PUMP HOUSE/GAS TANKS (REMOVED)
27	TWO GAS PITS
28	FORMER ORDNANCE TEST FACILITY, HANGER
29	FORMER UNKNOWN BUILDING #1 (HANGAR 5496-61)
30	FORMER PYROTECHNIC STORAGE
31	GAS PIT
32	FORMER DRUM STORAGE AREA
33	FORMER PAINT AND OIL STORAGE
34	THEATER/RESEARCH BUILDING (FOUNDATION REMAINING, CONCRETE PLATFORM)
35	BOILER HOUSE
36	DEGAUSSING BUILDING
37	MISCELLANEOUS STORAGE
38	STORAGE
39	WELLHOUSE #81
40	WELLHOUSE #78
41	AA GUNS ELECTRICAL SUPPLY (UTILITY POLE REMAINING)
42	5,000 GALLON AVGAS TANK
43	FORMER PAINT SHED
44	EMPTY DRUMS
45	FORMER FIRE APPARATUS HOUSES (2)
46	FORMER AA GUN EMPLACEMENTS (4)
47	UNDERGROUND WATER TANK
48	DEBRIS STOCKPILE
N/A	FORMER BACKGROUND SAMPLE #1 LOCATION (BG-48SS-001)
N/A	FORMER BACKGROUND SAMPLE #2 LOCATION (BG-48SS-002)

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FIGURE 4A
SAMPLE LOCATION PLAN
NORTHERN AREAS OF CONCERN

GOULD ISLAND
NARRAGANSETT BAY
JAMESTOWN, RHODE ISLAND

- PROPOSED SOIL BORING/OVERBURDEN MONITORING WELL LOCATION
- PROPOSED SURFACE SOIL SAMPLE LOCATION
- PROPOSED BEDROCK MONITORING WELL LOCATION
- PROPOSED SOIL BORING SAMPLE LOCATION
- EXISTING MONITORING WELL
- PREVIOUS SEDIMENT SAMPLE LOCATION (STONE AND WEBSTER, 1997)
- PREVIOUS SOIL SAMPLE LOCATION (STONE AND WEBSTER, 1997)
- 5 FOOT TOPOGRAPHIC CONTOUR LINE
- DU BOUNDARY
- HISTORICAL EDGE OF ROADWAY
- HISTORICAL RAIL LINE
- SAMPLING BOUNDARY
- HISTORICAL UNPAVED ROAD
- (10) NED SITE NUMBER
- US NAVY OR COAST GUARD EASEMENTS / NON-FUDS ELIGIBLE
- HISTORICAL STRUCTURES
- DISPOSAL AREA
- APPROXIMATE SITE BOUNDARY
- APPROXIMATE PARCEL BOUNDARY

NOTES:
EXISTING CONDITIONS FEATURES SHOWN ON THIS PLAN ARE APPROXIMATE AND ARE BASED ON INFORMATION OBTAINED FROM 2014 USDA NAIP DIGITAL TRUE COLOR ORTHOPHOTOGRAPHY, US COAST GUARD, AND ENGINEERING EVALUATION OF CONTAMINATION FORMER NAVY TORPEDO TESTING FACILITY BY STONE & WEBSTER ENVIRONMENTAL TECHNOLOGY & SERVICES.

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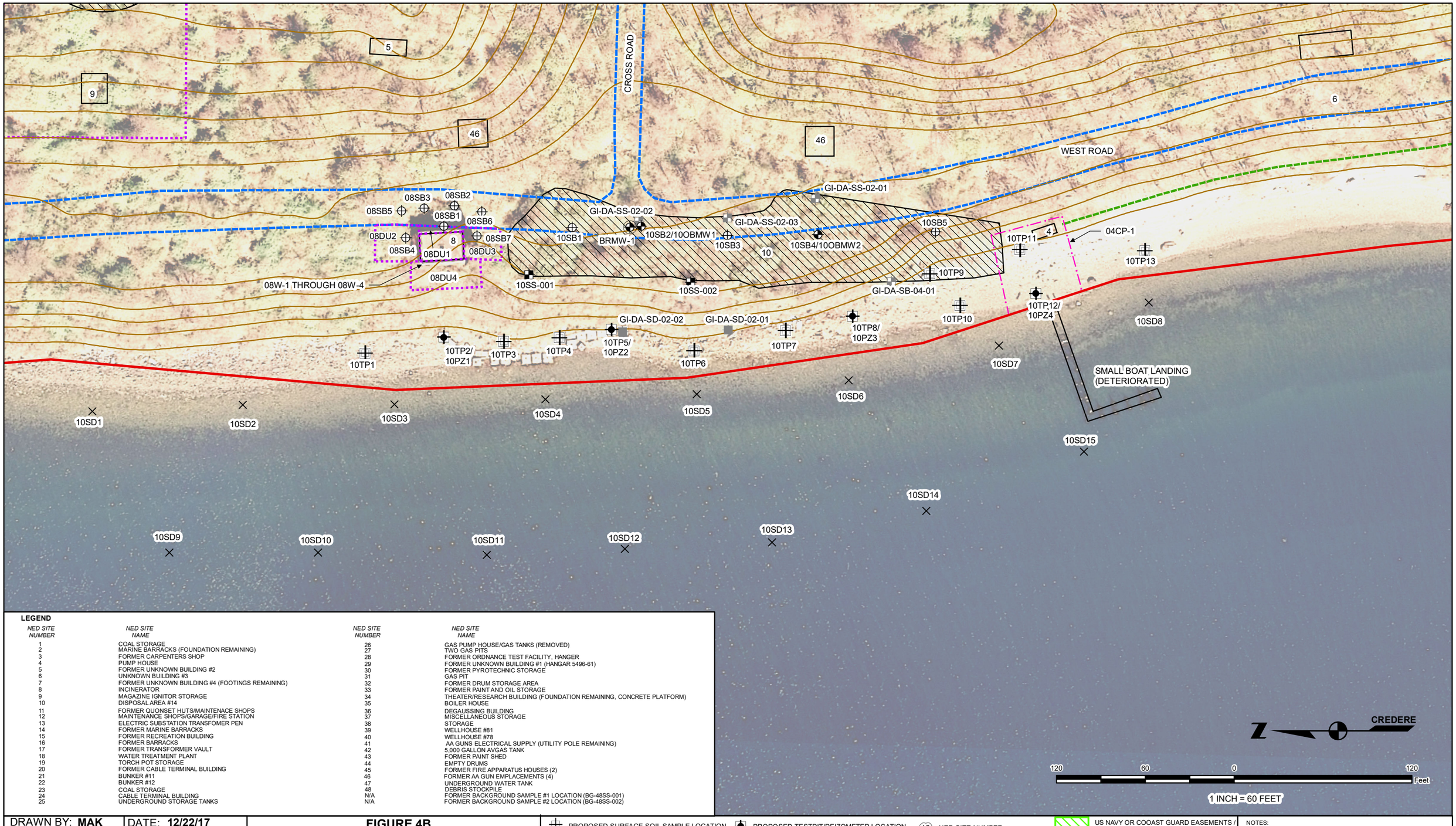

LEGEND

NED SITE NUMBER	*NED SITE NAME*	*NED SITE NUMBER*	*NED SITE NAME*
1	COAL STORAGE	26	GAS PUMP HOUSE/GAS TANKS (REMOVED)
2	MARINE BARRACKS (FOUNDATION REMAINING)	27	TWO GAS PITS
3	FORMER CARPENTERS SHOP	28	FORMER ORDNANCE TEST FACILITY, HANGER
4	PUMP HOUSE	29	FORMER UNKNOWN BUILDING #1 (HANGAR 5496-61)
5	FORMER UNKNOWN BUILDING #2	30	FORMER PYROTECHNIC STORAGE
6	UNKNOWN BUILDING #3	31	GAS PIT
7	FORMER UNKNOWN BUILDING #4 (FOOTINGS REMAINING)	32	FORMER DRUM STORAGE AREA
8	INCINERATOR	33	FORMER PAINT AND OIL STORAGE
9	MAGAZINE IGNITOR STORAGE	34	THEATER/RESEARCH BUILDING (FOUNDATION REMAINING, CONCRETE PLATFORM)
10	DISPOSAL AREA #14	35	BOILER HOUSE
11	FORMER QUONSET HUTS/MAINTENACE SHOPS	36	DEGAUSSING BUILDING
12	MAINTENANCE SHOPS/GARAGE/FIRE STATION	37	MISCELLANEOUS STORAGE
13	ELECTRIC SUBSTATION TRANSFOMER PEN	38	STORAGE
14	FORMER MARINE BARRACKS	39	WELLHOUSE #81
15	FORMER RECREATION BUILDING	40	WELLHOUSE #78
16	FORMER BARRACKS	41	AA GUNS ELECTRICAL SUPPLY (UTILITY POLE REMAINING)
17	FORMER TRANSFORMER VAULT	42	5,000 GALLON AVGAS TANK
18	WATER TREATMENT PLANT	43	FORMER PAINT SHED
19	TORCH POT STORAGE	44	EMPTY DRUMS
20	FORMER CABLE TERMINAL BUILDING	45	FORMER FIRE APPARATUS HOUSES (2)
21	BUNKER #11	46	FORMER AA GUN EMPLACEMENTS (4)
22	BUNKER #12	47	UNDERGROUND WATER TANK
23	COAL STORAGE	48	DEBRIS STOCKPILE
24	CABLE TERMINAL BUILDING	N/A	FORMER BACKGROUND SAMPLE #1 LOCATION (BG-48SS-001)
25	UNDERGROUND STORAGE TANKS	N/A	FORMER BACKGROUND SAMPLE #2 LOCATION (BG-48SS-002)

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FIGURE 4B
SAMPLE LOCATION PLAN
WESTERN AREAS OF CONCERN

GOULD ISLAND
NARRAGANSETT BAY
JAMESTOWN, RHODE ISLAND

- PROPOSED SURFACE SOIL SAMPLE LOCATION
- PROPOSED SOIL BORING SAMPLE LOCATION
- PROPOSED SOIL BORING/OVERBURDEN MONITORING WELL LOCATION
- PROPOSED BEDROCK MONITORING WELL LOCATION
- PREVIOUS SOIL SAMPLE LOCATION (STONE AND WEBSETER, 1997)
- PREVIOUS SEDIMENT SAMPLE LOCATION (STONE AND WEBSTER, 1997)
- PROPOSED TESTPIT/PEIZOMETER LOCATION
- PROPOSED SEDIMENT SAMPLE LOCATION
- PROPOSED TESTPIT LOCATION
- (10) NED SITE NUMBER
- 5 FOOT TOPOGRAPHIC CONTOUR LINE
- HISTORICAL EDGE OF ROADWAY
- HISTORICAL RAIL LINE
- SAMPLING BOUNDARY
- HISTORICAL UNPAVED ROAD
- DU BOUNDARY
- US NAVY OR COOAST GUARD EASEMENTS / NON-FUDS ELIGIBLE
- HISTORICAL STRUCTURES
- DISPOSAL AREA 14
- APPROXIMATE SITE BOUNDARY
- APPROXIMATE PARCEL BOUNDARY

NOTES:
1. EXISTING CONDITIONS FEATURES SHOWN ON THIS PLAN ARE APPROXIMATE AND ARE BASED ON INFORMATION OBTAINED FROM 2014 USDA NAIP DIGITAL TRUE COLOR ORTHOPHOTOGRAPHY, US COAST GUARD, AND ENGINEERING EVALUATION OF CONTAMINATION FORMER NAVY TORPEDO TESTING FACILITY BY STONE & WEBSTER ENVIRONMENTAL TECHNOLOGY & SERVICES.
2. SEE FIGURE 2 FOR FULL VIEW OF THE SITE.
3. PREVIOUS SEDIMENT SAMPLE LOCATIONS FROM THE 1984 AND 1985 INVESTIGATIONS BY LOUREIRO ENGINEERING ASSOCIATES ARE NOT SHOWN ON THIS FIGURE. SEE FIGURE 2 FOR THE APPROXIMATE LOCATION OF THESE SAMPLES.

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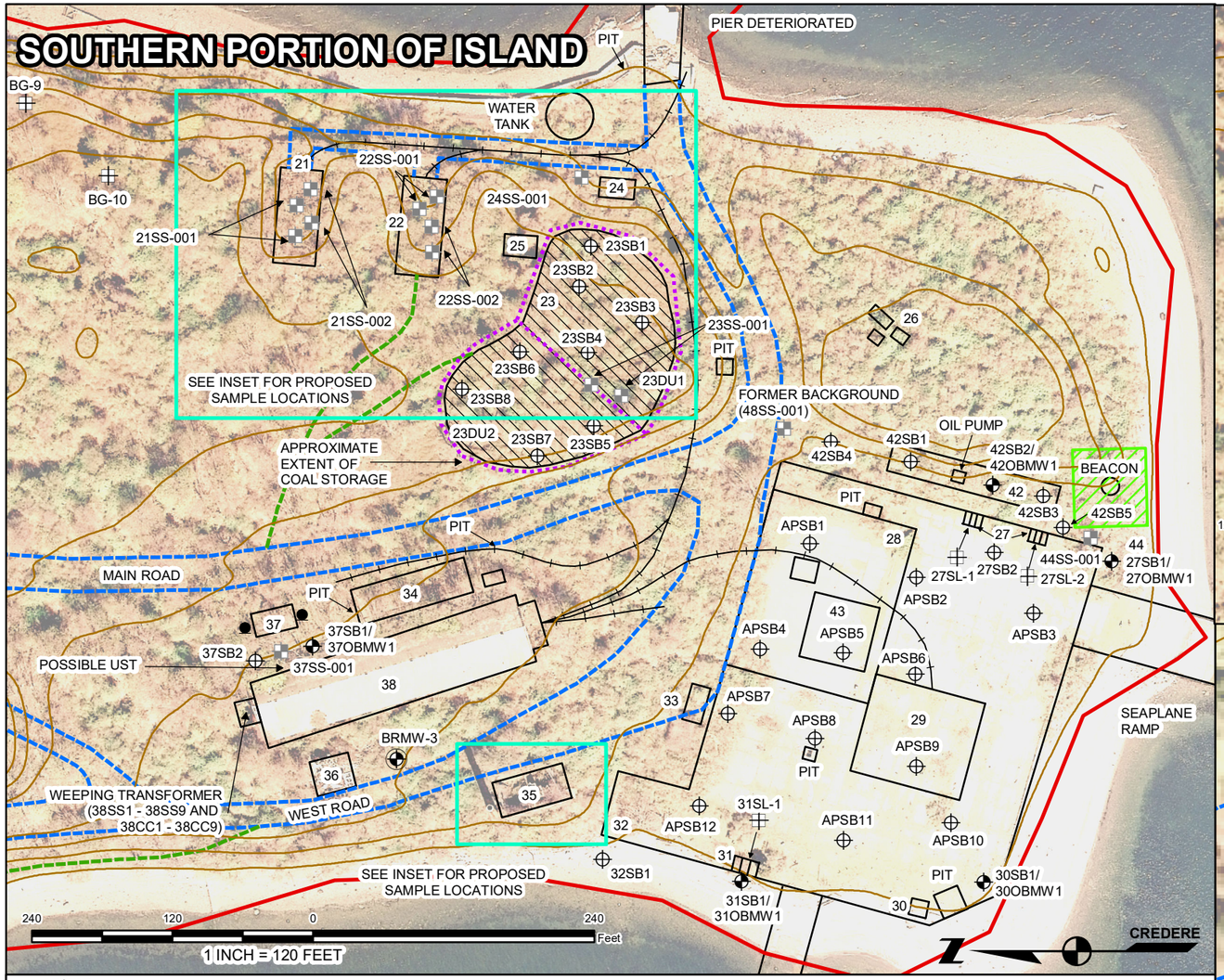

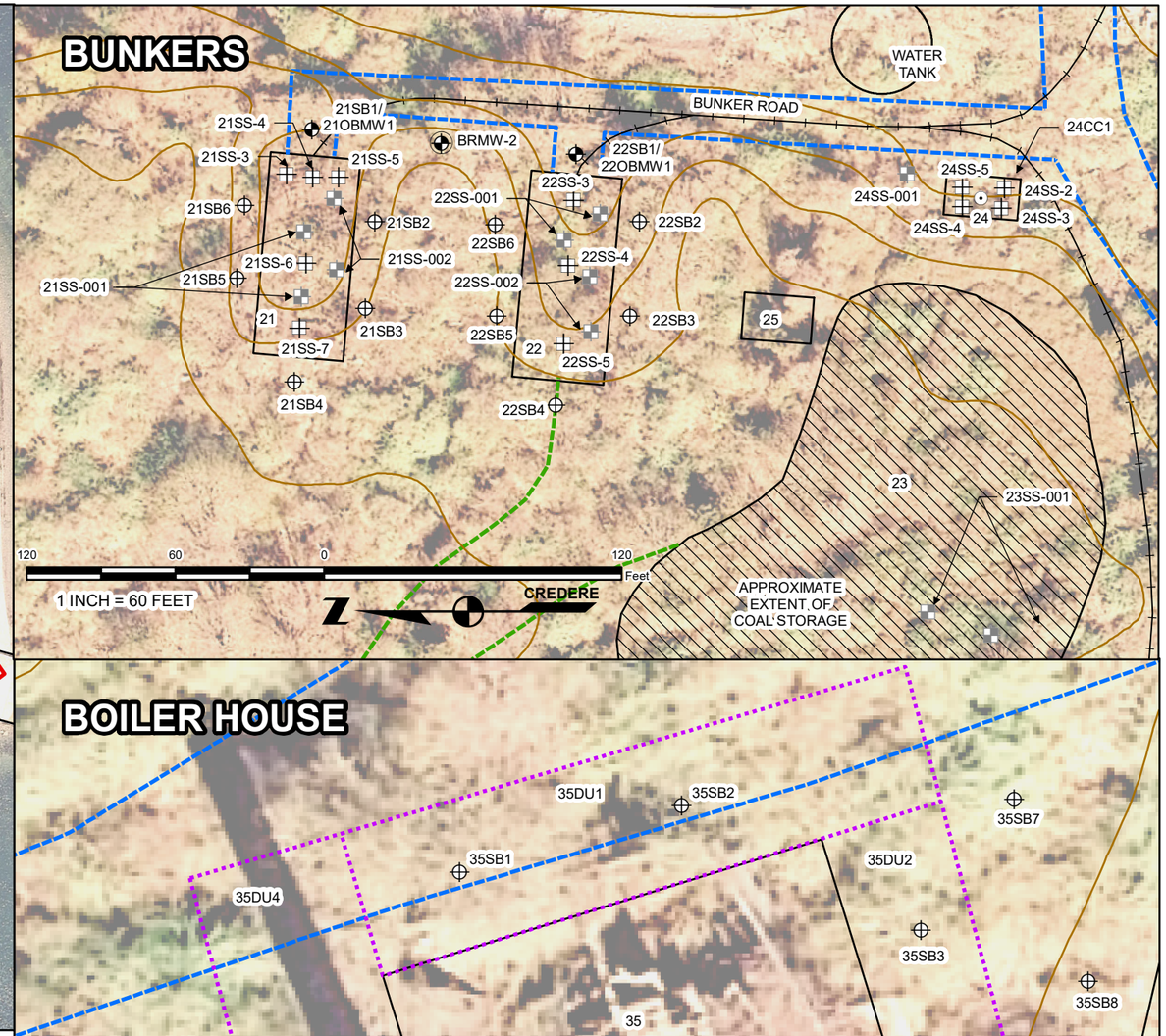

LEGEND

NED SITE NUMBER	NED SITE NAME	NED SITE NUMBER	NED SITE NAME
1	COAL STORAGE	26	GAS PUMP HOUSE/GAS TANKS (REMOVED)
2	MARINE BARRACKS (FOUNDATION REMAINING)	27	TWO GAS PITS
3	FORMER CARPENTERS SHOP	28	FORMER ORDNANCE TEST FACILITY, HANGER
4	PUMP HOUSE	29	FORMER UNKNOWN BUILDING #1 (HANGAR 5496-61)
5	FORMER UNKNOWN BUILDING #2	30	FORMER PYROTECHNIC STORAGE
6	UNKNOWN BUILDING #3	31	GAS PIT
7	FORMER UNKNOWN BUILDING #4 (FOOTINGS REMAINING)	32	FORMER DRUM STORAGE AREA
8	INCINERATOR	33	FORMER PAINT AND OIL STORAGE
9	MAGAZINE IGNITOR STORAGE	34	THEATER/RESEARCH BUILDING (FOUNDATION REMAINING, CONCRETE PLATFORM)
10	DISPOSAL AREA #14	35	BOILER HOUSE
11	FORMER QUONSET HUTS/MAINTENACE SHOPS	36	DEGAUSSING BUILDING
12	MAINTENANCE SHOPS/GARAGE/FIRE STATION	37	MISCELLANEOUS STORAGE
13	ELECTRIC SUBSTATION TRANSFOMER PEN	38	STORAGE
14	FORMER MARINE BARRACKS	39	WELLHOUSE #81
15	FORMER RECREATION BUILDING	40	WELLHOUSE #78
16	FORMER BARRACKS	41	AA GUNS ELECTRICAL SUPPLY (UTILITY POLE REMAINING)
17	FORMER TRANSFORMER VAULT	42	5,000 GALLON AVGAS TANK
18	WATER TREATMENT PLANT	43	FORMER PAINT SHED
19	TORCH POT STORAGE	44	EMPTY DRUMS
20	FORMER CABLE TERMINAL BUILDING	45	FORMER FIRE APPARATUS HOUSES (2)
21	BUNKER #11	46	FORMER AA GUN EMPLACEMENTS (4)
22	BUNKER #12	47	UNDERGROUND WATER TANK
23	COAL STORAGE	48	DEBRIS STOCKPILE
24	CABLE TERMINAL BUILDING	N/A	FORMER BACKGROUND SAMPLE #1 LOCATION (BG-48SS-001)
25	UNDERGROUND STORAGE TANKS	N/A	FORMER BACKGROUND SAMPLE #2 LOCATION (BG-48SS-002)

DRAWN BY: **MAK** | DATE: **12/22/17**
CHECKED BY: **SWM** | PROJECT: **16001327**

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FIGURE 4C
SAMPLE LOCATION PLAN
SOUTHERN AREAS OF CONCERN

GOULD ISLAND
NARRAGANSETT BAY
JAMESTOWN, RHODE ISLAND

- PROPOSED SURFACE SOIL SAMPLE LOCATION
- PREVIOUS SOIL SAMPLE LOCATION (STONE AND WEBSTER, 1997)
- PROPOSED SOIL BORING SAMPLE LOCATION
- PROPOSED SOIL BORING/OVERBURDEN MONITORING WELL LOCATION
- PROPOSED BEDROCK MONITORING WELL LOCATION
- PREVIOUS SEDIMENT SAMPLE LOCATION (STONE AND WEBSTER, 1997)
- PROPOSED CONCRETE SAMPLE LOCATION
- 5 FOOT TOPOGRAPHIC CONTOUR LINE
- DU BOUNDARY
- HISTORICAL EDGE OF ROADWAY
- HISTORICAL RAIL LINE
- HISTORICAL UNPAVED ROAD
- NED SITE NUMBER
- ABOVEGROUND STORAGE TANK
- US NAVY OR COAST GUARD EASEMENTS / NON-FUDS ELIGIBLE
- HISTORICAL STRUCTURES
- DISPOSAL AREA
- APPROXIMATE SITE BOUNDARY
- APPROXIMATE PARCEL BOUNDARY

NOTES:
1. EXISTING CONDITIONS FEATURES SHOWN ON THIS PLAN ARE APPROXIMATE AND ARE BASED ON INFORMATION OBTAINED FROM 2014 USDA NAIP DIGITAL TRUE COLOR ORTHOPHOTOGRAPHY, US COAST GUARD, AND ENGINEERING EVALUATION OF CONTAMINATION FORMER NAVY TORPEDO TESTING FACILITY BY STONE & WEBSTER ENVIRONMENTAL TECHNOLOGY & SERVICES.
2. SEE FIGURE 2 FOR FULL VIEW OF THE SITE.
3. PREVIOUS SEDIMENT SAMPLE LOCATIONS FROM THE 1984 AND 1985 INVESTIGATIONS BY LOUREIRO ENGINEERING ASSOCIATES ARE NOT SHOWN ON THIS FIGURE. SEE FIGURE 2 FOR THE APPROXIMATE LOCATION OF THESE SAMPLES.

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TABLE

Table 1
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Gould Island Site
Narragansett Bay, Jamestown, Rhode Island

NED Site	Sample Location	Sample ID	Associated QC Sample Type/ID	Sample Type	Justification	Target Depth/Methodology	Analyses: VOCs: EPA Method 8260C	SVOCs: EPA Method 8270D	PAHs by EPA Method 8270D	PAHs by EPA Method 8270D w/ SIM	PP Metals EPA Method 6020A & 7471B/7470A [2]	Hexavalent Chromium EPA Method 7196A	EPH by MADEP EPH-04-1.1	VPH by MADEP VPH-04-1.1	PCBs by EPA Method 8082A	Pesticides by EPA Method 8081B	Explosives EPA method 8330B [1]	Dioxin/furan EPA Method 1613B	pH EPA Method 9045C	Sample Volume and Containers/ Preservative (per sample)
1 Coal Storage North	01DU1	01DU1-1	-	Soil	Assess the nature and extent of previously identified surface contamination	ISM 01DU1 is depicted on Figure 4A and will consist of 30 0 to 1-foot aliquots per replicate			s		s	s	s							1, 1 liter widemouth amber
		01DU1-2	-	Soil					s		s	s	s							
		01DU1-3	-	Soil					s		s	s	s							
	01DU2	01DU2-1	-	Soil		ISM 01DU2 is depicted on Figure 4A and will consist of 30 0 to 1-foot aliquots per replicate			s		s	s	s							
		01DU2-2	-	Soil					s		s	s	s							
		01DU2-3	-	Soil					s		s	s	s							
	01DU3	01DU3-1	-	Soil		ISM 01DU3 is depicted on Figure 4A and will consist of 30 0 to 1-foot aliquots per replicate			s		s	s	s							
		01DU3-2	-	Soil					s		s	s	s							
		01DU3-3	-	Soil					s		s	s	s							
	01SB1	01SB1-1	FD: 01SB1-10	Soil	Horizontal and vertical extent of coal in soil by visual examination.	Sample to be collected from the entire thickness of observed coal up to a 4 foot interval (e.g., 0 to 4 feet bgs). If greater than 4 feet of coal is encountered the -2 samples will be collected (e.g., 4 to 8, 4 to 6 feet bgs, etc.).			s		s	s	s							4, 4 oz amber glass
		01SB1-2*	-	Soil					s		s	s	s							2, 4 oz amber glass
	01SB2	01SB2-1	MS/MSD	Soil					s		s	s	s							6, 4 oz amber glass
		01SB2-2*	-	Soil					s		s	s	s							2, 4 oz amber glass
	01SB3	01SB3-1	-	Soil					s		s	s	s							
		01SB3-2*	-	Soil					s		s	s	s							
	01SB4	01SB4-1	-	Soil					s		s	s	s							
		01SB4-2*	-	Soil					s		s	s	s							
	01SB5	01SB5-1	-	Soil					s		s	s	s							
		01SB5-2*	-	Soil					s		s	s	s							
	01SB6	01SB6-1	-	Soil					s		s	s	s							
		01SB6-2*	-	Soil					s		s	s	s							
	01SB7	01SB7-1	-	Soil					s		s	s	s							
		01SB7-2*	-	Soil					s		s	s	s							
	01SB8	01SB8-1	-	Soil					s		s	s	s							
		01SB8-2*	-	Soil					s		s	s	s							
	01SB9	01SB9-1	-	Soil					s		s	s	s							
		01SB9-2*	-	Soil					s		s	s	s							
	01SB10	01SB10-1	-	Soil					s		s	s	s							
		01SB10-2*	-	Soil					s		s	s	s							
4 Pump House	04CP-1	04CP-1	-	Soil	Confirm soil concentrations in vicinity of abandoned drum(s)	16 point composite to be collected on a 10-foot grid around pump house with aliquots bias to drum locations. If drums are no longer present, the aliquots will be evenly spaced.	s	s			s	s	s	s	s						1, 4 oz amber glass 1, 4 oz clear glass 1, 40 mL VOA/methanol

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8 Incinerator #49	Incinerator floor debris/ equipment/stack	08W-1*	FD: 08W-10	Solid	To assess source area concentrations	Contents of stack			s		s	s			s			s		2, 8 oz amber glass 2, 4 oz amber glass
		08W-2*	-	Solid		Content of equipment or floor debris if no equipment present			s		s	s			s			s		1, 8 oz amber glass 1, 4 oz amber glass
		08W-3*	-	Solid					s		s	s			s			s		
		08W-4*	-	Solid					s		s	s			s			s		
	08DU1	08DU1-1	MS/MSD	Soil	To assess perimeter soil	ISM 08DU1 is depicted on Figure 4B east of the incinerator and will consist of 30 0 to 1-foot aliquots per replicate			s		s	s			s			s		3, 1 liter widemouth amber
		08DU1-2	-	Soil					s		s	s			s			s		1, 1 liter widemouth amber
		08DU1-3	-	Soil					s		s	s			s			s		
	08DU2	08DU2-1	-	Soil		ISM 08DU2 is depicted on Figure 4B north of the incinerator and will consist of 30 0 to 1-foot aliquots			s		s	s			s			s		
		08DU2-2	-	Soil					s		s	s			s			s		
		08DU2-3	-	Soil					s		s	s			s			s		
	08DU3	08DU3-1	-	Soil		ISM 08DU3 is depicted on Figure 4B south of the incinerator and will consist of 30 0 to 1-foot aliquots			s		s	s			s			s		
		08DU3-2	-	Soil					s		s	s			s			s		
		08DU3-3	-	Soil					s		s	s			s			s		
	08DU4	08DU4-1	-	Soil		ISM 08DU4 is depicted on Figure 4B west of the incinerator and will consist of 30 0 to 1-foot aliquots			s		s	s			s			s		
		08DU4-2	-	Soil					s		s	s			s			s		
		08DU4-3	-	Soil					s		s	s			s			s		
	08SB1	08SB1-1	FD:08SB1-10	Soil		Interval of observable contamination or 0-1 foot			s		s	s			s			s		2, 8 oz amber glass 2, 4 oz amber glass
	08SB2	08SB2-1	-	Soil					s		s	s			s			s		1, 8 oz amber glass 1, 4 oz amber glass
	08SB3	08SB3-1	-	Soil					s		s	s			s			s		
	08SB4	08SB4-1	-	Soil					s		s	s			s			s		
	08SB5	08SB5-1	MS/MSD	Soil					s		s	s			s			s		3, 8 oz amber glass 3, 4 oz amber glass
	08SB6	08SB6-1	-	Soil					s		s	s			s			s		1, 8 oz amber glass 1, 4 oz amber glass
	08SB7	08SB7-1	-	Soil					s		s	s			s			s		

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10 Disposal Area #14	10SB1	10SB1-1	-	Soil	Vertical delineation and characterization of waste	Continuous sampling in 2-foot interval through waste material.	s	s			s	s	s	s	s	s	s		s	1, 40 mL VOA/methanol 1, 4 oz amber glass 2, 4 oz clear glass 1, 8 oz amber glass
		10SB1-2*,^	-	Soil			s	s			s	s	s	s	s	s	s		s	
		10SB1-3*, ^	-	Soil			s	s			s	s	s	s	s	s	s		s	
		10SB1-4*,^	-	Soil		Greatest observed contamination beyond waste material, if any. If no evidence of contamination (e.g., PID response or free product) then no samples will be collected.	s	s			s	s	s	s	s	s	s		s	
		10SB1-5*,^	-	Soil		2-foot interval above bedrock refusal, if waste extends to bedrock no sample will be collected	s	s			s	s	s	s	s	s	s		s	
	10SB2/ 10OBMW1	10SB2-1	-	Soil		Continuous sampling in 2-foot interval through waste material.	s	s			s	s	s	s	s	s	s		s	2, 40 mL VOA/methanol 2, 4 oz amber glass 4, 4 oz clear glass 2, 8 oz amber glass
		10SB2-2*,^	FD: 10SB2-20	Soil			s	s			s	s	s	s	s	s	s		s	
		10SB2-3*, ^		Soil			s	s			s	s	s	s	s	s	s		s	1, 40 mL VOA/methanol 1, 4 oz amber glass 2, 4 oz clear glass 1, 8 oz amber glass
		10SB2-4*,^	-	Soil		Greatest observed contamination beyond waste material, if any. If no evidence of contamination (e.g., PID response or free product) then no samples will be collected.	s	s			s	s	s	s	s	s	s		s	
		10SB2-5*,^	-	Soil		2-foot interval above bedrock refusal, if waste extends to bedrock no sample will be collected	s	s			s	s	s	s	s	s	s		s	
		10OBMW1-mmyy	-	Groundwater	To assess overburden groundwater impacts in the disposal area body of fill	Overburden groundwater, if present	w	w		w	w	w	w	w		w	w			4, 40 mL VOA/HCl 1, 250 mL HDPE/HNO3, field filtered Plastic w/HexCr Buffer 1, 1 liter amber/HCl 5, 1 liter amber 1, 125mL
	10SB3	10SB3-1	-	Soil	Vertical delineation and characterization of waste	Continuous sampling in 2-foot interval through waste material.	s	s			s	s	s	s	s	s	s		s	1, 40 mL VOA/methanol 1, 4 oz amber glass 2, 4 oz clear glass 1, 8 oz amber glass
		10SB3-2*,^	-	Soil			s	s			s	s	s	s	s	s	s		s	
		10SB3-3*, ^	-	Soil			s	s			s	s	s	s	s	s	s		s	
		10SB3-4*,^	-	Soil		Greatest observed contamination beyond waste material, if any. If no evidence of contamination (e.g., PID response or free product) then no samples will be collected.	s	s			s	s	s	s	s	s	s		s	
		10SB3-5*,^	-	Soil		2-foot interval above bedrock refusal, if waste extends to bedrock no sample will be collected	s	s			s	s	s	s	s	s	s		s	
	10SB4/ 10OBMW2	10SB4-1	-	Soil		Continuous sampling in 2-foot interval through waste material.	s	s			s	s	s	s	s	s	s		s	
		10SB4-2*,^	-	Soil			s	s			s	s	s	s	s	s	s		s	
		10SB4-3*, ^	-	Soil			s	s			s	s	s	s	s	s	s		s	
		10SB4-4*,^	-	Soil		Greatest observed contamination beyond waste material, if any. If no evidence of contamination (e.g., PID response or free product) then no samples will be collected.	s	s			s	s	s	s	s	s	s		s	
		10SB4-5*,^	-	Soil		2-foot interval above bedrock refusal, if waste extends to bedrock no sample will be collected	s	s			s	s	s	s	s	s	s		s	
		10OBMW2-mmyy	-	Groundwater	To assess overburden groundwater impacts in the disposal area body of fill	Overburden groundwater, if present	w	w		w	w	w	w	w		w	w			4, 40 mL VOA/HCl 1, 250 mL HDPE/HNO_3, field filtered 1, 125mL Plastic w/HexCr Buffer, field filtered 1, 1 liter amber/HCl 5, 1 liter amber
	10SB5	10SB5-1	MS/MSD	Soil	Vertical delineation and characterization of waste	Continuous sampling in 2-foot interval through waste material.	s	s			s	s	s	s	s	s	s		s	3, 40 mL VOA/methanol 3, 4 oz amber glass 6, 4 oz clear glass 3, 8 oz amber glass
		10SB5-2*,^	-	Soil			s	s			s	s	s	s	s	s	s		s	1, 40 mL VOA/methanol 1, 4 oz amber glass 2, 4 oz clear glass 1, 8 oz amber glass
		10SB5-3*, ^	-	Soil			s	s			s	s	s	s	s	s	s		s	
		10SB5-4*,^	-	Soil		Greatest observed contamination beyond waste material, if any. If no evidence of contamination (e.g., PID response or free product) then no samples will be collected.	s	s			s	s	s	s	s	s	s		s	
		10SB5-5*,^	-	Soil		2-foot interval above bedrock refusal, if waste extends to bedrock no sample will be collected	s	s			s	s	s	s	s	s	s		s	

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10 Disposal Area #14 (cont.)	10TP1	10TP1-1	MS/MSD	Soil	Assessment of impacts to the beach and extent of previously observed tar-like material	One sample from each test pit from the greatest observed contamination (i.e., PID response, staining, etc.). In the absence of evidence of contamination, sample will be collected from 0 to 2 feet bgs	s	s			s	s	s+	s+	s	s	s		s	3, 40 mL VOA/methanol 3, 4 oz amber glass 6, 4 oz clear glass 3, 8 oz amber glass
	10TP2/10PZ1	10TP2-1	-	Soil			s	s			s	s	s+	s+	s	s	s		s	1, 40 mL VOA/methanol 1, 4 oz amber glass 2, 4 oz clear glass 1, 8 oz amber glass
		10PZ1-mmyy	-	Groundwater		Well screened across groundwater interface in observable tar stained area	w	w		w	w	w	w	w		w	w			4, 40 mL VOA/HCl 1, 250 mL HDPE/HNO_3, field filtered 1, 125mL Plastic w/HexCr Buffer, field filtered 1, 1 liter amber/HCl 5, 1 liter amber
	10TP3	10TP3-1	-	Soil		One sample from each test pit from the greatest observed contamination (i.e., PID response, staining, etc.). In the absence of evidence of contamination, sample will be collected from 0 to 2 feet bgs	s	s			s	s	s+	s+	s	s	s		s	1, 40 mL VOA/methanol 1, 4 oz amber glass 2, 4 oz clear glass 1, 8 oz amber glass
	10TP4	10TP4-1	FD: 10TP4-10	Soil			s	s			s	s	s+	s+	s	s	s		s	2, 40 mL VOA/methanol 2, 4 oz amber glass 4, 4 oz clear glass 2, 8 oz amber glass
	10TP5/10PZ-2	10TP5-1	-	Soil			s	s			s	s	s+	s+	s	s	s		s	1, 40 mL VOA/methanol 1, 4 oz amber glass 2, 4 oz clear glass 1, 8 oz amber glass
		10TP5-2	-	Soil		2-foot interval above refusal	s	s			s	s	s+	s+	s	s	s		s	
		10PZ2-mmyy	FD: 10PZ-20-mmyy	Groundwater		Well screened across groundwater interface in observable tar stained area	w	w		w	w	w	w	w		w	w			8, 40 mL VOA/HCl 2, 250 mL HDPE/HNO_3, field filtered 2, 125mL Plastic w/HexCr Buffer, field filtered 2, 1 liter amber/HCl 10, 1 liter amber
	10TP6	10TP6-1	-	Soil		One sample from each test pit from the greatest observed contamination (i.e., PID response, staining, etc.). In the absence of evidence of contamination, sample will be collected from 0 to 2 feet bgs	s	s			s	s	s+	s+	s	s	s		s	1, 40 mL VOA/methanol 1, 4 oz amber glass 2, 4 oz clear glass 1, 8 oz amber glass
	10TP7	10TP7-1	-	Soil			s	s			s	s	s+	s+	s	s	s		s	
	10TP8/10PZ3	10TP8-1	-	Soil			s	s			s	s	s+	s+	s	s	s		s	
		10TP8-2	-	Soil		2-foot interval above refusal	s	s			s	s	s+	s+	s	s	s		s	
		10PZ3-mmyy	-	Groundwater		Well screened across groundwater interface in observable tar stained area	w	w		w	w	w	w	w		w	w			4, 40 mL VOA/HCl 1, 250 mL HDPE/HNO_3, field filtered 1, 125mL Plastic w/HexCr Buffer, field filtered 1, 1 liter amber/HCl 5, 1 liter amber
	10TP9	10TP9-1	-	Soil		One sample from each test pit from the greatest observed contamination (i.e., PID response, staining, etc.). In the absence of evidence of contamination, sample will be collected from 0 to 2 feet bgs	s	s			s	s	s	s	s	s	s		s	1, 40 mL VOA/methanol 2, 4 oz amber glass 2, 4 oz clear glass 1, 8 oz amber glass
	10TP10	10TP10-1	-	Soil			s	s			s	s	s	s	s	s	s		s	
	10TP11	10TP11-1	-	Soil			s	s			s	s	s	s	s	s	s		s	
	10TP12/10PZ4	10TP12-1	-	Soil			s	s			s	s	s	s	s	s	s		s	
		10PZ4-mmyy	-	Groundwater		Well screened across groundwater interface in observable tar stained area	w	w		w	w	w	w	w		w	w			4, 40 mL VOA/HCl 1, 250 mL HDPE/HNO_3, field filtered 1, 125mL Plastic w/HexCr Buffer, field filtered 1, 1 liter amber/HCl 5, 1 liter amber
	10TP13	10TP13-1	-	Soil		One sample from each test pit from the greatest observed contamination (i.e., PID response, staining, etc.). In the absence of evidence of contamination, sample will be collected from 0 to 2 feet bgs	s	s			s	s	s	s	s	s	x		x	1, 40 mL VOA/methanol 1, 4 oz amber glass 2, 4 oz clear glass 1, 8 oz amber glass

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10 Disposal Area #14 (cont.)	10SD1	10SD1	-	Sediment	Assess the extent of previously confirmed impacts to sediment	To be collected during low tide from 0-6 inches	s	s		s	s	s	s	s	s	s	s		s	1, 40 mL VOA, methanol 2, 4 oz amber glass 1, 8 oz amber glass 2, 4 oz glass
	10SD2	10SD2	MS/MSD	Sediment			s	s		s	s	s	s	s	s	s	s		s	3, 40 mL VOA, methanol 6, 4 oz amber glass 3, 8 oz amber glass 6, 4 oz glass
	10SD3	10SD3	-	Sediment			s	s		s	s	s	s	s	s	s	s		s	
	10SD4	10SD4S	-	Sediment		S samples to be collected during low tide from 0-6 inches, D samples will be collected from the greatest observed contamination within the top 4 feet	s	s		s	s	s	s	s	s	s	s		s	1, 40 mL VOA, methanol 2, 4 oz amber glass 1, 8 oz amber glass 2, 4 oz glass
		10SD4D	-	Sediment			s	s		s	s	s	s	s	s	s	s		s	
	10SD5	10SD5S	-	Sediment			s	s		s	s	s	s	s	s	s	s		s	
		10SD5D	-	Sediment			s	s		s	s	s	s	s	s	s	s		s	
	10SD6	10SD6S	-	Sediment			s	s		s	s	s	s	s	s	s	s		s	
		10SD6D	-	Sediment			s	s		s	s	s	s	s	s	s	s		s	
	10SD7	10SD7S	-	Sediment			s	s		s	s	s	s	s	s	s	s		s	
		10SD7D	FD: 10SD70D	Sediment			s	s		s	s	s	s	s	s	s	s		s	2, 40 mL VOA, methanol 4, 4 oz amber glass 2, 8 oz amber glass 4, 4 oz glass
	10SD8	10SD8	-	Sediment		To be collected during low tide from 0-6 inches	s	s		s	s	s	s	s	s	s	s		s	1, 40 mL VOA, methanol 1, 4 oz amber glass 1, 8 oz amber glass 1, 4 oz glass
	10SD9	10SD9	-	Sediment		To be collected with ponar sampler from landing craft	s	s		s	s	s	s	s	s	s	s		s	1, 40 mL VOA, methanol 1, 4 oz amber glass 1, 8 oz amber glass 1, 4 oz glass
	10SD10	10SD10	-	Sediment			s	s		s	s	s	s	s	s	s	s		s	
	10SD11	10SD11	-	Sediment			s	s		s	s	s	s	s	s	s	s		s	
	10SD12	10SD12	-	Sediment			s	s		s	s	s	s	s	s	s	s		s	
	10SD13	10SD13	-	Sediment			s	s		s	s	s	s	s	s	s	s		s	
	10SD14	10SD14	-	Sediment			s	s		s	s	s	s	s	s	s	s		s	
	10SD15	10SD15	-	Sediment			s	s		s	s	s	s	s	s	s	s		s	

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11 Quonset/ Maintenance Shops	11DU1	11DU1-1	-	Soil	Assessment of heating and maintenance related impacts	ISM 11DU1 shown on Figure 4A and will consist of 50 0 to 1-foot aliquots per replicate		s			s	s	s							1, 1 liter widemouth amber
		11DU1-2	-	Soil				s			s	s	s							
		11DU1-3	-	Soil				s			s	s	s							
		11SS-1	-	Soil		Grab sample from three aliquot locations showing evidence of contamination (i.e., staining, PID response, presence of drums/containers in vicinity, etc.)	s	s			s	s	s	s						1, 40mL VOA/methanol 1, 4 oz amber glass 1, 4 oz clear glass
		11SS-2	-	Soil			s	s			s	s	s	s						
		11SS-3	-	Soil			s	s			s	s	s	s						
12 Maintenance Shop/ Garage Fire Station	Spilled Transformer	12SS-1	FD: 12SS-10	Soil	Assessment of extent of spilled transformer contents	A 3 meter grid will be used to collect 9 soil/debris (0-0.5 feet) and 9 concrete samples (0-0.5 inches) around the spilled transformer contents in accordance with 40 CFR 761.265							s	s	s**						2, 40 mL VOA/methanol 2, 4 oz. amber glass
		12CC-1	-	Concrete											s**					1, 4 oz. clear glass, 20 oz sample
		12SS-2	-	Soil									s	s	s**					1, 40 mL VOA/methanol 1, 4 oz. amber glass
		12CC-2	-	Concrete											s**					1, 4 oz. clear glass, 20 oz sample
		12SS-3	-	Soil									s	s	s**					1, 40 mL VOA/methanol 1, 4 oz. amber glass
		12CC-3	-	Concrete											s**					1, 4 oz. clear glass, 20 oz sample
		12SS-4	-	Soil									s	s	s**					1, 40 mL VOA/methanol 1, 4 oz. amber glass
		12CC-4	-	Concrete											s**					1, 4 oz. clear glass, 20 oz sample
		12SS-5	-	Soil									s	s	s**					1, 40 mL VOA/methanol 1, 4 oz. amber glass
		12CC-5	-	Concrete											s**					1, 4 oz. clear glass, 20 oz sample
		12SS-6	-	Soil									s	s	s**					1, 40 mL VOA/methanol 1, 4 oz. amber glass
		12CC-6	-	Concrete											s**					1, 4 oz. clear glass, 20 oz sample
		12SS-7	-	Soil									s	s	s**					1, 40 mL VOA/methanol 1, 4 oz. amber glass
		12CC-7	-	Concrete											s**					1, 4 oz. clear glass, 20 oz sample
		12SS-8	-	Soil									s	s	s**					1, 40 mL VOA/methanol 1, 4 oz. amber glass
		12CC-8	-	Concrete											s**					1, 4 oz. clear glass, 20 oz sample
		12SS-9	MS/MSD	Soil									s	s	s**					3, 40 mL VOA/methanol 3, 4 oz. amber glass
		12CC-9	-	Concrete											s**					1, 4 oz. clear glass, 20 oz sample
	12SB1/ 12OBMW1	12SB1-1	-	Soil	To assess impacts from the vehicle maintenance pit	Sample from greatest observed contamination or groundwater interface in absence of contamination	s	s			s	s	s	s	s					1, 40mL VOA/methanol 1, 4 oz amber glass 1, 4 oz clear glass
		12OBMW1-mmyy	-	Groundwater		To be screened across the groundwater interface	w	w		w	w	w	w	w	w					4, 40 mL VOA/HCl 1, 250 mL HDPE/HNO_3, field filtered 1, 125mL Plastic w/HexCr Buffer, field filtered 1, 1 liter amber/HCl 3, 1 liter amber
	Maintenance pit sludge/debris	12W-1	-	Solid		Three samples to be collected from sludge or debris at base of maintenance pit.	s	s			s	s	s	s	s					1, 40mL VOA/methanol 1, 4 oz amber glass 1, 4 oz clear glass
		12W-2	-	Solid			s	s			s	s	s	s	s					
		12W-3	-	Solid			s	s			s	s	s	s	s					
	12SB2/ 12OBMW2	12SB2-1	-	Soil	To assess around suspected UST	Sample collected from greatest observed contamination or groundwater interface in absence of contamination	s	s			s	s	s	s	s					
		12OBMW2-mmyy	-	Groundwater		To be screened across the groundwater interface	w	w		w	w	w	w	w	w					4, 40 mL VOA/HCl 1, 250 mL HDPE/HNO_3, field filtered 1, 125mL Plastic w/HexCr Buffer, field filtered 1, 1 liter amber/HCl 3, 1 liter amber
	12SB3	12SB3-1	-	Soil		Sample collected from greatest observed contamination or groundwater interface in absence of contamination	s	s			s	s	s	s	s					1, 40mL VOA/methanol 1, 4 oz amber glass 1, 4 oz clear glass
	12SB4	12SB4-1	-	Soil			s	s			s	s	s	s	s					

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13 Electric Substation/ Transformer Pad	Concrete pad	13CC-1	-	Concrete	To assess impacts to concrete transformer pad	Top 0.5 inch of concrete transformer pad. Sample locations biased to areas of staining.									s**					1, 4 oz. clear glass, 20 oz sample
		13CC-2	-	Concrete											s**					
		13CC-3	-	Concrete											s**					
		13CC-4	-	Concrete											s**					
	Soil surrounding pad	13SS-3	-	Soil	To assess impacts surrounding transformer pad	Top 6 inches on each side of the transformer pad					s	s	s	s	s					1, 40 mL VOA/methanol 1, 4 oz. clear glass 1, 4 oz amber glass
		13SS-4	-	Soil							s	s	s	s	s					
		13SS-5	-	Soil							s	s	s	s	s					
		13SS-6	-	Soil							s	s	s	s	s					
16 Barracks	Buried Transformer Area	16T-1	FD: 16T-10	Soil	To assess beneath each transformer	Beneath each identified transformer removed from area. Additional samples may be warranted based on number of transformers identified. Samples biased to areas of staining.							s	s	s**					2, 40 mL VOA/methanol 2, 4 oz amber glass
		16T-2	-	Soil									s	s	s**					1, 40 mL VOA/methanol 1, 4 oz amber glass
		16T-3*	-	Soil									s	s	s**					
		16SS-1*	-	Soil	Assessment of extent of spilled transformer contents	Contingent on evidence of a release from buried transformers. A 3 meter grid will be used to collect 16 soil samples around the spilled transformer contents in accordance with 40 CFR 761.265							s	s	s**					
		16SS-2*	-	Soil									s	s	s**					
		16SS-3*	-	Soil									s	s	s**					
		16SS-4*	-	Soil									s	s	s**					
		16SS-5*	-	Soil									s	s	s**					
		16SS-6*	-	Soil									s	s	s**					
		16SS-7*	-	Soil									s	s	s**					
		16SS-8*	-	Soil									s	s	s**					
		16SS-9*	-	Soil									s	s	s**					
		16SS-10*	-	Soil									s	s	s**					
		16SS-11*	-	Soil									s	s	s**					
		16SS-12*	MS/MSD	Soil									s	s	s**					3, 40 mL VOA/methanol 3, 4 oz amber glass
		16SS-13*	-	Soil									s	s	s**					1, 40 mL VOA/methanol 1, 4 oz amber glass
		16SS-14*	-	Soil									s	s	s**					
		16SS-15*	-	Soil									s	s	s**					
		16SS-16*	-	Soil									s	s	s**					
19 Torch Pot Storage #51	19DU1	19DU1-1	FD: 19DU1-10	Soil	Confirm soil concentrations in vicinity of drum	ISM 19DU1 shown on Figure 4A and will consist of 30 0 to 1-foot aliquots per replicate							s							2, 1 liter widemouth amber
		19DU1-2	MS/MSD	Soil									s							3, 1 liter widemouth amber
		19DU1-3	-	Soil									s							1, 1 liter widemouth amber
21 Bunker #11	Within bunker #11	21SS-3*	-	Soil	To assess within bunker for impacts associated with use as a firing range and possible releases from stored drums within the bunker	To be collected of floor debris or 0-2 feet bgs within bunker depending if there is a concrete floor. SS-3 through SS-5 will be biased to firing target end of the bunker (i.e., east or west end). Samples contingent upon removal of collapsed bunker roof and removal of other hazards identified beneath collapsed debris.	s	s			s	s	s	s	s	s	s			1, 40 mL VOA/methanol 1, 4 oz amber glass 2, 4 oz clear glass 1, 8 oz amber glass
		21SS-4*	-	Soil			s	s			s	s	s	s	s	s	s			
		21SS-5*	-	Soil			s	s			s	s	s	s	s	s	s			
		21SS-6*	-	Soil			s	s			s	s	s	s	s	s	s			
		21SS-7*	-	Soil			s	s			s	s	s	s	s	s	s			
	21SB1/ 21OBMW1	21SB1-1	FD: 21SB1-10	Soil	To assess migration of contaminants possibly released to bunker	One sample from each boring from the greatest observed contamination (i.e., PID response, staining, etc.). In the absence of contamination, sample to be collected from groundwater interface or 2 foot interval above refusal.	s	s			s	s	s	s	s	s	s			2, 40 mL VOA/methanol 2, 4 oz amber glass 4, 4 oz clear glass 2, 8 oz amber glass
		21OBMW1-mmyy	-	Groundwater		To be screened across the overburden groundwater interface and to refusal. Screen may exceed 10 feet.	w	w		w	w	w	w	w	w	w	w			4, 40 mL VOA/HCl 1, 250 mL HDPE/HNO3, field filtered 1, 125mL Plastic w/HexCr Buffer, field filtered 1, 1 liter amber/HCl 6, 1 liter amber
	21SB2	21SB2-1	-	Soil		One sample from each boring from the greatest observed contamination (i.e., PID response, staining, etc.). In the absence of contamination, sample to be collected from groundwater interface or 2 foot interval above refusal.	s	s			s	s	s	s	s	s	s			1, 40 mL VOA/methanol 1, 4 oz amber glass 2, 4 oz clear glass 1, 8 oz amber glass
	21SB3	21SB3-1	-	Soil			s	s			s	s	s	s	s	s	s			
	21BS4	21SB4-1	-	Soil			s	s			s	s	s	s	s	s	s			
	21SB5	21SB-5-1	MS/MSD	Soil			s	s			s	s	s	s	s	s	s			3, 40 mL VOA/methanol 3, 4 oz amber glass 6, 4 oz clear glass 3, 8 oz amber glass
	21SB6	21SB-6-1	-	Soil			s	s			s	s	s	s	s	s	s			1, 40 mL VOA/methanol 1, 4 oz amber glass 2, 4 oz clear glass 1, 8 oz amber glass

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22 Bunker #12	Within bunker #12	22SS-3*	FD: 22SS-30	Soil	To assess within the bunker for impacts associated with possible release from drums stored within the bunker.	To be collected of floor debris or 0-2 feet bgs within bunker depending if there is a concrete floor. Samples contingent upon removal of collapsed bunker roof and removal of other hazards identified beneath collapsed debris.	s	s			s	s	s	s	s	s	s			2, 40 mL VOA/methanol 2, 4 oz amber glass 4, 4 oz clear glass 2, 8 oz amber glass
		22SS-4*	-	Soil			s	s			s	s	s	s	s	s	s			1, 40 mL VOA/methanol 1, 4 oz amber glass 2, 4 oz clear glass 1, 8 oz amber glass
		22SS-5*	-	Soil			s	s			s	s	s	s	s	s	s			
	22SB1/ 22OBMW1	22SB1-1	-	Soil	To assess migration of contaminants possibly released to bunker	One sample from each boring from the greatest observed contamination (i.e., PID response, staining, etc.). In the absence of contamination, sample to be collected from groundwater interface or 2 foot interval above refusal.	s	s			s	s	s	s	s	s	s			
		22OBMW1-mmyy	-	Groundwater		To be screened across the overburden groundwater interface and to refusal. Screen may exceed 10 feet.	w	w		w	w	w	w	w	w	w	w			4, 40 mL VOA/HCl 1, 250 mL HDPE/HNO3, field filtered 1, 125mL Plastic w/HexCr Buffer, field filtered 1, 1 liter amber/HCl 6, 1 liter amber
	22SB2	22SB2-1	-	Soil		One sample from each boring from the greatest observed contamination (i.e., PID response, staining, etc.). In the absence of contamination, sample to be collected from groundwater interface or 2 foot interval above refusal.	s	s			s	s	s	s	s	s	s			1, 40 mL VOA/methanol 1, 4 oz amber glass 2, 4 oz clear glass 1, 8 oz amber glass
	22SB3	22SB3-1	-	Soil			s	s			s	s	s	s	s	s	s			
	22SB4	22SB4-1	-	Soil			s	s			s	s	s	s	s	s	s			
	22SB5	22SB5-1	-	Soil			s	s			s	s	s	s	s	s	s			
	22SB6	22SB6-1	MS/MSD	Soil			s	s			s	s	s	s	s	s	s			3, 40 mL VOA/methanol 3, 4 oz amber glass 6, 4 oz clear glass 3, 8 oz clear glass
23 Coal Storage South	23DU1	23DU1-1	-	Soil	Nature and extent of previously identified surface contamination	Source Unit. 23DU1 is depicted on Figure 4A and will consist of 50 0 to 1-foot aliquots per replicate			s		s	s	s							1, 1 liter widemouth amber
		23DU1-2	-	Soil					s		s	s	s							
		23DU1-3	-	Soil					s		s	s	s							
	23DU2	23DU2-1	MS/MSD	Soil		ISM 23DU2 is depicted on Figure 4A and will consist of 50 0 to 1-foot aliquots per replicate			s		s	s	s							3, 1 liter widemouth amber
		23DU2-2	-	Soil					s		s	s	s							1, 1 liter widemouth amber
		23DU2-3	-	Soil					s		s	s	s							
	23SB1	23SB1-1	-	Soil	Horizontal and vertical extent of coal in soil by visual examination	Sample to be collected from the entire thickness of observed coal up to a 4 foot interval (e.g., 0 to 4 feet bgs). If greater than 4 feet of coal is encountered the -2 samples will be collected (e.g., 4 to 8, 4 to 6 feet bgs, etc.).			s		s	s	s							2, 4 oz amber glass
		23SB1-2*	-	Soil					s		s	s	s							
	23SB2	23SB2-1	FD: 23SB2-10	Soil					s		s	s	s							4, 4 oz amber glass
		23SB2-2*	-	Soil					s		s	s	s							2, 4 oz amber glass
	23SB3	23SB3-1	-	Soil					s		s	s	s							
		23SB3-2*	-	Soil					s		s	s	s							
	23SB4	23SB4-1	FD: 23SB4-10	Soil					s		s	s	s							4, 4 oz amber glass
		23SB4-2*	-	Soil					s		s	s	s							2, 4 oz amber glass
	23SB5	23SB5-1	-	Soil					s		s	s	s							
		23SB5-2*	-	Soil					s		s	s	s							
	23SB6	23SB6-1	-	Soil					s		s	s	s							
		23SB6-2*	-	Soil					s		s	s	s							
	23SB7	23SB7-1	-	Soil					s		s	s	s							
		23SB7-2*	-	Soil					s		s	s	s							
	23SB8	23SB8-1	-	Soil					s		s	s	s							
		23SB8-2*	-	Soil					s		s	s	s							

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24 Cable Terminal Bldg #16	Transformer pad	24CC-1	FD: 24CC-10	Concrete	Assess impacts from former transformer	Top 0.5 inch of concrete transformer pad. Sample location biased to area of staining.									s**					2, 4 oz. clear glass, 20 oz sample
	Soil surrounding pad	24SS-2	-	Soil		Top 6 inches on each side of the transformer pad							s	s	s**					1, 40 mL VOA/methanol 1, 4 oz. amber glass
		24SS-3	-	Soil									s	s	s**					
		24SS-4	-	Soil									s	s	s**					
		24SS-5	-	Soil									s	s	s**					
27 & 31 Three Gas Pits	Gas pits 27	27SL-1	FD: 27SL-10	Sludge	Assess contents of gas pits	Sludge grab sample from each of three gas pits. Two 27 pits located on east side of concrete pad, one 31 pit located on west side of pad.	s	s			s	s	s	s	s					2, 40 mL VOA/methanol 2, 4 oz amber glass 2, 4 oz glass
		27SL-2	-	Sludge			s	s			s	s	s	s	s					1, 40 mL VOA/methanol 1, 4 oz amber glass 1, 4 oz glass
	Gas pit 31	31SL-1	-	Sludge			s	s			s	s	s	s	s					
	27SB1/ 27OBMW1	27SB1-1	-	Soil	Assess possible release to subsurface	One sample from each boring from the greatest observed contamination (i.e., PID response, staining, etc.). In the absence of contamination, sample to be collected from groundwater interface or 2 foot interval above refusal.	s	s			s	s	s	s	s					
		27OBMW1-mmyy	MS/MSD	Groundwater	Assess impacts from gas pits as well as other NED Sites within the general aviation pad	Screened at groundwater interface above high tide	w	w		w	w	w	w	w	w		w			12, 40 mL VOA/HCl 3, 250 mL HDPE/HNO3, field filtered 3, 125mL Plastic w/HexCr Buffer, field filtered 3, 1 liter amber/HCl 15, 1 liter amber
	27SB2	27SB2-1	MS/MSD	Soil	Assess possible release to subsurface	One sample from each boring from the greatest observed contamination (i.e., PID response, staining, etc.). In the absence of contamination, sample to be collected from groundwater interface or 2 foot interval above refusal.	s	s			s	s	s	s	s					3, 40 mL VOA/methanol 3, 4 oz amber glass 3, 4 oz clear glass
	31SB1/ 31OBMW1	31SB1-1	-	Soil			s	s			s	s	s	s	s					1, 40 mL VOA/methanol 1, 4 oz amber glass 1, 4 oz glass
		31OBMW1-mmyy	-	Groundwater	Assess impacts from gas pits as well as other NED Sites within the general aviation pad	Screened at groundwater interface above high tide	w	w		w	w	w	w	w	w		w			4, 40 mL VOA/HCl 1, 250 mL HDPE/HNO3, field filtered 1, 125mL Plastic w/HexCr Buffer, field filtered 1, 1 liter amber/HCl 5, 1 liter amber
28, 30, 32, 33, 43 & 44 General Aviation Pad	APSB1	APSB1-1	-	Soil	Generally assess soil beneath the concrete aviation pad	2-foot interval below concrete pad	s	s			s	s	s	s	s		s			1, 40 mL VOA/methanol 1, 4 oz amber glass 1, 4 oz glass 1, 8 oz amber glass
	APSB2	APSB2-1	-	Soil			s	s			s	s	s	s	s		s			
	APSB3	APSB3-1	-	Soil			s	s			s	s	s	s	s		s			
	APSB4	APSB4-1	-	Soil			s	s			s	s	s	s	s		s			
	APSB5	APSB5-1	-	Soil			s	s			s	s	s	s	s		s			
	APSB6	APSB6-1	-	Soil			s	s			s	s	s	s	s		s			
	APSB7	APSB7-1	-	Soil			s	s			s	s	s	s	s		s			
	APSB8	APSB8-1	-	Soil			s	s			s	s	s	s	s		s			
	APSB9	APSB9-1	-	Soil			s	s			s	s	s	s	s		s			
	APSB10	APSB10-1	-	Soil			s	s			s	s	s	s	s		s			
	APSB11	APSB11-1	-	Soil			s	s			s	s	s	s	s		s			
	APSB12	APSB12-1	-	Soil			s	s			s	s	s	s	s		s			
	30SB1/ 30OBMW1	30SB1-1	-	Soil	To assess subsurface conditions downgradient of pad	One sample from each boring from the greatest observed contamination (i.e., PID response, staining, etc.). In the absence of contamination, sample to be collected from groundwater interface or 2 foot interval above refusal.	s	s			s	s	s	s	s		s			1, 40 mL VOA/methanol 1, 4 oz amber glass 1, 4 oz clear glass 1, 8 oz amber glass
		30OBMW1-mmyy	-	Groundwater		Screened at groundwater interface above high tide	w	w		w	w	w	w	w	w		w			4, 40 mL VOA/HCl 1, 250 mL HDPE/HNO3, field filtered 1, 125mL Plastic w/HexCr Buffer, field filtered 1, 1 liter amber/HCl 5, 1 liter amber
	32SB1	32SB1-1	-	Soil		One sample from each boring from the greatest observed contamination (i.e., PID response, staining, etc.). In the absence of contamination, sample to be collected from groundwater interface or 2 foot interval above refusal.	s	s			s	s	s	s	s		s			1, 40 mL VOA/methanol 1, 4 oz amber glass 1, 4 oz clear glass 1, 8 oz amber glass

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NED Site	Sample Location	Sample ID	Associated QC Sample Type/ID	Sample Type	Justification	Target Depth/Methodology	Analyses: VOCs: EPA Method 8260C	SVOCs: EPA Method 8270D	PAHs by EPA Method 8270D	PAHs by EPA Method 8270D-SIM	PP Metals EPA Method 6020A & 7471B/7470A [2]	Hexavalent Chromium EPA Method 7196A	EPH by MADEP EPH-04-1.1	VPH by MADEP VPH-04-1.1	PCBs by EPA Method 8082A	Pesticides by EPA Method 8081B	Explosives EPA method 8330B [1]	Dioxin/furan EPA Method 1613B	pH EPA Method 9045C	Sample Volume and Containers/ Preservative (per sample)
35 Boiler House #29	Stack debris	35W-1	-	Ash/debris	Assess contents of stack and fire boxes to characterize source	Sample ash and debris within stack and fire boxes. Sampling contingent upon safe access to these contents. Number of samples dependent on number of ash containing pieces of equipment remaining.			s		s	s					s			1, 4 oz amber glass 1, 8 oz amber glass
	Fire box	35W-2	-						s		s	s					s			
		35W-3	-						s		s	s					s			
		35W-4	-						s		s	s					s			
	35DU1	35DU1-1	FD: 35DU1-10	Soil/coal	To generally assess surface disposal of coal ash surrounding the boiler house	35DU1 is depicted on Figure 4C and will consist of 30 0 to 1-foot aliquots per replicate			s		s	s					s			2, 1 liter widemouth amber
		35DU1-2	-	Soil/coal					s		s	s					s			1, 1 liter widemouth amber
		35DU1-3	-	Soil/coal					s		s	s					s			
	35DU2	35DU2-1	-	Soil/coal		35DU2 is depicted on Figure 4C and will consist of 30 0 to 1-foot aliquots per replicate			s		s	s					s			
		35DU2-2	-	Soil/coal					s		s	s					s			
		35DU2-3	-	Soil/coal					s		s	s					s			
	35DU3	35DU3-1	-	Soil/coal		35DU3 is depicted on Figure 4C and will consist of 30 0 to 1-foot aliquots per replicate			s		s	s					s			
		35DU3-2	-	Soil/coal					s		s	s					s			
		35DU3-3	-	Soil/coal					s		s	s					s			
	35DU4	35DU4-1	MS/MSD	Soil/coal		35DU4 is depicted on Figure 4C and will consist of 30 0 to 1-foot aliquots per replicate			s		s	s					s			3, 1 liter widemouth amber
		35DU4-2	-	Soil/coal					s		s	s					s			1, 1 liter widemouth amber
		35DU4-3	-	Soil/coal					s		s	s					s			
	35SB1	35SB1-1	FD: 35SB1-10	Soil/coal	To vertically assess coal ash disposal around and downslope of the boiler house	Sample to be collected from the entire thickness of observed coal up to a 4 foot interval (e.g., 0 to 4 feet bgs). If greater than 4 feet of coal is encountered the -2 samples will be collected (e.g., 4 to 8, 4 to 6 feet bgs, etc.).			s		s	s					s			2, 4 oz amber glass 2, 8 oz amber glass
		35SB1-2*	-	Soil/coal					s		s	s					s			1, 4 oz amber glass 1, 8 oz amber glass
	35SB2	35SB2-1	-	Soil/coal					s		s	s					s			
		35SB2-2*	-	Soil/coal					s		s	s					s			
	35SB3	35SB3-1	-	Soil/coal					s		s	s					s			
		35SB3-2*	-	Soil/coal					s		s	s					s			
	35SB4	35SB4-1	-	Soil/coal					s		s	s					s			
		35SB4-2*	-	Soil/coal					s		s	s					s			
	35SB5	35SB5-1	-	Soil/coal					s		s	s					s			
		35SB5-2*	-	Soil/coal					s		s	s					s			
	35SB6	35SB6-1	-	Soil/coal					s		s	s					s			
		35SB6-2*	-	Soil/coal					s		s	s					s			
	35SB7	35SB7-1	MS/MSD	Soil/coal					s		s	s					s			3, 4 oz amber glass 3, 8 oz amber glass
		35SB7-2*	-	Soil/coal					s		s	s					s			1, 4 oz amber glass 1, 8 oz amber glass
	35SB8	35SB8-1	-	Soil/coal					s		s	s					s			
		35SB8-2*	-	Soil/coal					s		s	s					s			
	35SB9	35SB9-1	-	Soil/coal					s		s	s					s			
		35SB9-2*	-	Soil/coal					s		s	s					s			
	35SB10	35SB10-1	-	Soil/coal					s		s	s					s			
		35SB10-2*	-	Soil/coal					s		s	s					s			
	35SB11	35SB11-1	-	Soil/coal					s		s	s					s			
		35SB11-2*	-	Soil/coal					s		s	s					s			
37 Misc. Storage Bldg	37SB-1/ 37OBMW1	37SB1-1	-	Soil	Assess possible release from tank of unknown use	Greatest observed contamination (i.e., PID response, staining, etc.). In the absence of contamination, sample to be collected from groundwater interface or 2 foot interval above refusal.	s	s			s	s	s	s	s						1, 40 mL VOA/methanol 1, 4 oz amber glass 1, 4 oz clear glass
		37OBMW1-mmyy	-	Groundwater		Screened at the groundwater interface	w	w		w	w	w	w	w	w						4, 40 mL VOA/HCl 1, 250 mL HDPE/HNO3, field filtered 1, 125mL Plastic w/HexCr Buffer, field filtered 1, 1 liter amber/HCl 3, 1 liter amber
	37SB2	37SB2-1	-	Soil		Greatest observed contamination (i.e., PID response, staining, etc.). In the absence of contamination, sample to be collected from groundwater interface or 2 foot interval above refusal.	s	s			s	s	s	s	s						1, 40 mL VOA/methanol 1, 4 oz amber glass 1, 4 oz clear glass

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38 Torpedo Storage #10	Vicinity of spilled transformer	38SS-1	FD: 38SS-10	Soil	Assessment of extent of spilled transformer contents	A 3 meter grid will be used to collect 9 soil samples around the spilled transformer contents in accordance with 40 CFR 761.265. Concrete samples contingent upon underlying concrete.							s	s	s**					2, 40 mL VOA/methanol 2, 4 oz. amber glass
		38CC-1*	-	Concrete											s**					1, 4 oz. clear glass, 20 oz sample
		38SS-2	-	Soil									s	s	s**					1, 40 mL VOA/methanol 1, 4 oz. amber glass
		38CC-2*	-	Concrete											s**					1, 4 oz. clear glass, 20 oz sample
		38SS-3	-	Soil									s	s	s**					1, 40 mL VOA/methanol 1, 4 oz. amber glass
		38CC-3*	-	Concrete											s**					1, 4 oz. clear glass, 20 oz sample
		38SS-4	-	Soil									s	s	s**					1, 40 mL VOA/methanol 1, 4 oz. amber glass
		38CC-4*	-	Concrete											s**					1, 4 oz. clear glass, 20 oz sample
		38SS-5	-	Soil									s	s	s**					1, 40 mL VOA/methanol 1, 4 oz. amber glass
		38CC-5*	-	Concrete											s**					1, 4 oz. clear glass, 20 oz sample
		38SS-6	-	Soil									s	s	s**					1, 40 mL VOA/methanol 1, 4 oz. amber glass
		38CC-6*	-	Concrete											s**					1, 4 oz. clear glass, 20 oz sample
		38SS-7	-	Soil									s	s	s**					1, 40 mL VOA/methanol 1, 4 oz. amber glass
		38CC-7*	-	Concrete											s**					1, 4 oz. clear glass, 20 oz sample
		38SS-8	MS/MSD	Soil									s	s	s**					3, 40 mL VOA/methanol 3, 4 oz. amber glass
		38CC-8*	-	Concrete											s**					1, 4 oz. clear glass, 20 oz sample
		38SS-9	-	Soil									s	s	s**					1, 40 mL VOA/methanol 1, 4 oz. amber glass
		38CC-9*	-	Concrete											s**					1, 4 oz. clear glass, 20 oz sample
39 Well House #81	Transformer pad	39CC-1	-	Concrete	Assess impacts from former transformer	Sample top 0.5 inch of concrete transformer pad. Sample location biased to area of staining.									s**					1, 4 oz. clear glass, 20 oz sample
	Soil surrounding pad	39SS-2	-	Soil		Top 6 inches on each side of the transformer pad							s	s	s**					1, 40 mL VOA/methanol 1, 4 oz. amber glass
		39SS-3	-	Soil									s	s	s**					
		39SS-4	-	Soil									s	s	s**					
		39SS-5	-	Soil									s	s	s**					
42 5,000-gallon Tank (ad Gas) Ordnance test Facility Gasoline Outlet #30	42SB1	42SB1-1	-	Soil	Assess the location of former tank	-1 will be collected from 0-2 feet. -2 sample from each boring from the greatest observed contamination (i.e., PID response, staining, etc.). In the absence of contamination, sample to be collected from groundwater interface or 2 foot interval above refusal.	s	s			s	s	s	s	s					1, 40 mL VOA/methanol 2, 4 oz amber glass 1, 4 oz clear glass
		42SB1-2	-	Soil			s	s			s	s	s	s	s					
	42SB2	42SB2-1	-	Soil			s	s			s	s	s	s	s					
		42SB2-2	-	Soil			s	s			s	s	s	s	s					
	42SB2/ 42OBMW1	42OBMW1-mmyy	-	Groundwater		Screened at the groundwater interface	w	w		w	w	w	w	w	w					4, 40 mL VOA/HCl 1, 250 mL HDPE/HNO3, field filtered 1, 125mL Plastic w/HexCr Buffer, field filtered 1, 1 liter amber/HCl 3, 1 liter amber
	42SB3	42SB3-1	-	Soil		-1 will be collected from 0-2 feet. -2 sample from each boring from the greatest observed contamination (i.e., PID response, staining, etc.). In the absence of contamination, sample to be collected from groundwater interface or 2 foot interval above refusal.	s	s			s	s	s	s	s					1, 40 mL VOA/methanol 2, 4 oz amber glass 1, 4 oz clear glass
		42SB3-2	-	Soil			s	s			s	s	s	s	s					
	42SB4	42SB4-1	-	Soil			s	s			s	s	s	s	s					
		42SB4-2	-	Soil			s	s			s	s	s	s	s					
	42SB5	42SB5-1	-	Soil			s	s			s	s	s	s	s					
		42SB5-2	-	Soil			s	s			s	s	s	s	s					
9 & 48 Debris Stockpile and Magazine Ignitor Storage	48DU1	48DU1-1	-	Soil	Assess possible landfill or stockpiling	48DU1 is depicted on Figure 4A and will consist of 50 0 to 1-foot aliquots per replicate		s			s	s	s		s		s			1, 1 liter widemouth amber
		48DU1-2	-	Soil				s			s	s	s		s		s			
		48DU1-3	-	Soil				s			s	s	s		s		s			
	48SB1	48SB1-1	-	Soil		Sample from each boring from the greatest observed contamination (i.e., PID response, staining, etc.). In the absence of contamination, sample to be collected from groundwater interface or 2 foot interval above refusal.	s	s			s	s	s	s	s		s			1, 40 mL VOA/methanol 2, 4 oz amber glass 1, 4 oz clear glass 1, 8 oz amber glass
	48SB2	48SB2-1	-	Soil			s	s			s	s	s	s	s		s			

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Sitewide	BRMW-1	BRMW-1-mmyy	FD: BRMW-10-mmyy	Groundwater	To assess the bedrock aquifer in the bay ward sides of the island to intercept any possible contamination migrating into the bay at depth	To be screened in a bedrock water bearing zone.	w	w		w	w	w	w	w	w		w			8, 40 mL VOA/HCl 2, 250 mL HDPE/HNO_3, field filtered 2, 125mL Plastic w/HexCr Buffer, field filtered 2, 1 liter amber/HCl 10, 1 liter amber
	BRMW-2	BRMW-2-mmyy	-	Groundwater			w	w		w	w	w	w	w	w		w			4, 40 mL VOA/HCl 1, 250 mL HDPE/HNO3, field filtered 1, 125mL Plastic w/HexCr Buffer, field filtered 1, 1 liter amber/HCl 5, 1 liter amber
	BRMW-3	BRMW-3-mmyy	-	Groundwater			w	w		w	w	w	w	w	w		w			
	BRMW-4	BRMW-4-mmyy	MS/MSD	Groundwater			w	w		w	w	w	w	w	w		w			12, 40 mL VOA/HCl 3, 250 mL HDPE/HNO3, field filtered 3, 125mL Plastic w/HexCr Buffer, field filtered 3, 1 liter amber/HCl 15, 1 liter amber
	BRMW-5	BRMW-5-mmyy		Groundwater			w	w		w	w	w	w	w	w		w			4, 40 mL VOA/HCl 1, 250 mL HDPE/HNO3, field filtered 1, 125mL Plastic w/HexCr Buffer, field filtered 1, 1 liter amber/HCl 5, 1 liter amber
Background	Eastern undisturbed areas of island	BG1	-	Soil	To evaluate background concentrations of arsenic for comparison to other areas of the Site in the risk assessment	0 to 2 feet					As									1, 4 oz. clear glass
		BG2	-	Soil							As									1, 4 oz. clear glass
		BG3	-	Soil							As									1, 4 oz. clear glass
		BG4	-	Soil							As									1, 4 oz. clear glass
		BG5	-	Soil							As									1, 4 oz. clear glass
		BG6	-	Soil							As									1, 4 oz. clear glass
		BG7	-	Soil							As									1, 4 oz. clear glass
		BG8	-	Soil							As									1, 4 oz. clear glass
		BG9	-	Soil							As									1, 4 oz. clear glass
		BG10	-	Soil							As									1, 4 oz. clear glass

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Notes

*Samples contingent on Site conditions. Conditions warranting collection indicated under Target Depth/Methodology.

^Sample depths will not be duplicated. If target intervals overlap, multiple samples from the same interval will not be collected. Sample IDs will be adjusted based on required number of samples in each boring.

1 - Explosives analysis will include nitroglycerin

2 - Metals groundwater samples will be field filtered for dissolved analysis

As - only arsenic to be analyzed for background samples

FD - field duplicate

MS/MSD - matrix spike/matrix spike duplicate

s+ - Fingerprinting will be performed on two samples containing tar-like material

s**- Soxhlet extraction required

APPENDIX A

TIDE CHARTS – FEBRUARY AND MARCH 2018

US HARBORS RHODE ISLAND

Jamestown Tides - Feb / 2018

41°30'N 71°22'W

DATE		HIGH				LOW						
		AM	ft	PM	ft	AM	ft	PM	ft	RISE	SET	MOON
1	Thu	8:10	4.6	8:35	4.3	1:15	-0.8	2:18	-0.7	6:54	5:03	
2	Fri	9:00	4.4	9:25	4.2	2:08	-0.8	2:58	-0.6	6:53	5:04	
3	Sat	9:49	4.2	10:17	4.1	2:58	-0.6	3:34	-0.5	6:52	5:06	
4	Sun	10:40	3.8	11:09	3.8	3:44	-0.4	4:09	-0.3	6:51	5:07	
5	Mon	11:31	3.4			4:30	-0.1	4:47	-0.1	6:50	5:08	
6	Tue	12:03	3.6	12:23	3.1	5:22	0.3	5:30	0.2	6:49	5:09	
7	Wed	12:56	3.3	1:15	2.8	6:30	0.5	6:23	0.4	6:47	5:11	
8	Thu	1:50	3.1	2:10	2.6	8:10	0.6	7:27	0.5	6:46	5:12	
9	Fri	2:49	3.0	3:10	2.5	9:22	0.6	8:35	0.5	6:45	5:13	
10	Sat	3:52	2.9	4:13	2.5	10:10	0.5	9:35	0.4	6:44	5:14	
11	Sun	4:50	3.0	5:07	2.6	10:49	0.4	10:26	0.2	6:42	5:16	
12	Mon	5:37	3.1	5:51	2.8	11:27	0.2	11:11	0.1	6:41	5:17	
13	Tue	6:17	3.2	6:30	3.0			12:04 11:54	0.0 -0.1	6:40	5:18	
14	Wed	6:52	3.4	7:07	3.1			12:42	-0.1	6:39	5:19	
15	Thu	7:26	3.5	7:42	3.3	12:36	-0.2	1:18	-0.2	6:37	5:21	
16	Fri	8:00	3.5	8:18	3.3	1:17	-0.3	1:51	-0.3	6:36	5:22	
17	Sat	8:36	3.5	8:55	3.4	1:56	-0.3	2:22	-0.4	6:34	5:23	
18	Sun	9:14	3.5	9:35	3.4	2:33	-0.3	2:52	-0.4	6:33	5:24	
19	Mon	9:56	3.4	10:19	3.4	3:10	-0.3	3:24	-0.4	6:32	5:25	
20	Tue	10:43	3.2	11:07	3.4	3:48	-0.2	4:00	-0.3	6:30	5:27	
21	Wed	11:34	3.1	11:59	3.4	4:31	-0.0	4:43	-0.2	6:29	5:28	
22	Thu			12:29	3.0	5:25	0.1	5:34	-0.1	6:27	5:29	
23	Fri	12:55	3.4	1:27	3.0	6:36	0.3	6:38	-0.0	6:26	5:30	
24	Sat	1:56	3.5	2:31	3.0	8:22	0.3	7:53	-0.0	6:24	5:32	
25	Sun	3:04	3.6	3:40	3.1	9:53	0.1	9:10	-0.1	6:23	5:33	
26	Mon	4:15	3.8	4:46	3.4	10:52	-0.1	10:19	-0.3	6:21	5:34	
27	Tue	5:18	4.0	5:44	3.8	11:41	-0.3	11:19	-0.5	6:20	5:35	
28	Wed	6:14	4.3	6:37	4.1			12:27	-0.4	6:18	5:36	

Local Time

Tidal Data Source: Newport (8452660)

US HARBORS RHODE ISLAND

Jamestown Tides - Mar / 2018

41°30'N 71°22'W

DATE		HIGH				LOW						
		AM	ft	PM	ft	AM	ft	PM	ft	RISE	SET	MOON
1	Thu	7:04	4.4	7:27	4.3	12:14	-0.6	1:10	-0.5	6:16	5:37	
2	Fri	7:52	4.4	8:15	4.4	1:07	-0.7	1:49	-0.6	6:15	5:39	
3	Sat	8:39	4.3	9:03	4.3	1:56	-0.7	2:24	-0.5	6:13	5:40	
4	Sun	9:26	4.0	9:50	4.2	2:41	-0.5	2:56	-0.4	6:12	5:41	
5	Mon	10:12	3.7	10:39	3.9	3:22	-0.3	3:30	-0.3	6:10	5:42	
6	Tue	11:00	3.3	11:28	3.6	4:02	-0.1	4:05	-0.0	6:08	5:43	
7	Wed	11:50	3.0			4:45	0.2	4:45	0.2	6:07	5:44	
8	Thu	12:18	3.2	12:40	2.7	5:35	0.5	5:33	0.4	6:05	5:46	
9	Fri	1:10	3.0	1:31	2.5	6:43	0.7	6:34	0.6	6:04	5:47	
10	Sat	2:04	2.8	2:27	2.4	8:17	0.7	7:48	0.6	6:02	5:48	
11	Sun	4:06	2.7	4:30	2.5	10:27	0.6	10:04	0.5	7:00	6:49	
12	Mon	5:10	2.7	5:29	2.6	11:15	0.5	11:04	0.4	6:59	6:50	
13	Tue	6:02	2.9	6:18	2.8	11:55	0.3	11:52	0.2	6:57	6:51	
14	Wed	6:43	3.1	6:58	3.1			12:32	0.1	6:55	6:52	
15	Thu	7:19	3.3	7:35	3.3	12:36	-0.0	1:07	-0.1	6:54	6:53	
16	Fri	7:55	3.5	8:12	3.5	1:17	-0.2	1:42	-0.2	6:52	6:54	
17	Sat	8:31	3.6	8:49	3.7	1:58	-0.3	2:15	-0.4	6:50	6:56	
18	Sun	9:10	3.7	9:28	3.8	2:37	-0.4	2:48	-0.4	6:49	6:57	
19	Mon	9:51	3.7	10:10	3.9	3:16	-0.4	3:22	-0.5	6:47	6:58	
20	Tue	10:35	3.6	10:55	3.9	3:54	-0.4	3:58	-0.4	6:45	6:59	
21	Wed	11:24	3.4	11:45	3.8	4:34	-0.3	4:37	-0.4	6:43	7:00	
22	Thu			12:18	3.3	5:18	-0.1	5:21	-0.2	6:42	7:01	
23	Fri	12:40	3.7	1:15	3.2	6:11	0.1	6:14	-0.0	6:40	7:02	
24	Sat	1:39	3.6	2:14	3.2	7:24	0.3	7:20	0.2	6:38	7:03	
25	Sun	2:41	3.6	3:17	3.2	9:34	0.4	8:42	0.2	6:37	7:04	
26	Mon	3:49	3.6	4:25	3.4	10:50	0.2	10:12	0.1	6:35	7:05	
27	Tue	4:59	3.7	5:30	3.6	11:42	0.1	11:24	-0.1	6:33	7:07	
28	Wed	6:02	3.9	6:28	4.0			12:25	-0.1	6:32	7:08	
29	Thu	6:57	4.1	7:20	4.3	12:20	-0.2	1:03	-0.2	6:30	7:09	
30	Fri	7:46	4.2	8:08	4.5	1:09	-0.4	1:38	-0.3	6:28	7:10	
31	Sat	8:32	4.2	8:54	4.5	1:56	-0.4	2:11	-0.3	6:27	7:11	

Local Time

Tidal Data Source: Newport (8452660)

APPENDIX B

COMPREHENSIVE CONCEPTUAL SITE MODEL AND DATA GAP ANALYSIS

Appendix B
Comprehensive Conceptual Site Model and Data Gap Analysis
Gould Island (D01RI033800)
Narragansett Bay, Jamestown, Rhode Island

NED Site No.	Navy Structure Name/No./Building Description	History and 1997-2009 Site Observations (SWETS, 1997; Alion 2009)	Source and Anticipated Extent of Contamination	Contaminants of Potential Concern (COPCs)			Credere Identified Data Gaps
				Soil	Groundwater	Sediment	
1	Coal Storage North	Coal pile shown on 1943 aerial photos. Labeled "coal" on 1945 site plan. Not present on 1919, 1942, 1943, 1957, or 1963 site plans. Surface soil contains pieces of coal and coal dust. Vegetation sparse relative to surrounding area. One aliquot-composite sample collected for TPH, SVOC, and RCRA metals analysis. PAHs, arsenic, chromium, and TPH detected in surface/near surface soils with PAHs exceeding current comparison criteria. Concentrations of two SVOCs and arsenic exceeded RIDEM soil screening levels.	Typically PAHs and metals have limited mobility. COPCs are anticipated to be within the main body of the residual coal pile. The estimated extent of the coal as viewed on historical aerial photographs is depicted on **Figure 3**.	PAHs Metals EPH	None	NA	1. Horizontal and vertical extent of coal in soil by visual examination. 2. Nature and extent of previously identified contamination.
2	Marine Barracks	Shown on 1945 site plan. Not shown on 1919, 1942, 1943, 1957, or 1963 site plans. Not discernable on 1942/1943 aerial photos. No indication of USTs or other contamination sources on plans or aerial photos. Structure no longer standing. Concrete foundation for building present. Possible UST pipes found. Other pipes located appear to discharge to the Bay. No evidence of stressed vegetation. No evidence of USTs by geophysical survey (metal detection). No stressed vegetation or discolored soil. Location not sampled.	NA	None	None	None	No further action.
3	Carpenter's shop	Shown on 1943 aerial photos. Present on 1943 site plan and 1945 site plan. Not shown on 1919, 1942, 1957, or 1963 site plans. Evidence of site could not be found. No evidence of stressed vegetation. Records provide no indication of contamination sources. No visual evidence of site or stressed vegetation. Location not sampled.	NA	None	None	None	No further action.
4	Pump House	Pipeline labeled salt water on July 17, 1942, drawing "1.10.A.A Machine Gun Emplacements – Electrical, PW Drawing No. 5406-61". Drawings indicate facility was a saltwater pumphouse Small building on coast, still standing. Several empty, rusted 5-gallon containers were present. No evidence of stressed vegetation. No evidence of USTs, transformers, discolored soil or stressed vegetation. Location not sampled.	COPCs are anticipated to be encountered within the vicinity of rusted containers. Due to storm and daily tidal action, the drums' current locations may not be the point of discharge. Therefore, an approximate 50-foot radius from the drums and the pump house will be considered the sampling area. Depending on the drums' contents, contamination is expected to be encountered in the surface, vadose zone, and around the groundwater interface, which is expected to be shallower on the beach than up on the main island. Groundwater impacts are considered a lesser concern at this time; however, assessment will dually support assessment of NED Site No. 10.	VOCs SVOCs EPH VPH PCBs Metals	VOCs SVOCs EPH VPH PCBs Metals	NA	1. Remove empty 5-gallon containers from the island if still present. Sample in vicinity of containers for confirmatory sampling to satisfy AOC risk assessment/closure. 2. Assess groundwater impacts from release of drums contents (dual assessment with NED Site No. 10).

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Gould Island (D01RI033800)
Narragansett Bay, Jamestown, Rhode Island

NED Site No.	Navy Structure Name/No./Building Description	History and 1997-2009 Site Observations (SWETS, 1997; Alion 2009)	Source and Anticipated Extent of Contamination	Contaminants of Potential Concern (COPCs)			Credere Identified Data Gaps
				Soil	Groundwater	Sediment	
5	Unknown BLDG #2	Unknown structure (tent?) on 1943 aerial photos. Dark soil shown in vicinity on August 1943 aerial photo. Not shown on any site plans. Structure not found. Building debris located. No surficial evidence of contamination, USTs, or stressed vegetation. Records provide no indication of potential contamination sources. No surficial evidence of contamination, USTs or stressed vegetation. Location not sampled.	NA	None	None	None	No further action.
6	Unknown BLDG #3	Shown on 1943 aerial photos. Unknown structure (tent?). Not shown on any site plans. Structure not found. Retaining wall and wooden poles mark site. No debris. No surficial evidence of contamination, USTs, or stressed vegetation. Records provide no indication of potential contamination sources. No visual evidence of USTs, stained surface soil or stressed vegetation. Location not sampled.	NA	None	None	None	No further action.
7	Former Unknown BLDG #4	Undefined (apparent) construction activity on 1943 aerial photos. Labeled as "Shed" on 1919 site plan. Also shown on above-referenced 1943 and 1945 site plans (but unlabeled). Not present on 1957 or 1963 site plans. Structure no longer standing. Pieces of concrete mark site remnants of foundation. No surficial evidence of contamination, USTs, or stressed vegetation. No visual or geophysical (metal detection) indication of USTs. No evidence of surficial stained soils or stressed vegetation. Location not sampled.	NA	None	None	None	No further action.
8	Incinerator #49	Not shown on 1919 site plan. Building shown on 1943 plan and subsequent (1945, 1957, and 1963) site plans. Located adjacent to disposal area (NED Site 10). Structure still standing in 1997. Located valve possibly for a pipe or tank. Approximately 15 empty, rusted 5-gallon pails were present outside the building on a concrete surface. No other surficial evidence of potential contamination or stressed vegetation. No geophysical evidence of USTs. Location of pipe traced using geophysics consistent with water line shown on plan. Location not sampled.	COPCs are those typical of burned wastes. They are typically immobile contaminants and are expected to be encountered in surface soil surrounding the incinerator building and as debris/adhesions to the incinerator floor.	Dioxins/furans PAHs Metals PCBs	None	NA	1. Removal of empty 5-gallon containers from the island, if present (not observed 12/19/2017). 2. Assessment of incinerator floor and perimeter soil.
9	Magazine Ignitor Storage #37	Building shown on 1943 aerial photos. Shown on June 1943 plan, and subsequent drawings. Not shown on 1919 or 1942 plans. Evidence of building could not be found. No evidence of stressed vegetation. Location not sampled.	The primary COPCs for this AOC is explosives; however, due to limited historical information associated with the building and the proximity within the area of NED Site No. 48, other Site COPCs warrant screening. COPCs are expected to be encountered in surface soil.	SVOCs Explosives VOCs PCBs EPH VPH Metals	None	NA	1. GPR survey to screen for anomalies 2. Assessment of soil to satisfy AOC risk assessment/closure. (To be assessed with NED Site No. 48.)

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Gould Island (D01RI033800)
Narragansett Bay, Jamestown, Rhode Island

NED Site No.	Navy Structure Name/No./Building Description	History and 1997-2009 Site Observations (SWETS, 1997; Alion 2009)	Source and Anticipated Extent of Contamination	Contaminants of Potential Concern (COPCs)			Credere Identified Data Gaps
				Soil	Groundwater	Sediment	
10	Disposal Area #14	1983 (Envirodyne, 1983) and 1986 (Loureiro, 1986) reports indicate use from the World War II era until approximately the early 1950s. No waste known to have been transported back to mainland and was all disposed here. Possible outfall from electroplating building reported. Not shown on any site plans. Filling not discernable on 1942 or 1943 aerial photos. Large amounts of metal, concrete, and wood debris located in several areas. Incinerator/boiler slag and several (rusted) torpedo drive-sections also identified. Numerous areas of tar located throughout the area and subsurface soil was reportedly black and hardened when exposed to the air. Two areas, each containing several empty, rusted 55-gallon drums (approximately 10 total), found on top of area. Portions of the open face of disposal area are in contact with Bay at high tide. One composite soil sample collected near drums was analyzed for TPH, VOC, SVOC, pesticides, PCBs, RCRA metals, and cyanide. One additional sample collected due to one additional area of drums identified on the ground surface and was analyzed for TPH, VOC, SVOC, pesticide, PCB, RCRA metals, explosives, nitroglycerin, and cyanide. Nitroglycerin, PAHs, lead, chromium, and arsenic detected at concentrations higher than at least one of the current comparison criteria. Highest concentration previously detected. Lead, chromium, nickel, and copper above background control sample results in sediment and lead and copper in mussel tissue samples. Some prior sediment results exceed the current marine TEL/PEL. Extent of the disposal area was previously determined by magnetometry.	COPCs are anticipated to be encountered within the previously defined body of waste in the disposal area. Soluble COPCs have the potential to have leached from the waste into groundwater or directly into the adjoining bay. Based on prior reports, waste is likely disposed over thin overburden or directly on bedrock. Soil on the beach was previously observed to rocky with little fines. This absence of fines on the beach may indicate facilitated migration of COPCs into the bay due to lack of binding materials. Metals have previously been documented at elevated concentrations in the bay; however, their extent has not been assessed. Liquid free products may be present at the groundwater interface as LNAPL or deeper above confining layers, bedrock or within bedrock fractures for DNAPL. Based on the age of the releases and adjoining action of the bay, any free-product is presumed to be in a mature stage of plume stability. Tar was observed to be present during the December 19, 2017 site visit and was in contact with the water at high tide.	Explosives VOCs SVOCs EPH VPH Acids (via pH) Metals PCBs Pesticides	Explosives VOCs SVOCs EPH VPH Acids (via pH) Metals Pesticides	Explosives VOCs SVOCs EPH VPH Acids (via pH) Metals PCBs Pesticides	1. Vertical delineation of waste onshore. 2. Assessment of nature and extent of previously identified contamination including soil, groundwater, and sediment into the bay. 3. Removal of containers and solid waste. 4. Identify possible outfall from the electroplating building.
11	Quonset Huts/Maintenance Shops	Shown on 1943 aerial photos. Shown on 1943 site plan. Fewer Quonset huts shown on 1945 site plan. Labeled as “Maintenance Shop” and “Heating Plant” on 1957 site plan. Not present on 1919, 1942, or 1963 site plans. Structures no longer standing. Building and other debris, including wood, nails, and roofing materials found. No surficial evidence of contamination, USTs, or stressed vegetation. No geophysical (metal detection) evidence of USTs. Location not sampled.	COPCs are anticipated to be encountered in surface soil throughout the area. Releases in the area may have impacted overburden groundwater locally; however, given the history, impacts to bedrock groundwater from this source is not a concern at this time.	VOCs SVOCs EPH VPH Metals	VOCs SVOCs EPH VPH Metals	NA	1. Soil assessment for use as a maintenance and heating plant. 2. Groundwater may require assessment depending on soil results.

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NED Site No.	Navy Structure Name/No./Building Description	History and 1997-2009 Site Observations (SWETS, 1997; Alion 2009)	Source and Anticipated Extent of Contamination	Contaminants of Potential Concern (COPCs)			Credere Identified Data Gaps
				Soil	Groundwater	Sediment	
12	Maintenance Shop/Garage/Fire Station #39	Buildings not shown on 1919 site plan. Northernmost building shown as "Under Construction" on 1942 site plan. Northernmost structure shown on 1943 aerial photos and 1943 site plan. Eastern and southern attached buildings shown on 1945 site plan and subsequent plans. RIDEM records suggest a 5,000-gallon fuel oil UST was removed from southwest of the building. Structures were still present in a dilapidated state in 2009. Southernmost building collapsed. Possible UST vent pipe found on north side of northernmost building. Oil-fired boiler, manhole for electrical utilities, transformer with spilled contents, and maintenance pit in concrete floor located inside buildings. Sparse vegetation relative to surroundings observed in area of possible former UST southwest of the buildings at Site 12. No prior data or confirmatory assessment associated with UST removal. Location not sampled. Magnetometer survey identified suspect additional UST anomaly unassociated with previously removed UST.	PCBs associated with the transformer within the Site building are expected to have impacted the underlying concrete or have migrated through the concrete to the underlying soil. PCBs, TPH, VOCs, SVOCs, and metals associated with the maintenance pit are likely confined to debris or soil within the maintenance pit due to the known concrete bottom; however, due to the age and dilapidated nature, cracks may be present allowing for limited migration out of the pit. Underlying soil and local overburden groundwater may also have become impacted. A release for the additional suspect UST would have impacted soil surrounding and beneath the UST. Petroleum would have migrated to the groundwater interface as LNAPL and the dissolved phase COPCs would be present in groundwater at and downgradient of the UST. Partially full drum observed outside garage bay.	PCBs EPH VPH VOCs SVOCs Metals	PCBs EPH VPH VOCs SVOCs Metals	NA	1. Possible additional UST northwest of building that would require assessment. Confirm with GPR survey. 2. Assessment of spilled transformer contents and impacted media for PCBs. 3. Assessment of vehicle maintenance pit contents despite concrete bottom for PCBs, TPH, VOCs, SVOCs and metals. 4. Previously removed USTs were removed by the Navy and, therefore, are not covered under FUDS. 5. Remove partially full drum.
13	Electric Substation/ Transformer Pen #43	Building and fenced transformer pen shown on 1942 site plan and subsequent site plans. Cannot tell if present on 1943 aerial photos. Fence and adjacent building still standing. Former transformer pads present. Transformers not present. One empty, rusted 5-gallon pail found on ground surface. No other surficial evidence of contamination, USTs, or stressed vegetation. Two soil samples collected to evaluate soil associated with former transformers and were analyzed for TPH, PCB, and RCRA metals. PCBs detected below RIDEM soil screening levels and TSCA cleanup goal or 1 mg/kg, but above EPA RSLs in surface soil samples. TPH, arsenic, chromium, and lead detected at concentrations exceeding current comparison criteria.	COPCs are expected to be encountered in surface soil within and surrounding the transformer pen and substation, particularly beneath and surrounding the 5-gallon container. Based on prior sample results, COPCs are not considered concentrated enough to warrant groundwater sampling for this AOC.	PCBs EPH Metals	None	NA	1. Removal of empty 5-gallon container from island and sample soil in vicinity of container. 2. Nature and extent of previous identified contamination.
14	Marine Barracks	Not shown on 1919 site plan. Building shown on 1943 aerial photos. Shown on the "Gould Island Conditions, 1949" site plan as "Residence," and on the 1943 site plan, but is not present on subsequent (1945, 1957, 1963) site plans. "Concrete Platform" shown on 1957 and 1963 site plans to south. Evidence of building could not be found. Records provide no indication of contamination sources. No evidence of site or stressed vegetation could be found. Location not sampled.	NA	None	None	None	No further action.

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NED Site No.	Navy Structure Name/No./Building Description	History and 1997-2009 Site Observations (SWETS, 1997; Alion 2009)	Source and Anticipated Extent of Contamination	Contaminants of Potential Concern (COPCs)			Credere Identified Data Gaps
				Soil	Groundwater	Sediment	
15	Recreation Building	It is not present on earlier (1919, 1942, and 1943) site plans, or in the 1942 and 1943 aerial photos. Building first appears on the 1945 site plan. It is not present on the 1957 or 1963 site plans. Evidence of building could not be found. Records provide no indication of contamination sources. No evidence of site or stressed vegetation could be found. Location not sampled.	NA	None	None	None	No further action.
16	Barracks	Buildings are present in 1943 aerial photos. The easternmost building is present, and the westernmost building is shown as under construction in the 1942 site plan. The buildings are shown as present on the 1943 and 1945 site plan. The buildings are shown as present on the 1943 and 1945 site plans, but are not present on the 1919 or the 1957 and 1963 site plans. Structures no longer standing. Concrete platform present. Transformers partially buried due north of Site 16 in small mounds of soil. No other surficial evidence of contamination or USTs. Sparse vegetative growth near transformers relative to surroundings. No geophysical (metal detection) anomalies indicative of USTs. Location not sampled.	COPCs are anticipated to be encountered within the vicinity of the spilled transformers. Contamination is expected to be encountered in the surface soil. Migration to groundwater is not a concern at this time.	PCBs EPH VPH	None	NA	1. Removal of transformers from the island and soil assessment to satisfy AOC risk assessment/closure. Transformers in separate location relative to Transformer Vault.
17	Transformer Vault #25A	The building is not shown on the 1919, 1942, 1943, and 1945 site plans, and is not shown on the 1942/1943 aerial photos. Building is shown on the 1957 and 1963 site plans. Evidence of building could not be found. No evidence of site or stressed vegetation could be found. Location not sampled.	NA	None	None	None	Navy Easement – Not FUDS eligible. No further action.
18	Water Treatment Plant #42	Building is not present in the January 1943 aerial photo, is under construction in an April 1943 aerial photo, and is present in August 1943 aerial photo. Consistent with the above, it is not shown on the 1919 and 1942 site plans, but is shown on the 1943 site plan and subsequent (1945, 1957, and 1963) site plans. Building still standing. Tanks in building empty and clean. No surficial evidence of contamination, USTs, or stressed vegetation found. Treatment tanks in building empty and clean. No evidence of USTs, surficial soil contamination, or stressed vegetation. Location not sampled.	NA	None	None	None	Navy Easement – Not FUDS eligible. No further action.

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				Soil	Groundwater	Sediment	
19	Torch Pot Storage #51	Building under construction shown on the January 1943 aerial photo and is present on the August 1943 aerial photo. The building is shown as scheduled for construction on the 1942 site plan, and is present on the 1943, 1957, and 1963 site plans. Standing three-walled concrete structure without roof present. One empty, rusted 30-gallon drum marked "Low Pressure Hexane Vapor" found. No other surficial evidence of contamination, USTs, or stressed vegetation. One soil sample collected near drum was analyzed for TPH and VOC. No VOCs detected in surface soil beneath drum. TPH in surface soil beneath drum just below RIDEM soil screening levels.	COPCs are expected to be encountered in the vicinity of the drum, if still present. Prior analytical results indicate concentrations were below the comparison criteria; however, a single grab sample may not represent the vicinity of the drum. Migration to groundwater is not a concern at this time.	EPH	None	NA	1. Removal of empty drum from the island. 2. Confirmatory soil sampling after removal. If no drum is currently identified, no confirmatory sampling beneath the drum is warranted based on prior data. 3. General assessment in the vicinity of the drum.
20	Cable Terminal BLDG #87	Not present on prior (1919, 1942, and 1943) site plans. Not apparent on 1942/1943 aerial photos. Building first appears on 1957 site plan, and is also shown on 1967 site plan. Structure no longer standing. Single utility pole at site. No surficial evidence of contamination, USTs, or stressed vegetation. Records provide no indication of contamination sources. No visual evidence of USTs, surficial soil contamination, or stressed vegetation. Location not sampled.	NA	None	None	None	No further action.

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				Soil	Groundwater	Sediment	
21	Bunker #11	First appears on 1919 site plan, with railroad tracks extending to/from torpedo storehouse (NED Site 38) and pier at southeast corner of island. Railroad lines no longer apparent on 1943 site plan or 1943 aerial photo. Building behind (west of) each bunker retaining wall constructed of wood. Used for warhead storage through 1941; pistol shooting range thereafter. Superstructure still present, badly deteriorated. Wood building no longer present. Contains variable debris, including numerous (approximately 20 or more) empty, rusted 55-gallon drums and 5-gallon containers. Torpedo mid-section present beneath debris. No evidence of surficial contamination or USTs found. Vegetation sparse near some drums and containers than surrounding areas. Some drums under debris not readily accessible. Two composite soil samples collected near drums were analyzed for TPH, VOC, RCRA metals, explosives, and nitroglycerin. TPH, arsenic, chromium and lead concentrations exceeding current comparison criteria. A torpedo section was found that the Navy EOD Unit determined poses no explosion hazard.	The complete nature and extent of this area is not known due to the unknowns located beneath the collapsed bunker debris. Several additional drums of unknown content remain beneath the debris. COPCs in soil are expected to be encountered beneath the structure and near the entrance in surface soil, as well as in the soil vadose zone and at the groundwater interface. As this bunker was used a shooting range, lead contamination is expected to be highest at the target end of the range. Liquid free products may be present at the groundwater interface as LNAPL or deeper above confining layers, bedrock or within bedrock fractures for DNAPL. Based on the age of the releases, any free-product is presumed to be in a mature stage of plume stability. This area is currently unsafe to enter and represents a continued threat of further release due to unknown contents of the bunker when it collapsed.	EPH VPH Metals PCBs Explosives SVOCs VOCs Pesticides	EPH VPH Metals PCBs Explosives SVOCs VOCs Pesticides	NA	1. Visible and buried containers and solid waste beneath collapsed structure require removal. 2. Nature and extent of previously identified contamination in soil and groundwater.
22	Bunker #12	First appears on 1919 site plan, with railroad tracks extending to/from torpedo storehouse (NED Site 38) and pier at southeast corner of island. Railroad lines no longer apparent on 1943 site plan or on August 1943 aerial photo. Building behind (west of) each bunker retaining wall constructed of wood. Structure still present, badly deteriorated. Contains variable debris, including numerous (up to 30 or more) empty, rusted 55-gallon drums and 5-gallon containers. No evidence of surficial contamination or USTs found. Vegetation sparse near some drums and containers relative to surrounding area. Two composite soil samples collected near drums were analyzed for TPH, VOC, RCRA metals, explosives, and nitroglycerin. Lead, chromium and arsenic were detected at concentrations exceeding current comparison criteria.	The complete nature and extent of this area is not known due to the unknowns located beneath the collapsed bunker debris. Several additional drums of unknown content remain beneath the debris. COPCs in soil are expected to be encountered beneath the structure and near the entrance in surface soil, as well as in the soil vadose zone and at the groundwater interface. Liquid free products may be present at the groundwater interface as LNAPL or deeper above confining layers, bedrock or within bedrock fractures for DNAPL. Based on the age of the releases, any free-product is presumed to be in a mature stage of plume stability. This area is currently unsafe to enter and represents a continued threat of further release due to unknown contents of the bunker when it collapsed.	EPH VPH Metals PCBs Explosives SVOCs VOCs Pesticides	EPH VPH Metals PCBs Explosives SVOCs VOCs Pesticides	NA	1. Visible and buried containers and solid waste beneath collapsed structure that requires removal. 2. Nature and extent of previously identified contamination in soil and groundwater.

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NED Site No.	Navy Structure Name/No./Building Description	History and 1997-2009 Site Observations (SWETS, 1997; Alion 2009)	Source and Anticipated Extent of Contamination	Contaminants of Potential Concern (COPCs)			Credere Identified Data Gaps
				Soil	Groundwater	Sediment	
23	Coal Storage South	Coal pile shown on 1943 aerial photos. Labeled "Coal" on 1945 site plan. Not present on 1919, 1943, 1957, or 1963 site plans. Surface soil contains pieces of coal and coal dust. Vegetation sparse relative to surrounding area. One composite soil sample from two locations previously staked was analyzed for TPH, SVOC, and RCRA metals. PAHs, arsenic, chromium and TPH detected in surface /near surface soils with PAHs, arsenic, and chromium exceeding current comparison criteria	Typically PAHs and metals have limited mobility. COPCs are anticipated to be within the main body of the residual coal pile. The estimated extent of the coal as viewed on historical aerial photographs is depicted on **Figure 3**.	PAHs Metals EPH	None	NA	1. Horizontal and vertical extent of coal in soil by visual examination. 2. Nature and extent of previously identified contamination in soil.
24	Cable Terminal BLDG #16	Building appears at this location on 1919, 1942, 1943, and 1945 site plans, labeled "Waiting Room." Same structure labeled "Fresh Water Pump House" on 1957 site plan, and "Hydrophone Cable Terminal Building" on 1963 site plan. Building present in 1943 aerial photos. Structure still standing. One empty, rusted 55-gallon drum found. Concrete pad at site (possible former transformer pad?). No other surficial evidence of contamination, USTs, or stressed vegetation. One composite soil sample collected near drums and pad was analyzed for TPH, PCB, and VOC. PCBs and TPH were detected in surface/near surface soil samples collected near probable former transformer pad and an empty, rusted 55 gallon drum. The TPH and PCB concentrations were less than the current comparison criteria.	Based on the prior results, COPCs are expected to be encountered in surface soil. Migration to groundwater is not a concern at this time.	PCBs EPH VPH	None	NA	1. Removal of drum. 2. Additional limited sampling/analysis of surface/near surface soils to confirm the prior results to satisfy AOC risk assessment/closure.
25	Underground Storage Tanks	Unknown buried structure present in 1943 aerial photos, but not present on any site plans. The proximity of this NED site to a 500,000 gallon water tank and cable terminal building, the linear feature between buried structure at this Site and the vicinity of NED Sites 34/38 suggests structure could be vault associated with subsurface utilities (vs. USTs). Low brush and weeds at top of hill relative to surrounding area. No signs of USTs. No surficial evidence of USTs, contamination, or stressed vegetation. (Note: structure at NED Sites 34/38 appears to be manholes for utility vaults, consistent with theory based on aerial photos reviewed.) Site location could not be found in the field. Broad geophysical (magnetometer) sweep of area did not locate any metallic anomalies that could be USTs. Location not sampled.	NA	None	None	NA	No further action.

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				Soil	Groundwater	Sediment	
26	Gas Pump House BLDG #23/Gas Tanks	Not present on 1919 and 1942 site plans. Labeled "Gas Tanks" and "Pump House" on 1943 site plan, and subsequent plans. Building present on January 1943 and subsequent aerial photos. Navy and RIDEM records suggest two 5,000-gallon gasoline USTs were removed in 1989. RIDEM reportedly observed tank removals and no remediation was deemed necessary at the time. Collapsed wooden structure. Area clear of brush found where USTs may previously have been located. No other surficial evidence of contamination or stressed vegetation. Water from USTs contained BTEX, lead and chlorinated/non-chlorinated solvents. Soil in location not sampled. No geophysical indication of UST or piping.	NA	None	None	None	1. No further action. Previously removed USTs were removed by the Navy and, therefore, NED site is not covered under FUDS.
27	Two Gas Pits (east side of pad)	Appears labeled as "Gasoline Pits" along eastern edge of concrete apron at south end of island on 1943 and 1945 site plans. Not present on 1919, 1942, 1957, or 1963 site plans. Slightly depressed areas shown on 1943 aerial photos, possibly representing refueling or drainage collection pits. Evidence of site could not be found. No visual evidence of site or stressed vegetation. Location not sampled.	Design of gasoline pits is not known. Leaks of gasoline from the pits would impact surrounding soil and likely consolidate above groundwater interface. Pit contents may contain residual sludge.	VOCs SVOCs EPH VPH Metals PCBs	VOCs SVOCs EPH VPH Metals PCBs	NA	1. Generalized assessment in vicinity to confirm lack of contaminated soil and groundwater.
28	Ordnance Test Facility/Hangar	First appears on 1943 site plan, labeled "Hangar." Same label on 1945 and 1957 site plans. Labeled "Ordnance R&D Test Facility" on 1963 site plan. Not shown on 1919 and 1942 site plans. Present in January 1943 aerial photo. No standing structures remain after 1985 hurricane. Concrete mat (floor/apron) covering large area found, including NED Sites 28, 31, 32, 42, and 43. Could only find concrete apron. Building contents washed to sea during hurricane (1985). Location not sampled.	General Site COPCs may be present beneath the concrete apron as operations in the former ordnance test facility are not known. The hangar may also have stored and used solvents and aviation fuels during operation. Releases to the subsurface may have migrated to groundwater. Overburden groundwater would likely migrate quickly into the adjoining bay.	VOCs SVOCs Explosives PCBs EPH VPH Metals	VOCs SVOCs Explosives PCBs EPH VPH Metals	NA	1. General assessment beneath concrete apron. 2. General assessment of likely comingled groundwater contamination with downgradient wells. (Combined assessment with NED Site Nos. 30, 32, 33, 43 and 44, groundwater assessed in wells for NED Site Nos. 27, 30, and 31)
29	Unknown BLDG #1 (Hangar 5496-61)	Present in 1942 site plan, and in January and April 1943 aerial photos. No longer present in August 1943 aerial photo. Not shown on 1919, 1943, 1945, 1957, and 1963 site plans. No standing structures remain after 1985 hurricane. Site could not be located precisely. Underground concrete vault found, open at the top, near the site. Could only find concrete apron. Building contents washed to sea during hurricane (1985). Location not sampled.	NA	None	None	None	1. No further action. 2. Upgradient sites 28, 33, and 43 are being sampled as and will satisfy risk assessment of general area, including NED Site 29.

Appendix B
Comprehensive Conceptual Site Model and Data Gap Analysis
Gould Island (D01RI033800)
Narragansett Bay, Jamestown, Rhode Island

NED Site No.	Navy Structure Name/No./Building Description	History and 1997-2009 Site Observations (SWETS, 1997; Alion 2009)	Source and Anticipated Extent of Contamination	Contaminants of Potential Concern (COPCs)			Credere Identified Data Gaps
				Soil	Groundwater	Sediment	
30	Pyrotechnic Storage	Shown only on 1945 site plan. Not evident at site plan location on 1943 aerial photos. Evidence of site could not be found. No visual evidence of site or stressed vegetation. Building contents washed to sea during hurricane (1985). Location not sampled.	General Site COPCs may be present beneath the concrete apron. Particularly in this vicinity explosives are anticipated. Releases to the subsurface may have migrated to groundwater. Overburden groundwater would likely migrate quickly into the adjoining bay.	VOCs SVOCs Explosives PCBs EPH VPH Metals	VOCs SVOCs Explosives PCBs EPH VPH Metals	NA	1. General assessment beneath concrete apron. 2. Assessment of groundwater that may be comingled with upgradient sources from beneath the concrete pad. (Combined assessment with NED Site Nos. 28, 32, 33, 43 and 44)
31	Gas Pit (one gas pit west side of pad)	Appears labeled as "Gasoline Pits" along western edge of concrete apron at south end of island on 1943 site plan, and on the 1945 site plan. Not present on 1919, 1942, 1957, or 1963 site plans. Slightly depressed areas shown on 1943 aerial photos. May represent refueling or drainage collection pits. No standing structures. Concrete mat (floor/apron) covering large area including NED Sites 28, 31, 32, 42, and 43. Could only find concrete apron. Feature could not be found in the field. Location not sampled.	Design of gasoline pits is not known. Leaks of gasoline from the pits would impact surrounding soil and likely consolidate above groundwater interface. Pit contents may contain residual sludge.	VOCs SVOCs PCBs EPH VPH Metals	VOCs SVOCs PCBs EPH VPH Metals	NA	1. Generalized assessment in vicinity to assess soil and groundwater.
32	Drum Storage Area	Drums apparent on northwest corner of concrete apron at southern end of island in January and April 1943 aerial photos. Not discernable on other aerial photos. Not indicated on any of the site plans. No standing structures. Concrete mat (floor/apron) covering large area including NED Sites 28, 31, 32, 42, and 43. Could only find concrete apron. Location not sampled.	General Site COPCs may be present beneath the concrete apron. Particularly in this vicinity explosives are anticipated. Releases to the subsurface may have migrated to groundwater. Overburden groundwater would likely migrate quickly into the adjoining bay.	VOCs SVOCs Explosives PCBs EPH VPH Metals	VOCs SVOCs Explosives PCBs EPH VPH Metals	NA	1. Generalized assessment in vicinity and beneath concrete apron to assess soil. 2. General assessment of likely comingled groundwater contamination with downgradient wells. (Combined assessment with NED Site Nos. 28, 30, 33, 43 and 44, groundwater assessed in wells for NED Site Nos. 27, 30, and 31)
33	Paint and Oil Storage #47	First appears on 1945 site plan, and is also shown on 1957 and 1963 site plans. Evident on 1943 aerial photos, but not on August 1942 aerial photo. Evidence of site could not be found. No visual evidence of site or stressed vegetation. Location not sampled.	General Site COPCs may be present beneath the concrete apron. Particularly in this vicinity VOCs and metals are anticipated. Releases to the subsurface may have migrated to groundwater. Overburden groundwater would likely migrate quickly into the adjoining bay.	VOCs SVOCs Explosives PCBs EPH VPH Metals	VOCs SVOCs Explosives PCBs EPH VPH Metals	NA	1. Generalized assessment in vicinity and beneath concrete apron to assess soil. 2. General assessment of likely comingled groundwater contamination with downgradient wells. (Combined assessment with NED Site Nos. 28, 30, 32, 43 and 44, groundwater assessed in wells for NED Site Nos. 27, 30, and 31)
34	Theater/Research BLDG #27	Not shown on the 1919 site plan. Present in 1942 and 1943 aerial photos. Shown on 1942, 1943, and 1945 site plans as "Research Building." Labeled as "Theatre" on 1957 and 1953 site plans. No building present. Foundation and elevated concrete structure (not typical of a transformer pad) still standing. No surficial evidence of contamination, USTs, or stressed vegetation. No visual evidence of surficial contamination or stressed vegetation. Location not sampled.	NA	None	None	None	No further action.

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NED Site No.	Navy Structure Name/No./Building Description	History and 1997-2009 Site Observations (SWETS, 1997; Alion 2009)	Source and Anticipated Extent of Contamination	Contaminants of Potential Concern (COPCs)			Credere Identified Data Gaps
				Soil	Groundwater	Sediment	
35	Boiler House #29	Not shown on 1919 or 1942 site plans. Shown on 1943 site plan, and all subsequent site plans. Not evident on August 1942 aerial photo, but is present on 1943 aerial photos. Structure still standing. Three boilers present; fireboxes contain shaker grates, indicating coal-fired. No evidence of USTs. No surficial evidence of contamination, other than coal pieces and coal dust on ground surface. Vegetative cover in areas with coal less dense than surroundings. Coal and coal ash were present. Location not sampled.	COPCs are expected to be encountered within any coal ash associated with the boiler house. Typical of the time, coal ash would have been discarded outside the boiler house. As COPCs typically have limits mobility, groundwater is not expected to be impacted by these COPCs. Residual waste ash is also expected to remain in the fireboxes and the stack.	PAHs Metals Dioxins/furans	None	NA	1. Horizontal and vertical extent of coal ash. 2. Characterization of the residual contents of the fireboxes and stack.
36	Degaussing BLDG #26	Building shown on 1942 and all subsequent site plans, but not on 1919 site plan. Shown on 1942 and 1943 aerial photos. According to CENAE, the degaussing facility (demagnetizing of ships to prevent them from attracting mines or torpedoes) was put into operation in 1944. Wooden structure still standing. Pipe exiting ground (UST fill pipe?). No other surficial evidence of contamination or stressed vegetation. Geophysical survey did not identify any metallic anomalies resembling a UST including the vicinity of a pipe protruding from the ground. No visual evidence of surficial contamination or stressed vegetation. Location not sampled.	NA	None	None	None	No further action.
37	Misc. Storage #28	Building not present on 1919 site plan. Building is shown (but unlabeled) on 1942 site plan. Present on 1942 and 1943 aerial photos. Labeled "South Compressor Building" on 1943 and 1945 site plans, and "Misc. Storage" on 1957 and 1963 site plans. Evidence of UST found in the field also evident in the April 1943 aerial photo. Concrete and brick structure still standing. Apparent UST found west of structure, between Bldgs. #28 and #10 (NED Sites 37 and 38). About 5 feet to top of liquid in UST. UST bottom about 8 feet deep. Sparse vegetative cover, but no surficial evidence of contamination. One water sample was analyzed for TPH and VOC. No NAPLs present inside UST. Laboratory analysis of UST water sample revealed no detectable concentrations of VOCs or TPH. Soil in location not sampled. Two ASTs observed on either end of the building.	The use and purpose of the remaining UST and ASTs is not known. COPCs are those required by RIDEM for an unknown contents storage tank removal as well as additional typical Site COPCs. Impacts soil would be expected surrounding the UST. As water was present in the UST, the UST is assumed to be compromised and filled with groundwater or stormwater. Any free product released from the tank would migrate vertically until encountering groundwater or a confining media. Groundwater may be impacted by dissolved phase COPCs. Surface soil beneath the ASTs may also be impacted depending on the prior storage.	EPH VPH VOCs SVOCs PCBs Metals	EPH VPH VOCs SVOCs PCBs Metals	NA	1. UST removal and confirmatory assessment of soil and groundwater. 2. Remove ASTs.

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NED Site No.	Navy Structure Name/No./Building Description	History and 1997-2009 Site Observations (SWETS, 1997; Alion 2009)	Source and Anticipated Extent of Contamination	Contaminants of Potential Concern (COPCs)			Credere Identified Data Gaps
				Soil	Groundwater	Sediment	
38	Torpedo Storage #10	Building present on 1919 site plan and all subsequent site plans. Also shown on 1942 and 1943 aerial photos. Shed attached to northeast corner of building. Large, elongated concrete and brick structure still standing. Mostly empty, but contains some electrical and water purifying equipment, some still in shipping creates. Weeping transformer found in alcove at northeast corner of bldg; oil residue present on building floor and/or ground surface near transformer. No other surficial evidence of contamination, USTs, or stressed vegetation. No samples collected in vicinity of leaking transformers	COPCs are anticipated to be encountered within the vicinity of the spilled transformers. Contamination is expected to be encountered in the surface soil or migrated into underlying concrete, if present. Migration to groundwater is not a concern at this time.	PCBs EPH VPH	None	NA	1. Removal of transformer and contents from island. 2. Assess below transformers and general area.
39	Well House #81	Building first appears on 1943 site plan, labeled "Drilled Well." Present in all subsequent site plans, labeled "Drilled Well" or "Deep Well Hose." Not discernable on 1942 and 1943 aerial photos. Brick structure still standing, containing pumping equipment. Evidence of removed electrical equipment, perhaps transformer. No surficial evidence of contamination, USTs, or stressed vegetation. One composite soil sample collected in location of former transformer pad was analyzed for TPH and PCB. PCBs and TPH were detected in vicinity of former transformer pad. TPH and PCBs were below RIDEM soil screening levels, but PCBs exceed EPA RSL and the 1 mg/kg cleanup goal for liquid releases assumed to be greater than 50 mg/kg PCBs under TSCA.	PCBs are expected to be limited to surface soil surrounding the transformer pad and the concrete of the transformer pad.	PCBs EPH VPH	None	NA	1. Nature and extent of previously identified PCBs in soil surrounding transformer pad. Observe pad location relative to well.
40	Well House #78	Building first evident on 1942 site plan, labeled "Drilled Well." Present in all subsequent site plans, labeled "Drilled Well" or "Deep Well House." Present in 1943 aerial photos. Small wooden structure, still standing. No surficial evidence of contamination, USTs, or stressed vegetation. No evidence of transformer/pad, UST, surficial soil contamination, or stressed vegetation. Location not sampled	NA	None	None	None	No further action.

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Gould Island (D01RI033800)
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NED Site No.	Navy Structure Name/No./Building Description	History and 1997-2009 Site Observations (SWETS, 1997; Alion 2009)	Source and Anticipated Extent of Contamination	Contaminants of Potential Concern (COPCs)			Credere Identified Data Gaps
				Soil	Groundwater	Sediment	
41	AA Guns Electrical Supply	First shown on 1942 plan. Pole-mounted transformers shown on plan. Portions of gun mount stations shown on all subsequent plans. Gun mount stations shown under construction in January 1943 aerial photo, and present in April and August 1943 aerial photos. Not present in August 1942 aerial photo. Transformer pole not discernable on aerial photos. Transformer pole still standing, but transformers not present. No surficial evidence of contamination, USTs, or stressed vegetation. One composite soil sample collected around base of pole where transformer was formerly located was analyzed for TPH and PCB. No evidence of transformer/pad, UST, (no metal anomalies representative of possible USTs during geophysical survey), surficial soil contamination, or stressed vegetation. PCBs not detected. TPH detected just below RIDEM soil standard.	NA	None	None	NA	No further action
42	5,000-Gallon Tank (Av gas) Ordnance Test Facility Gasoline Outlet #30	"5000 gal. Aviation Gas Tank" shown on 1942 site plan. Labeled "Gas Tanks" and "Gasoline Pump" on 1942 site plan. Not shown on other site plans. Not discernable on aerial photos. No standing structures. Concrete mat (floor/apron) covering large area, including Sites 28, 30, 31, 32, 42, and 43. Concrete apron present. No visual evidence of site or stressed vegetation could be found. Location not sampled.	COPCs would be expected to be encountered in surface and vadose zone soil and at the groundwater interface surrounding the former pump, and possibly in the subsurface if the tank was a UST. Dissolved phase COPCs may also be present in groundwater if a release occurred from the tank.	VOCs SVOCs EPH VPH Metals PCBs	VOCs SVOCs EPH VPH Metals PCBs	NA	1. Assessment of surface and subsurface soil and groundwater for release of petroleum from tank. 2. Geophysical survey to confirm if possible UST evidence is present.
43	Boiler House and Ordnance Test Facility Paint Shed #30 (Located in more recent location of NED Site No. 28)	First present on 1942 site plan, labeled "Paint Storage." Labeled "Paint Shed" on 1942 plan. Not present on 1943 and subsequent site plans. Evident on August 1942 aerial photo, but not present on January 1943 and subsequent 1943 aerial photos. No standing structures. Concrete mat (floor/apron) covering large area, including Sites NED Site Nos. 28, 30, 31, 32, 42, and 43. Concrete apron present. No visual evidence of site or stressed vegetation could be found. Location not sampled.	Coal ash associated with the former boiler house is expected to be encountered immediately beneath the concrete apron. Releases to the subsurface may have migrated to groundwater. Overburden groundwater would likely migrate quickly into the adjoining bay.	VOCs SVOCs EPH VPH Metals PCBs Explosives	VOCs SVOCs EPH VPH Metals PCBs Explosives	NA	1. Generalized assessment in vicinity and beneath concrete apron to assess soil. 2. General assessment of likely comingled groundwater contamination with downgradient wells. (Combined assessment with NED Site Nos. 28, 30, 32, 33 and 44, groundwater assessed in wells for NED Site Nos. 27, 30, and 31)
44	Empty Drums at south end of concrete mat area near sites 27-33	No records pertaining to these. Not shown on aerial photos. Two empty, rusted, 55-gallon drums found on ground surface. No other surficial evidence of contamination, USTs, or stressed vegetation found. One composite soil sample was analyzed for TPH and PCB. PCBs and TPH were detected in a soil sample at concentrations below current comparison criteria.	General Site COPCs may be present beneath the concrete apron. Particularly in this vicinity VOCs and metals are anticipated. Releases to the subsurface may have migrated to groundwater. Overburden groundwater would likely migrate quickly into the adjoining bay.	PCBs EPH VPH SVOCs Metals	PCBs EPH VPH SVOCs Metals	NA	1. Removal of empty drums from the island. 2. Generalized assessment in vicinity and beneath concrete apron to assess soil. 3. General assessment of likely comingled groundwater contamination with downgradient wells. (Combined assessment with NED Site Nos. 28, 30, 32, 33 and 43, groundwater assessed in wells for NED Site Nos. 27, 30, and 31)

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NED Site No.	Navy Structure Name/No./Building Description	History and 1997-2009 Site Observations (SWETS, 1997; Alion 2009)	Source and Anticipated Extent of Contamination	Contaminants of Potential Concern (COPCs)			Credere Identified Data Gaps
				Soil	Groundwater	Sediment	
45	Fire Apparatus Houses (hose houses) (2 locations)	First present on 1942 site plan. Also present on 1943 site plan. Only southernmost building shown on 1945 and 1957 drawings, unlabeled. Not present on 1963 site plan. Not evident on August 1942 aerial photo, but present on 1943 aerial photos. Evidence of site could not be found. No visual evidence of site or stressed vegetation found. Location not sampled.	NA	None	None	None	No further action.
46	AA Gun Emplacements/ Possible Underground Ordnance Bunkers (4 locations)	No records related to this. Potential site identified by RIDEM. Bunkers observed in 2009 with no evidence of storage or oil or hazardous materials. No stressed vegetation found. Location not sampled.	NA	None	None	None	No further action.
47	Underground Water Tank #31	Under construction on August 1942 aerial photo. First present on February 4, 1943, plan titled "Fresh & Salt Water Lines". Labeled "500,000 Gals. Water Tank" with lines leading from water treatment plant building. Present on all subsequent site plans. Present on 1943 aerial photos. Site (top of large underground concrete tank) mostly covered with shallow water at time of inspection. Pump house still standing. Pipe vent and manhole entrance to tank found. Tank empty except trace water on bottom, concrete bottom visible about 15 feet below top of manhole. No evidence of contamination found. Little water present in tank, insufficient depth to sample. No visual/olfactory evidence of contamination. Location not sampled.	NA	None	None	None	Navy Easement – Not FUDS eligible. No further action.
48	Debris Stockpiles	Stockpile is visible in 1943 aerial photograph. Contents of the stockpile are not discernable.	Due to the unknown contents of the stockpile, COPCs may have been buried in place or would be expected to be encountered in surface soil, the vadose zone, at the groundwater interface, or above confining layers. Liquid free product from possible containers disposed here may be present at the groundwater interface as LNAPL or deeper above confining layers, bedrock or within bedrock fractures for DNAPL. Based on the age of the releases, any free-product is presumed to be in a mature stage of plume stability.	VOCs SVOCs PCBs Metals EPH VPH Explosives	VOCs SVOCs PCBs Metals EPH VPH Explosives	None	1. Assess the presence of buried waste through geophysics. 2. Assess the presence of COPCs associated with possible waste stockpiling in soil and groundwater.

Notes:
PAHs – polycyclic aromatic hydrocarbons
COPC – contaminant of potential concern
PEL – predicted effect level
NAPL – non-aqueous phase liquid
TSCA – Toxic Substances Control Act
EPH – extractable petroleum hydrocarbons
PCB – polychlorinated biphenyl
VOC – volatile organic compound
DNAPL – dense NAPL
VPH – volatile petroleum hydrocarbons
UST – underground storage tank
TEL – threshold effect level
EOD – Explosive Ordnance Division
LNAPL – light NAPL
SVOC – semi-volatile organic compounds
RIDEM – Rhode Island Department of Environmental Management
BTEX – benzene, toluene, ethylbenzene, xylenes
"Metals" list currently is considered to include priority pollutant metals (Sb, As, Be, Cd, Cr, Cu, Pb, Ni. Se, Ag, Tl, Zn) and hexavalent chromium

Appendix B
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Gould Island (D01RI033800)
Narragansett Bay, Jamestown, Rhode Island

"Current comparison criteria" referenced herein is the U.S. EPA Regional Screening Levels (RSLs; Target Hazard Quotient [THQ] of 0.1) and the RIDEM Direct Exposure Soil criteria.
1919 Site Plan: Gould Island Conditions, June 30, 1919
1942 Site Plan: Topographic Survey, P.W. Drawing No. 5496-61
1943 Site Plan: Gould Island, U.S. Naval Torpedo Station, U.S. Naval Air Facility, Newport, RI Showing Conditions on June 30, 1943
1945 Site Plan: Gould Island, U.S. Naval Torpedo Station, U.S. Naval Air Facility, Newport, RI Showing Conditions on June 30, 1945
1957 Site Plan: Master Shore Station Development Plan, General Development Plan, Existing and Planned Peacetime Conditions as of December 31, 1957
1963 Site Plan: General Development Plan, Existing and Planned Pre-M-Day, Gould Island site plan dated October 4, 1963

Appendix B
Generalize Conceptual Site Model Flow Chart
Gould Island Site
Narragansett Bay, Jamestown, Rhode Island

APPENDIX C

LABORATORY DOD-ELAP CERTIFICATION DOCUMENTATION

PERRY JOHNSON LABORATORY ACCREDITATION, INC.

Certificate of Accreditation

Perry Johnson Laboratory Accreditation, Inc. has assessed the Laboratory of:

Absolute Resource Associates

124 Heritage Ave. Unit 16, Portsmouth, NH 03801

(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 5.1 January 2017 and is accredited is accordance with the:

United States Department of Defense
Environmental Laboratory Accreditation Program (DoD-ELAP)
EPA NLLAP-LQSR Revision 3.0

This accreditation demonstrates technical competence for the defined scope:
Environmental Testing
(As detailed in the supplement)

Accreditation claims for such testing and/or calibration services shall only be made from addresses referenced within this certificate. 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:

Tracy Szerszen
President/Operations Manager

Perry Johnson Laboratory
Accreditation, Inc. (PJLA)
755 W. Big Beaver, Suite 1325
Troy, Michigan 48084

Initial Accreditation Date:	*Issue Date:*	*Expiration Date:*
May 12, 2014	July 10, 2017	July 31, 2019

Accreditation No.:	*Certificate No.:*
78237	L17-282

The validity of this certificate is maintained through ongoing assessments based on a continuous accreditation cycle. The validity of this certificate should be confirmed through the PJLA website: www.pjlabs.com

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP
EPA NLLAP-LQSR Revision 3.0

Absolute Resource Associates

124 Heritage Ave. Unit 16, Portsmouth, NH 03801
Contact Name: Jennifer Guerette Phone: 603-436-2001

Accreditation is granted to the facility to perform the following testing:

ISO/IEC 17025:2005 and EPA NLLAP LQSR Version 3.0

FIELD OF TEST	ITEMS, MATERIALS OR PRODUCTS TESTED	SPECIFIC TESTS OR PROPERTIES MEASURED	SPECIFICATION, STANDARD METHOD OR TECHNIQUE USED
Environmental	Air	Lead	NIOSH 7303
	Air		EPA 6020A
	TCLP Leachates		EPA 3005A
	Water, Solid & Hazardous Waste	TCLP	EPA 1311

ISO/IEC 17025:2005 and DoD ELAP

Matrix	Standard/Method	Technology	Analyte
Aqueous	EPA 8081B	GC-ECD	Chlordane (technical)
Aqueous	EPA 8260C	GC-MS	4-Isopropyltolune
Aqueous	EPA 8260C	GC-MS	Ethyl-t-butylether (ETBE)
Aqueous	EPA 8260C	GC-MS	N-propylbenzene
Aqueous	EPA 8260C	GC-MS	tert-Butyl alcohol
Aqueous	EPA 7470A	CVAA	Mercury
Drinking Water	EPA 524.2	GC-MS	Bromodichloromethane
Drinking Water	EPA 524.2	GC-MS	Bromoform
Drinking Water	EPA 524.2	GC-MS	Chlorodibromomethane
Drinking Water	EPA 524.2	GC-MS	Chloroform
Drinking Water	EPA 524.2	GC-MS	1,1,1,2-Tetrachloroethane
Drinking Water	EPA 524.2	GC-MS	1,1,1-Trichloroethane
Drinking Water	EPA 524.2	GC-MS	1,1,2,2-Tetrachloroethane
Drinking Water	EPA 524.2	GC-MS	1,1,2-Trichloroethane
Drinking Water	EPA 524.2	GC-MS	1,1-Dichloro-2-propanone
Drinking Water	EPA 524.2	GC-MS	1,1-Dichloroethane
Drinking Water	EPA 524.2	GC-MS	1,1-Dichloroethylene
Drinking Water	EPA 524.2	GC-MS	1,1-Dichloropropene
Drinking Water	EPA 524.2	GC-MS	1,2,3-Trichlorobenzene
Drinking Water	EPA 524.2	GC-MS	1,2,3-Trichloropropane
Drinking Water	EPA 524.2	GC-MS	1,2,4-Trichlorobenzene
Drinking Water	EPA 524.2	GC-MS	1,2,4-Trimethylbenzene

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Matrix	Standard/Method	Technology	Analyte
Drinking Water	EPA 524.2	GC-MS	1,2-Dibromo-3-chloropropane (DBCP)
Drinking Water	EPA 524.2	GC-MS	1,2-Dibromoethane (EDB)
Drinking Water	EPA 524.2	GC-MS	1,2-Dichlorobenzene
Drinking Water	EPA 524.2	GC-MS	1,2-Dichloroethane
Drinking Water	EPA 524.2	GC-MS	1,2-Dichloropropane
Drinking Water	EPA 524.2	GC-MS	1,3,5-Trimethylbenzene
Drinking Water	EPA 524.2	GC-MS	1,3-Dichlorobenzene
Drinking Water	EPA 524.2	GC-MS	1,3-Dichloropropane
Drinking Water	EPA 524.2	GC-MS	1,4-Dichlorobenzene
Drinking Water	EPA 524.2	GC-MS	2,2-Dichloropropane
Drinking Water	EPA 524.2	GC-MS	2-Chlorotoluene
Drinking Water	EPA 524.2	GC-MS	4-Chlorotoluene
Drinking Water	EPA 524.2	GC-MS	4-Isopropyltoluene
Drinking Water	EPA 524.2	GC-MS	Benzene
Drinking Water	EPA 524.2	GC-MS	Bromobenzene
Drinking Water	EPA 524.2	GC-MS	Bromochloromethane
Drinking Water	EPA 524.2	GC-MS	Bromodichloromethane
Drinking Water	EPA 524.2	GC-MS	Bromoform
Drinking Water	EPA 524.2	GC-MS	Bromomethane
Drinking Water	EPA 524.2	GC-MS	c 1,3-Dichloropropene
Drinking Water	EPA 524.2	GC-MS	c-1,2-Dichloroethene
Drinking Water	EPA 524.2	GC-MS	Carbon Disulfide
Drinking Water	EPA 524.2	GC-MS	Carbon Tetrachloride
Drinking Water	EPA 524.2	GC-MS	Chlorobenzene
Drinking Water	EPA 524.2	GC-MS	Chloroethane
Drinking Water	EPA 524.2	GC-MS	Chloroform
Drinking Water	EPA 524.2	GC-MS	Chloromethane
Drinking Water	EPA 524.2	GC-MS	Dibromochloromethane
Drinking Water	EPA 524.2	GC-MS	Dibromomethane
Drinking Water	EPA 524.2	GC-MS	Dichlorodifluoromethane

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Matrix	Standard/Method	Technology	Analyte
Drinking Water	EPA 524.2	GC-MS	Di-isopropylether (DIPE)
Drinking Water	EPA 524.2	GC-MS	Ethylbenzene
Drinking Water	EPA 524.2	GC-MS	Ethyl-t-butylether (ETBE)
Drinking Water	EPA 524.2	GC-MS	Hexachlorobutadiene
Drinking Water	EPA 524.2	GC-MS	Isopropylbenzene
Drinking Water	EPA 524.2	GC-MS	M&P Xylenes
Drinking Water	EPA 524.2	GC-MS	Methylene Chloride
Drinking Water	EPA 524.2	GC-MS	Methyl t-Butyl Ether (MTBE)
Drinking Water	EPA 524.2	GC-MS	Naphthalene
Drinking Water	EPA 524.2	GC-MS	n-Butylbenzene
Drinking Water	EPA 524.2	GC-MS	n-Propylbenzene
Drinking Water	EPA 524.2	GC-MS	O-xylene
Drinking Water	EPA 524.2	GC-MS	sec-Butylbenzene
Drinking Water	EPA 524.2	GC-MS	Styrene
Drinking Water	EPA 524.2	GC-MS	t 1,2-Dichloroethylene
Drinking Water	EPA 524.2	GC-MS	t 1,3-Dichloropropene
Drinking Water	EPA 524.2	GC-MS	T-amylmethylether (TAME)
Drinking Water	EPA 524.2	GC-MS	tert-Butyl alcohol (TBA)
Drinking Water	EPA 524.2	GC-MS	tert-Butylbenzene
Drinking Water	EPA 524.2	GC-MS	Tetrachloroethylene
Drinking Water	EPA 524.2	GC-MS	Toluene
Drinking Water	EPA 524.2	GC-MS	Total Trihalomethanes
Drinking Water	EPA 524.2	GC-MS	Total Xylenes
Drinking Water	EPA 524.2	GC-MS	Trichloroethylene
Drinking Water	EPA 524.2	GC-MS	Trichlorofluoromethane
Drinking Water	EPA 524.2	GC-MS	Trichlorotrifluoroethane (Freon 113)
Drinking Water	EPA 524.2	GC-MS	Vinyl Chloride
Solid	EPA 7471B	CVAA	Mercury
Solid	SM 2540B	Gravimetric	Total Solids
Solid	SM 2540G	Gravimetric	Total Fixed and Volatile Solids
Aqueous/Solid	EPA 6020A	ICP-MS	Aluminum
Aqueous/Solid	EPA 6020A	ICP-MS	Antimony
Aqueous/Solid	EPA 6020A	ICP-MS	Arsenic
Aqueous/Solid	EPA 6020A	ICP-MS	Barium
Aqueous/Solid	EPA 6020A	ICP-MS	Beryllium

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP
EPA NLLAP-LQSR Revision 3.0

Absolute Resource Associates

124 Heritage Ave. Unit 16, Portsmouth, NH 03801
Contact Name: Jennifer Guerette Phone: 603-436-2001

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Aqueous/Solid	EPA 6020A	ICP-MS	Boron
Aqueous/Solid	EPA 6020A	ICP-MS	Cadmium
Aqueous/Solid	EPA 6020A	ICP-MS	Calcium
Aqueous/Solid	EPA 6020A	ICP-MS	Chromium
Aqueous/Solid	EPA 6020A	ICP-MS	Cobalt
Aqueous/Solid	EPA 6020A	ICP-MS	Copper
Aqueous/Solid	EPA 6020A	ICP-MS	Iron
Aqueous/Solid	EPA 6020A	ICP-MS	Lead
Aqueous/Solid	EPA 6020A	ICP-MS	Magnesium
Aqueous/Solid	EPA 6020A	ICP-MS	Manganese
Aqueous/Solid	EPA 6020A	ICP-MS	Molybdenum
Aqueous/Solid	EPA 6020A	ICP-MS	Nickel
Aqueous/Solid	EPA 6020A	ICP-MS	Potassium
Aqueous/Solid	EPA 6020A	ICP-MS	Selenium
Aqueous/Solid	EPA 6020A	ICP-MS	Silicon
Aqueous/Solid	EPA 6020A	ICP-MS	Silver
Aqueous/Solid	EPA 6020A	ICP-MS	Sodium
Aqueous/Solid	EPA 6020A	ICP-MS	Strontium
Aqueous/Solid	EPA 6020A	ICP-MS	Thallium
Aqueous/Solid	EPA 6020A	ICP-MS	Tin
Aqueous/Solid	EPA 6020A	ICP-MS	Titanium
Aqueous/Solid	EPA 6020A	ICP-MS	Vanadium
Aqueous/Solid	EPA 6020A	ICP-MS	Zinc
Aqueous/Solid	EPA 6020A	ICP-MS	Hardness (by calculation)
Aqueous/Solid	EPA 8015D	GC-FID	Diesel Range Organics (DRO)
Aqueous/Solid	EPA 8015D	GC-FID	Gasoline Range Organics (GRO)
Aqueous/Solid	EPA 8081B	GC-ECD	Aldrin
Aqueous/Solid	EPA 8081B	GC-ECD	alpha-BHC
Aqueous/Solid	EPA 8081B	GC-ECD	alpha-Chlordane
Aqueous/Solid	EPA 8081B	GC-ECD	beta-BHC
Aqueous/Solid	EPA 8081B	GC-ECD	DDD (4,4)
Aqueous/Solid	EPA 8081B	GC-ECD	DDE (4,4)
Aqueous/Solid	EPA 8081B	GC-ECD	DDT (4,4)
Aqueous/Solid	EPA 8081B	GC-ECD	delta-BHC
Aqueous/Solid	EPA 8081B	GC-ECD	Dieldrin
Aqueous/Solid	EPA 8081B	GC-ECD	Endosulfan I

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124 Heritage Ave. Unit 16, Portsmouth, NH 03801
Contact Name: Jennifer Guerette Phone: 603-436-2001

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Aqueous/Solid	EPA 8081B	GC-ECD	Endosulfan II
Aqueous/Solid	EPA 8081B	GC-ECD	Endosulfan sulfate
Aqueous/Solid	EPA 8081B	GC-ECD	Endrin
Aqueous/Solid	EPA 8081B	GC-ECD	Endrin aldehyde
Aqueous/Solid	EPA 8081B	GC-ECD	Endrin ketone
Aqueous/Solid	EPA 8081B	GC-ECD	gamma-BHC (Lindane)
Aqueous/Solid	EPA 8081B	GC-ECD	gamma-Chlordane
Aqueous/Solid	EPA 8081B	GC-ECD	Heptachlor
Aqueous/Solid	EPA 8081B	GC-ECD	Heptachlor Epoxide (beta)
Aqueous/Solid	EPA 8081B	GC-ECD	Methoxychlor
Aqueous/Solid	EPA 8082A	GC-ECD	Aroclor 1016
Aqueous/Solid	EPA 8082A	GC-ECD	Aroclor 1016/1242
Aqueous/Solid	EPA 8082A	GC-ECD	Aroclor 1221
Aqueous/Solid	EPA 8082A	GC-ECD	Aroclor 1232
Aqueous/Solid	EPA 8082A	GC-ECD	Aroclor 1242
Aqueous/Solid	EPA 8082A	GC-ECD	Aroclor 1248
Aqueous/Solid	EPA 8082A	GC-ECD	Aroclor 1254
Aqueous/Solid	EPA 8082A	GC-ECD	Aroclor 1260
Aqueous/Solid	EPA 8082A	GC-ECD	Aroclor 1268
Aqueous/Solid	EPA 8082A	GC-ECD	Aroclor-1262
Aqueous/Solid	EPA 8260C	GC-MS	1,1,1,2-Tetrachloroethane
Aqueous/Solid	EPA 8260C	GC-MS	1,1,1-Trichloroethane
Aqueous/Solid	EPA 8260C	GC-MS	1,1,2,2-Tetrachloroethane
Aqueous/Solid	EPA 8260C	GC-MS	1,1,2-Trichloroethane
Aqueous/Solid	EPA 8260C	GC-MS	1,1-Dichloroethane
Aqueous/Solid	EPA 8260C	GC-MS	1,1-Dichloroethene
Aqueous/Solid	EPA 8260C	GC-MS	1,1-Dichloropropene
Aqueous/Solid	EPA 8260C	GC-MS	1,2 Dichlorobenzene
Aqueous/Solid	EPA 8260C	GC-MS	1,2 Dichloroethane
Aqueous/Solid	EPA 8260C	GC-MS	1,2,3-Trichlorobenzene
Aqueous/Solid	EPA 8260C	GC-MS	1,2,3-Trichloropropane
Aqueous/Solid	EPA 8260C	GC-MS	1,2,4-Trichlorobenzene
Aqueous/Solid	EPA 8260C	GC-MS	1,2,4-Trimethylbenzene
Aqueous/Solid	EPA 8260C	GC-MS	1,2-Dibromo-3-chloropropane (DBCP)
Aqueous/Solid	EPA 8260C	GC-MS	1,2-Dibromoethane (EDB)
Aqueous/Solid	EPA 8260C	GC-MS	1,2-Dichloropropane

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP
EPA NLLAP-LQSR Revision 3.0

Absolute Resource Associates

124 Heritage Ave. Unit 16, Portsmouth, NH 03801
Contact Name: Jennifer Guerette Phone: 603-436-2001

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Aqueous/Solid	EPA 8260C	GC-MS	1,3 Dichlorobenzene
Aqueous/Solid	EPA 8260C	GC-MS	1,3,5-Trimethylbenzene
Aqueous/Solid	EPA 8260C	GC-MS	1,3-Dichloropropane
Aqueous/Solid	EPA 8260C	GC-MS	1,4 Dichlorobenzene
Aqueous/Solid	EPA 8260C	GC-MS	2,2-Dichloropropane
Aqueous/Solid	EPA 8260C	GC-MS	2-Butanone (MEK)
Aqueous/Solid	EPA 8260C	GC-MS	2-Chloroethylvinylether
Aqueous/Solid	EPA 8260C	GC-MS	2-Chlorotoluene
Aqueous/Solid	EPA 8260C	GC-MS	2-Hexanone
Aqueous/Solid	EPA 8260C	GC-MS	4-Chlorotoluene
Aqueous/Solid	EPA 8260C	GC-MS	4-Methyl-2-pentanone (MIBK)
Aqueous/Solid	EPA 8260C	GC-MS	Acetone
Aqueous/Solid	EPA 8260C	GC-MS	Benzene
Aqueous/Solid	EPA 8260C	GC-MS	Bromobenzene
Aqueous/Solid	EPA 8260C	GC-MS	Bromochloromethane
Aqueous/Solid	EPA 8260C	GC-MS	Bromodichloromethane
Aqueous/Solid	EPA 8260C	GC-MS	Bromoform
Aqueous/Solid	EPA 8260C	GC-MS	Bromomethane
Aqueous/Solid	EPA 8260C	GC-MS	Carbon disulfide
Aqueous/Solid	EPA 8260C	GC-MS	Carbon tetrachloride
Aqueous/Solid	EPA 8260C	GC-MS	Chlorobenzene
Aqueous/Solid	EPA 8260C	GC-MS	Chloroform
Aqueous/Solid	EPA 8260C	GC-MS	Chloromethane
Aqueous/Solid	EPA 8260C	GC-MS	cis-1,2-Dichloroethene
Aqueous/Solid	EPA 8260C	GC-MS	cis-1,3-Dichloropropylene
Aqueous/Solid	EPA 8260C	GC-MS	Dibromochloromethane
Aqueous/Solid	EPA 8260C	GC-MS	Dibromomethane
Aqueous/Solid	EPA 8260C	GC-MS	Dichlorodifluoromethane
Aqueous/Solid	EPA 8260C	GC-MS	Di-isopropyl ether (DIPE)
Aqueous/Solid	EPA 8260C	GC-MS	Ethylbenzene
Aqueous/Solid	EPA 8260C	GC-MS	Hexachlorobutadiene
Aqueous/Solid	EPA 8260C	GC-MS	Isopropylbenzene
Aqueous/Solid	EPA 8260C	GC-MS	M&p-Xylenes
Aqueous/Solid	EPA 8260C	GC-MS	Methyl tert-butyl ether (MTBE)
Aqueous/Solid	EPA 8260C	GC-MS	Methylene Chloride
Aqueous/Solid	EPA 8260C	GC-MS	Naphthalene

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP
EPA NLLAP-LQSR Revision 3.0

Absolute Resource Associates

124 Heritage Ave. Unit 16, Portsmouth, NH 03801
Contact Name: Jennifer Guerette Phone: 603-436-2001

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Aqueous/Solid	EPA 8260C	GC-MS	n-Butylbenzene
Aqueous/Solid	EPA 8260C	GC-MS	o-Xylene
Aqueous/Solid	EPA 8260C	GC-MS	sec-Butylbenzene
Aqueous/Solid	EPA 8260C	GC-MS	Styrene
Aqueous/Solid	EPA 8260C	GC-MS	TAME
Aqueous/Solid	EPA 8260C	GC-MS	tert-Butylbenzene
Aqueous/Solid	EPA 8260C	GC-MS	Tetrachloroethene
Aqueous/Solid	EPA 8260C	GC-MS	Toluene
Aqueous/Solid	EPA 8260C	GC-MS	trans-1,2-Dichloroethene
Aqueous/Solid	EPA 8260C	GC-MS	trans-1,3-Dichloropropene
Aqueous/Solid	EPA 8260C	GC-MS	Trichloroethene
Aqueous/Solid	EPA 8260C	GC-MS	Trichlorofluoromethane
Aqueous/Solid	EPA 8260C	GC-MS	Vinyl acetate
Aqueous/Solid	EPA 8260C	GC-MS	Vinyl chloride
Aqueous/Solid	EPA 8260C	GC-MS	Xylenes, total
Aqueous/Solid	EPA 8270D	GC-MS	1,2,4-Trichlorobenzene
Aqueous/Solid	EPA 8270D	GC-MS	1,2-Dichlorobenzene
Aqueous/Solid	EPA 8270D	GC-MS	1,3-Dichlorobenzene
Aqueous/Solid	EPA 8270D	GC-MS	1,4-Dichlorobenzene
Aqueous/Solid	EPA 8270D	GC-MS	2,4,5-Trichlorophenol
Aqueous/Solid	EPA 8270D	GC-MS	2,4,6-Trichlorophenol
Aqueous/Solid	EPA 8270D	GC-MS	2,4-Dichlorophenol
Aqueous/Solid	EPA 8270D	GC-MS	2,4-Dimethylphenol
Aqueous/Solid	EPA 8270D	GC-MS	2,4-Dinitrophenol
Aqueous/Solid	EPA 8270D	GC-MS	2,4-Dinitrotoluene
Aqueous/Solid	EPA 8270D	GC-MS	2,6-Dinitrotoluene
Aqueous/Solid	EPA 8270D	GC-MS	2-Chloronaphthalene
Aqueous/Solid	EPA 8270D	GC-MS	2-Chlorophenol
Aqueous/Solid	EPA 8270D	GC-MS	2-Methyl-4,6-Dinitrophenol
Aqueous/Solid	EPA 8270D	GC-MS	2-Methylnaphthalene
Aqueous/Solid	EPA 8270D	GC-MS	2-Methylphenol
Aqueous/Solid	EPA 8270D	GC-MS	2-Nitroaniline
Aqueous/Solid	EPA 8270D	GC-MS	2-Nitrophenol
Aqueous/Solid	EPA 8270D	GC-MS	3,3'-Dichlorobenzidine
Aqueous/Solid	EPA 8270D	GC-MS	3-Nitroaniline
Aqueous/Solid	EPA 8270D	GC-MS	4-Bromophenyl-phenylether

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP
EPA NLLAP-LQSR Revision 3.0

Absolute Resource Associates

124 Heritage Ave. Unit 16, Portsmouth, NH 03801
Contact Name: Jennifer Guerette Phone: 603-436-2001

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Aqueous/Solid	EPA 8270D	GC-MS	4-Chloro-3-methylphenol
Aqueous/Solid	EPA 8270D	GC-MS	4-Chloroaniline
Aqueous/Solid	EPA 8270D	GC-MS	4-Chlorophenyl-phenylether
Aqueous/Solid	EPA 8270D	GC-MS	4-Methylphenol (and/or 3-Methylphenol)
Aqueous/Solid	EPA 8270D	GC-MS	4-Nitroaniline
Aqueous/Solid	EPA 8270D	GC-MS	4-Nitrophenol
Aqueous/Solid	EPA 8270D	GC-MS	Acenaphthene
Aqueous/Solid	EPA 8270D	GC-MS	Acenaphthylene
Aqueous/Solid	EPA 8270D	GC-MS	Aniline
Aqueous/Solid	EPA 8270D	GC-MS	Anthracene
Aqueous/Solid	EPA 8270D	GC-MS	Benzidine
Aqueous/Solid	EPA 8270D	GC-MS	Benzo(a)anthracene
Aqueous/Solid	EPA 8270D	GC-MS	Benzo(a)pyrene
Aqueous/Solid	EPA 8270D	GC-MS	Benzo(b)fluoranthene
Aqueous/Solid	EPA 8270D	GC-MS	Benzo(g,h,i)perylene
Aqueous/Solid	EPA 8270D	GC-MS	Benzo(k)fluoranthene
Aqueous/Solid	EPA 8270D	GC-MS	Benzoic acid
Aqueous/Solid	EPA 8270D	GC-MS	Benzyl alcohol
Aqueous/Solid	EPA 8270D	GC-MS	bis(2-Chloroethoxy)methane
Aqueous/Solid	EPA 8270D	GC-MS	bis(2-Chloroethyl)ether
Aqueous/Solid	EPA 8270D	GC-MS	bis(2-Chloroiospropyl) ether
Aqueous/Solid	EPA 8270D	GC-MS	bis(2-ethylhexyl) phthalate
Aqueous/Solid	EPA 8270D	GC-MS	Butyl benzyl phthalate
Aqueous/Solid	EPA 8270D	GC-MS	Carbazole
Aqueous/Solid	EPA 8270D	GC-MS	Chrysene
Aqueous/Solid	EPA 8270D	GC-MS	Dibenzo(a,h)anthracene
Aqueous/Solid	EPA 8270D	GC-MS	Dibenzofuran
Aqueous/Solid	EPA 8270D	GC-MS	Diethyl phthalate
Aqueous/Solid	EPA 8270D	GC-MS	Dimethyl phthalate
Aqueous/Solid	EPA 8270D	GC-MS	Di-n-butylphthalate
Aqueous/Solid	EPA 8270D	GC-MS	Di-n-octylphthalate
Aqueous/Solid	EPA 8270D	GC-MS	Fluoranthene
Aqueous/Solid	EPA 8270D	GC-MS	Fluorene
Aqueous/Solid	EPA 8270D	GC-MS	Hexachlorobenzene
Aqueous/Solid	EPA 8270D	GC-MS	Hexachlorobutadiene
Aqueous/Solid	EPA 8270D	GC-MS	Hexachlorocyclopentadiene

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP
EPA NLLAP-LQSR Revision 3.0

Absolute Resource Associates

124 Heritage Ave. Unit 16, Portsmouth, NH 03801
Contact Name: Jennifer Guerette Phone: 603-436-2001

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Aqueous/Solid	EPA 8270D	GC-MS	Hexachloroethane
Aqueous/Solid	EPA 8270D	GC-MS	Indeno(1,2,3, cd)pyrene
Aqueous/Solid	EPA 8270D	GC-MS	Isophorone
Aqueous/Solid	EPA 8270D	GC-MS	Naphthalene
Aqueous/Solid	EPA 8270D	GC-MS	Nitrobenzene
Aqueous/Solid	EPA 8270D	GC-MS	N-Nitrosodimethylamine
Aqueous/Solid	EPA 8270D	GC-MS	N-Nitroso-di-n-propylamine
Aqueous/Solid	EPA 8270D	GC-MS	N-Nitrosodiphenylamine
Aqueous/Solid	EPA 8270D	GC-MS	Pentachlorophenol
Aqueous/Solid	EPA 8270D	GC-MS	Phenanthrene
Aqueous/Solid	EPA 8270D	GC-MS	Phenol
Aqueous/Solid	EPA 8270D	GC-MS	Pyrene
Aqueous/Solid	EPA 8270D	GC-MS	Pyridine
Aqueous/Solid	MA EPH	GC-FID	2-Methylnaphthalene
Aqueous/Solid	MA EPH	GC-FID	Acenaphthene
Aqueous/Solid	MA EPH	GC-FID	Acenaphthylene
Aqueous/Solid	MA EPH	GC-FID	Anthracene
Aqueous/Solid	MA EPH	GC-FID	Benzo(a)anthracene
Aqueous/Solid	MA EPH	GC-FID	Benzo(a)pyrene
Aqueous/Solid	MA EPH	GC-FID	Benzo(b)fluoranthene
Aqueous/Solid	MA EPH	GC-FID	Benzo(g,h,i)perylene
Aqueous/Solid	MA EPH	GC-FID	Benzo(k)fluoranthene
Aqueous/Solid	MA EPH	GC-FID	C11-C22 Aromatic Hydrocarbons
Aqueous/Solid	MA EPH	GC-FID	C19-C36 Aliphatic Hydrocarbons
Aqueous/Solid	MA EPH	GC-FID	C9-C18 Aliphatic Hydrocarbons
Aqueous/Solid	MA EPH	GC-FID	Chrysene
Aqueous/Solid	MA EPH	GC-FID	Dibenzo(a,h)anthracene
Aqueous/Solid	MA EPH	GC-FID	Fluoranthene
Aqueous/Solid	MA EPH	GC-FID	Fluorene
Aqueous/Solid	MA EPH	GC-FID	Indeno(1,2,3-cd) pyrene
Aqueous/Solid	MA EPH	GC-FID	Naphthalene
Aqueous/Solid	MA EPH	GC-FID	Phenanthrene
Aqueous/Solid	MA EPH	GC-FID	Pyrene
Aqueous/Solid	MA VPH	GC-FID/PID	Benzene
Aqueous/Solid	MA VPH	GC-FID/PID	C5-C8 Aliphatic Hydrocarbons
Aqueous/Solid	MA VPH	GC-FID/PID	C9-C10 Aromatic Hydrocarbons

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP
EPA NLLAP-LQSR Revision 3.0

Absolute Resource Associates

124 Heritage Ave. Unit 16, Portsmouth, NH 03801
Contact Name: Jennifer Guerette Phone: 603-436-2001

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Aqueous/Solid	MA VPH	GC-FID/PID	C9-C12 Aliphatics
Aqueous/Solid	MA VPH	GC-FID/PID	Ethylbenzene
Aqueous/Solid	MA VPH	GC-FID/PID	m+p-Xylene
Aqueous/Solid	MA VPH	GC-FID/PID	Methyl tert-butyl ether (MTBE)
Aqueous/Solid	MA VPH	GC-FID/PID	Naphthalene
Aqueous/Solid	MA VPH	GC-FID/PID	o-Xylene
Aqueous/Solid	MA VPH	GC-FID/PID	Toluene
Aqueous/Solid	MA VPH	GC-FID/PID	Unadjusted C5-C8 Aliphatic HCs
Aqueous/Solid	MA VPH	GC-FID/PID	Unadjusted C9-C12 Aliphatic HCs
Aqueous/Solid	MA VPH	GC-FID/PID	Xylene, total

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP
EPA NLLAP-LQSR Revision 3.0

Absolute Resource Associates

124 Heritage Ave. Unit 16, Portsmouth, NH 03801
Contact Name: Jennifer Guerette Phone: 603-436-2001

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology
Aqueous	EPA 5030C	Purge-and-Trap for Aqueous Samples
Aqueous	EPA 3005A	Hot Plate
Aqueous	EPA 3510C	Separatory Funnel Extraction
Solid	EPA 5035A	Purge-and-Trap for Aqueous Samples
Solid	EPA 3546	Microwave Extraction
Solid	EPA 3550C	Ultrasonic Extraction
Solid	EPA 3051A	Micro Assisted Acid Digest/Metals
Solid	EPA 3540C	Soxhlet Extraction
Oil	EPA 3580A	Waste Dilution
Aqueous/Solid/Oil	EPA 3660B	Sulfur Clean-up
Aqueous/ Solid & Hazardous Waste	EPA 1311	TCLP Leaching Procedure

LABORATORY ACCREDITATION BUREAU a division of A-S-B

Certificate of Accreditation

ISO/IEC 17025:2005 **Certificate Number L2468**

TestAmerica Sacramento

880 Riverside Parkway
West Sacramento CA 95605

has met the requirements set forth in L-A-B's policies and procedures, all requirements of ISO/IEC 17025:2005 "General Requirements for the competence of Testing and Calibration Laboratories" and the U.S. Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP).*

The accredited lab has demonstrated technical competence to a defined "Scope of Accreditation" and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009).

Accreditation valid through: January 20, 2018

R. Douglas Leonard, Jr., President, COO
Laboratory Accreditation Bureau
Presented the 20th of January 2017

*See the laboratory's Scope of Accreditation for details of accredited parameters
**Laboratory Accreditation Bureau is found to be in compliance with ISO/IEC 17011:2004 and recognized by ILAC (International Laboratory Accreditation Cooperation) and NACLA (National Cooperation for Laboratory Accreditation).
Form 403.14 – Rev 1 7/3/13

Scope of Accreditation
For
TestAmerica Sacramento

880 Riverside Parkway
West Sacramento, CA 95605
Ms. Lisa Stafford
916-373-5600

In recognition of a successful assessment to ISO/IEC 17025:2005 and the requirements of the DoD Environmental Laboratory Accreditation Program (LABPR 403 DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (DoD QSM V5) based on the TNI Standard - Environmental Laboratory Sector, Volume 1 – Management and Technical Requirements for Laboratories Performing Environmental Analysis, Sept 2009 (EL-V1-2009); accreditation is granted to **TestAmerica Sacramento** to perform the following tests:

Accreditation granted through: **January 20, 2018**

Testing - Environmental

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP-AES	EPA 6010B/6010C	Aluminum
ICP-AES	EPA 6010B/6010C	Antimony
ICP-AES	EPA 6010B/6010C	Arsenic
ICP-AES	EPA 6010B/6010C	Barium
ICP-AES	EPA 6010B/6010C	Beryllium
ICP-AES	EPA 6010B/6010C	Boron
ICP-AES	EPA 6010B/6010C	Cadmium
ICP-AES	EPA 6010B/6010C	Calcium
ICP-AES	EPA 6010B/6010C	Chromium (Total)
ICP-AES	EPA 6010B/6010C	Cobalt
ICP-AES	EPA 6010B/6010C	Copper
ICP-AES	EPA 6010B/6010C	Iron
ICP-AES	EPA 6010B/6010C	Lead
ICP-AES	EPA 6010B/6010C	Magnesium
ICP-AES	EPA 6010B/6010C	Manganese
ICP-AES	EPA 6010B/6010C	Molybdenum
ICP-AES	EPA 6010B/6010C	Nickel
ICP-AES	EPA 6010B/6010C	Potassium

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP-AES	EPA 6010B/6010C	Selenium
ICP-AES	EPA 6010B/6010C	Silica
ICP-AES	EPA 6010B/6010C	Silicon
ICP-AES	EPA 6010B/6010C	Silver
ICP-AES	EPA 6010B/6010C	Sodium
ICP-AES	EPA 6010B/6010C	Thallium
ICP-AES	EPA 6010B/6010C	Tin
ICP-AES	EPA 6010B/6010C	Titanium
ICP-AES	EPA 6010B/6010C	Vanadium
ICP-AES	EPA 6010B/6010C	Zinc
ICP-MS	EPA 6020/6020A	Aluminum
ICP-MS	EPA 6020/6020A	Antimony
ICP-MS	EPA 6020/6020A	Arsenic
ICP-MS	EPA 6020/6020A	Barium
ICP-MS	EPA 6020/6020A	Beryllium
ICP-MS	EPA 6020/6020A	Cadmium
ICP-MS	EPA 6020/6020A	Calcium
ICP-MS	EPA 6020/6020A	Chromium (Total)
ICP-MS	EPA 6020/6020A	Cobalt
ICP-MS	EPA 6020/6020A	Copper
ICP-MS	EPA 6020/6020A	Iron
ICP-MS	EPA 6020/6020A	Lead
ICP-MS	EPA 6020/6020A	Magnesium
ICP-MS	EPA 6020/6020A	Manganese
ICP-MS	EPA 6020/6020A	Molybdenum
ICP-MS	EPA 6020/6020A	Nickel
ICP-MS	EPA 6020/6020A	Phosphorus
ICP-MS	EPA 6020/6020A	Potassium
ICP-MS	EPA 6020/6020A	Selenium
ICP-MS	EPA 6020/6020A	Silver
ICP-MS	EPA 6020/6020A	Sodium
ICP-MS	EPA 6020/6020A	Strontium
ICP-MS	EPA 6020/6020A	Thallium
ICP-MS	EPA 6020/6020A	Tin
ICP-MS	EPA 6020/6020A	Titanium
ICP-MS	EPA 6020/6020A	Uranium
ICP-MS	EPA 6020/6020A	Vanadium
ICP-MS	EPA 6020/6020A	Zinc
CVAAS	EPA 7470A	Mercury

Non-Potable Water		
Technology	**Method**	**Analyte**
Colorimetric	EPA 353.2	Nitrate
Colorimetric	EPA 353.2	Nitrate-nitrite
Colorimetric	EPA 353.2	Nitrite
Colorimetric	EPA 410.4	Chemical Oxygen Demand (COD)
LC/MS/MS	EPA 6850	Perchlorate
Colorimetric	EPA 7196A	Chromium (Hexavalent)
Probe	EPA 9040B/9040C	pH
Ion Chromatography	EPA 9056A/300.0	Bromide
Ion Chromatography	EPA 9056A/300.0	Chloride
Ion Chromatography	EPA 9056A/300.0	Fluoride
Ion Chromatography	EPA 9056A/300.0	Nitrate
Ion Chromatography	EPA 9056A/300.0	Nitrite
Ion Chromatography	EPA 9056A/300.0	Orthophosphate
Ion Chromatography	EPA 9056A/300.0	Sulfate
Titration	SM 2320B	Alkalinity
Gravimetric	SM 2540B	Solids, Total
Gravimetric	SM 2540C	Solids, Total Dissolved
Gravimetric	SM 2540D	Solids, Total Suspended
Colorimetric/Hydrolysis	EPA 353.2 Modified / WS-WC-0050	Nitrocellulose
GC/MS	EPA 8260B/8260C	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B/8260C	1,1,1-Trichloroethane
GC/MS	EPA 8260B/8260C	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B/8260C	1,1,2-Trichloroethane
GC/MS	EPA 8260B/8260C	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA 8260B/8260C	1,1-Dichloroethane
GC/MS	EPA 8260B/8260C	1,1-Dichloroethene
GC/MS	EPA 8260B/8260C	1,1-Dichloropropene
GC/MS	EPA 8260B/8260C	1,2,3-Trichlorobenzene
GC/MS	EPA 8260B/8260C	1,2,3-Trichloropropane
GC/MS	EPA 8260B/8260C	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B/8260C	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B/8260C	1,2-Dibromo-3-chloropropane
GC/MS	EPA 8260B/8260C	1,2-Dibromoethane
GC/MS	EPA 8260B/8260C	1,2-Dichlorobenzene
GC/MS	EPA 8260B/8260C	1,2-Dichloroethane
GC/MS	EPA 8260B/8260C	1,2-Dichloropropane
GC/MS	EPA 8260B/8260C	1,3,5-Trimethylbenzene
GC/MS	EPA 8260B/8260C	1,3-Dichlorobenzene
GC/MS	EPA 8260B/8260C	1,3-Dichloropropane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/8260C	1,4-Dichlorobenzene
GC/MS	EPA 8260B/8260C	1-Chlorohexane
GC/MS	EPA 8260B/8260C	2,2-Dichloropropane
GC/MS	EPA 8260B/8260C	2-Butanone (MEK)
GC/MS	EPA 8260B/8260C	2-Chlorotoluene
GC/MS	EPA 8260B/8260C	2-Hexanone (MBK)
GC/MS	EPA 8260B/8260C	2-Methyl-2-propanol (tert- Butyl Alcohol, TBA)
GC/MS	EPA 8260B/8260C	4-Chlorotoluene
GC/MS	EPA 8260B/8260C	4-Isopropyltoluene
GC/MS	EPA 8260B/8260C	4-Methyl-2-pentanone (MIBK)
GC/MS	EPA 8260B/8260C	Acetone
GC/MS	EPA 8260B/8260C	Allyl Chloride
GC/MS	EPA 8260B/8260C	Benzene
GC/MS	EPA 8260B/8260C	Bromobenzene
GC/MS	EPA 8260B/8260C	Bromochloromethane
GC/MS	EPA 8260B/8260C	Bromodichloromethane
GC/MS	EPA 8260B/8260C	Bromoform
GC/MS	EPA 8260B/8260C	Bromomethane
GC/MS	EPA 8260B/8260C	Carbon Disulfide
GC/MS	EPA 8260B/8260C	Carbon Tetrachloride
GC/MS	EPA 8260B/8260C	Chlorobenzene
GC/MS	EPA 8260B/8260C	Chloroethane
GC/MS	EPA 8260B/8260C	Chloroform
GC/MS	EPA 8260B/8260C	Chloromethane
GC/MS	EPA 8260B/8260C	cis-1,2-Dichloroethene
GC/MS	EPA 8260B/8260C	cis-1,3-Dichloropropene
GC/MS	EPA 8260B/8260C	Cyclohexane
GC/MS	EPA 8260B/8260C	Dibromochloromethane
GC/MS	EPA 8260B/8260C	Dibromomethane
GC/MS	EPA 8260B/8260C	Dichlorodifluoromethane
GC/MS	EPA 8260B/8260C	Diisopropyl Ether (DIPE)
GC/MS	EPA 8260B/8260C	Ethylbenzene
GC/MS	EPA 8260B/8260C	Ethylmethacrylate
GC/MS	EPA 8260B/8260C	Ethyl tert-butyl Ether (ETBE)
GC/MS	EPA 8260B/8260C	Hexachlorobutadiene
GC/MS	EPA 8260B/8260C	Hexane
GC/MS	EPA 8260B/8260C	Iodomethane
GC/MS	EPA 8260B/8260C	Isobutanol (2-Methyl-1-propanol)
GC/MS	EPA 8260B/8260C	Isopropylbenzene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/8260C	m & p Xylene
GC/MS	EPA 8260B/8260C	Methyl tert-butyl Ether (MTBE)
GC/MS	EPA 8260B/8260C	Methylene Chloride
GC/MS	EPA 8260B/8260C	Naphthalene
GC/MS	EPA 8260B/8260C	n-Butylbenzene
GC/MS	EPA 8260B/8260C	n-Propylbenzene
GC/MS	EPA 8260B/8260C	o-Xylene
GC/MS	EPA 8260B/8260C	sec-Butylbenzene
GC/MS	EPA 8260B/8260C	Styrene
GC/MS	EPA 8260B/8260C	t-Amyl methyl Ether (TAME)
GC/MS	EPA 8260B/8260C	t-1,4-Dichloro-2-Butene
GC/MS	EPA 8260B/8260C	tert-Butylbenzene
GC/MS	EPA 8260B/8260C	Tetrachloroethene
GC/MS	EPA 8260B/8260C	Toluene
GC/MS	EPA 8260B/8260C	trans-1,2-Dichloroethene
GC/MS	EPA 8260B/8260C	trans-1,3-Dichloropropene
GC/MS	EPA 8260B/8260C	Trichloroethene
GC/MS	EPA 8260B/8260C	Trichlorofluoromethane
GC/MS	EPA 8260B/8260C	Vinyl Acetate
GC/MS	EPA 8260B/8260C	Vinyl Chloride
GC/MS	EPA 8260B/8260C	Xylenes, Total
GC/MS	EPA 8260B/AK101MS	Gasoline (GRO)
GC/MS	EPA 8270C/8270D	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270C/8270D	1,2,4-Trichlorobenzene
GC/MS	EPA 8270C/8270D	1,2-Dichlorobenzene
GC/MS	EPA 8270C/8270D	1,2-Diphenylhydrazine (as Azobenzene)
GC/MS	EPA 8270C/8270D	1,3-Dichlorobenzene
GC/MS	EPA 8270C/8270D	1,3-Dinitrobenzene
GC/MS	EPA 8270C/8270D	1,4-Dichlorobenzene
GC/MS	EPA 8270C/8270D	1-Methylnaphthalene
GC/MS	EPA 8270C/8270D	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270C/8270D	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/8270D	2,4,6-Trichlorophenol
GC/MS	EPA 8270C/8270D	2,4-Dichlorophenol
GC/MS	EPA 8270C/8270D	2,4-Dimethylphenol
GC/MS	EPA 8270C/8270D	2,4-Dinitrophenol
GC/MS	EPA 8270C/8270D	2,4-Dinitrotoluene
GC/MS	EPA 8270C/8270D	2,6-Dichlorophenol
GC/MS	EPA 8270C/8270D	2,6-Dinitrotoluene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/8270D	2-Chloronaphthalene
GC/MS	EPA 8270C/8270D	2-Chlorophenol
GC/MS	EPA 8270C/8270D	2-Methylnaphthalene
GC/MS	EPA 8270C/8270D	2-Methylphenol
GC/MS	EPA 8270C/8270D	2-Nitroaniline
GC/MS	EPA 8270C/8270D	2-Nitrophenol
GC/MS	EPA 8270C/8270D	3&4-Methylphenol
GC/MS	EPA 8270C/8270D	3,3'-Dichlorobenzidine
GC/MS	EPA 8270C/8270D	3-Nitroaniline
GC/MS	EPA 8270C/8270D	4,6-Dinitro-2-methylphenol
GC/MS	EPA 8270C/8270D	4-Bromophenyl phenyl ether
GC/MS	EPA 8270C/8270D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/8270D	4-Chloroaniline
GC/MS	EPA 8270C/8270D	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270C/8270D	4-Nitroaniline
GC/MS	EPA 8270C/8270D	4-Nitrophenol
GC/MS	EPA 8270C/8270D	Acenaphthene
GC/MS	EPA 8270C/8270D	Acenaphthylene
GC/MS	EPA 8270C/8270D	Aniline
GC/MS	EPA 8270C/8270D	Anthracene
GC/MS	EPA 8270C/8270D	Benzo(a)anthracene
GC/MS	EPA 8270C/8270D	Benzo(a)pyrene
GC/MS	EPA 8270C/8270D	Benzo(b)fluoranthene
GC/MS	EPA 8270C/8270D	Benzo(g,h,i)perylene
GC/MS	EPA 8270C/8270D	Benzo(k)fluoranthene
GC/MS	EPA 8270C/8270D	Benzoic Acid
GC/MS	EPA 8270C/8270D	Benzyl Alcohol
GC/MS	EPA 8270C/8270D	Benzyl butyl Phthalate
GC/MS	EPA 8270C/8270D	Biphenyl
GC/MS	EPA 8270C/8270D	Bis(2-chloroethoxy) Methane
GC/MS	EPA 8270C/8270D	Bis(2-chloroethyl) Ether
GC/MS	EPA 8270C/8270D	Bis(2-chloroisopropyl) Ether
GC/MS	EPA 8270C/8270D	Carbazole
GC/MS	EPA 8270C/8270D	Chrysene
GC/MS	EPA 8270C/8270D	Bis (2-ethylhexyl) Phthalate
GC/MS	EPA 8270C/8270D	Dibenz(a,h)anthracene
GC/MS	EPA 8270C/8270D	Dibenzofuran
GC/MS	EPA 8270C/8270D	Diethyl Phthalate
GC/MS	EPA 8270C/8270D	Dimethyl Phthalate

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/8270D	Di-n-butyl Phthalate
GC/MS	EPA 8270C/8270D	Di-n-octyl Phthalate
GC/MS	EPA 8270C/8270D	Fluoranthene
GC/MS	EPA 8270C/8270D	Fluorene
GC/MS	EPA 8270C/8270D	Hexachlorobenzene
GC/MS	EPA 8270C/8270D	Hexachlorobutadiene
GC/MS	EPA 8270C/8270D	Hexachlorocyclopentadiene
GC/MS	EPA 8270C/8270D	Hexachloroethane
GC/MS	EPA 8270C/8270D	Indeno(1,2,3-c,d) Pyrene
GC/MS	EPA 8270C/8270D	Isophorone
GC/MS	EPA 8270C/8270D	Naphthalene
GC/MS	EPA 8270C/8270D	Nitrobenzene
GC/MS	EPA 8270C/8270D	n-Nitrosodimethylamine
GC/MS	EPA 8270C/8270D	n-Nitrosodi-n-propylamine
GC/MS	EPA 8270C/8270D	n-Nitrosodiphenylamine
GC/MS	EPA 8270C/8270D	Pentachlorophenol
GC/MS	EPA 8270C/8270D	Phenanthrene
GC/MS	EPA 8270C/8270D	Phenol
GC/MS	EPA 8270C/8270D	Pyrene
GC/MS	EPA 8270C/8270D	Pyridine
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	1-Methylnaphthalene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	2-Methylnaphthalene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Acenaphthene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Acenaphthylene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Anthracene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(a)anthracene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(a)pyrene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(b)fluoranthene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(g,h,i)perylene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(k)fluoranthene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Chrysene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Dibenz(a,h)anthracene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Fluoranthene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Fluorene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Indeno(1,2,3-c,d) Pyrene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Naphthalene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Phenanthrene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Pyrene
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0011	1,4-Dioxane
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	1,4-Dithiane
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	Benzothiazole
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	p-Chlorophenyl methylsulfide
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	p-Chlorophenyl methylsulfoxide
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	p-Chlorophenyl methylsulfone
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	Chloropicrin
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	Acetophenone
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	2-Chloroacetophenone
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	1,4-Oxathiane
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	Dimethyl Disulfide
GC-IT/MS	EPA 521 Modified / WS-MS-0012	N-Nitrosodimethyl amine (NDMA)
GC-FID	EPA 8015B/8015C/8015D AK102	Diesel Range Organics (DRO)
GC-FID	AK103	Residual Range Organics
GC-FID	EPA 8015B/8015C/8015D	Motor Oil Range Organics (MRO)
GC-ECD	EPA 8081A/8081B	Aldrin
GC-ECD	EPA 8081A/8081B	a-BHC
GC-ECD	EPA 8081A/8081B	b-BHC

Non-Potable Water		
Technology	**Method**	**Analyte**
GC-ECD	EPA 8081A/8081B	d-BHC
GC-ECD	EPA 8081A/8081B	g-BHC (Lindane)
GC-ECD	EPA 8081A/8081B	a-Chlordane
GC-ECD	EPA 8081A/8081B	g-Chlordane
GC-ECD	EPA 8081A/8081B	4,4'-DDD
GC-ECD	EPA 8081A/8081B	4,4'-DDE
GC-ECD	EPA 8081A/8081B	4,4'-DDT
GC-ECD	EPA 8081A/8081B	Dieldrin
GC-ECD	EPA 8081A/8081B	Endosulfan I
GC-ECD	EPA 8081A/8081B	Endosulfan II
GC-ECD	EPA 8081A/8081B	Endosulfan sulfate
GC-ECD	EPA 8081A/8081B	Endrin
GC-ECD	EPA 8081A/8081B	Endrin Aldehyde
GC-ECD	EPA 8081A/8081B	Endrin Ketone
GC-ECD	EPA 8081A/8081B	Heptachlor
GC-ECD	EPA 8081A/8081B	Heptachlor Epoxide
GC-ECD	EPA 8081A/8081B	Methoxychlor
GC-ECD	EPA 8081A/8081B	Toxaphene
GC-ECD	EPA 8081A/8081B	Chlordane (technical)
GC-ECD	EPA 8082/8082A	PCB-1016
GC-ECD	EPA 8082/8082A	PCB-1221
GC-ECD	EPA 8082/8082A	PCB-1232
GC-ECD	EPA 8082/8082A	PCB-1242
GC-ECD	EPA 8082/8082A	PCB-1248
GC-ECD	EPA 8082/8082A	PCB-1254
GC-ECD	EPA 8082/8082A	PCB-1260
GC-ECD	EPA 8082/8082A	PCB-1262
GC-ECD	EPA 8082/8082A	PCB-1268
GC/MS	EPA 8280A/8280B	2,3,7,8-TeCDD
GC/MS	EPA 8280A/8280B	1,2,3,7,8-PeCDD
GC/MS	EPA 8280A/8280B	1,2,3,4,7,8-HxCDD
GC/MS	EPA 8280A/8280B	1,2,3,6,7,8-HxCDD
GC/MS	EPA 8280A/8280B	1,2,3,7,8,9-HxCDD
GC/MS	EPA 8280A/8280B	1,2,3,4,6,7,8-HpCDD
GC/MS	EPA 8280A/8280B	OCDD
GC/MS	EPA 8280A/8280B	2,3,7,8-TeCDF
GC/MS	EPA 8280A/8280B	1,2,3,7,8-PeCDF
GC/MS	EPA 8280A/8280B	2,3,4,7,8-PeCDF
GC/MS	EPA 8280A/8280B	1,2,3,4,7,8-HxCDF

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8280A/8280B	1,2,3,6,7,8-HxCDF
GC/MS	EPA 8280A/8280B	1,2,3,7,8,9-HxCDF
GC/MS	EPA 8280A/8280B	2,3,4,6,7,8-HxCDF
GC/MS	EPA 8280A/8280B	1,2,3,4,6,7,8-HpCDF
GC/MS	EPA 8280A/8280B	1,2,3,4,7,8,9-HpCDF
GC/MS	EPA 8280A/8280B	OCDF
GC/MS	EPA 8280A/8280B	Total TCDD
GC/MS	EPA 8280A/8280B	Total PeCDD
GC/MS	EPA 8280A/8280B	Total HxCDD
GC/MS	EPA 8280A/8280B	Total HeptaCDD
GC/MS	EPA 8280A/8280B	Total TCDF
GC/MS	EPA 8280A/8280B	Total PeCDF
GC/MS	EPA 8280A/8280B	Total HxCDF
GC/MS	EPA 8280A/8280B	Total HpCDF
GC/HRMS	EPA 8290/8290A/1613B	2,3,7,8-TeCDD
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,7,8-PeCDD
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,4,7,8-HxCDD
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,6,7,8-HxCDD
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,7,8,9-HxCDD
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,4,6,7,8-HpCDD
GC/HRMS	EPA 8290/8290A/1613B	OCDD
GC/HRMS	EPA 8290/8290A/1613B	2,3,7,8-TeCDF
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,7,8-PeCDF
GC/HRMS	EPA 8290/8290A/1613B	2,3,4,7,8-PeCDF
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,4,7,8-HxCDF
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,6,7,8-HxCDF
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,7,8,9-HxCDF
GC/HRMS	EPA 8290/8290A/1613B	2,3,4,6,7,8-HxCDF
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,4,6,7,8-HpCDF
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,4,7,8,9-HpCDF
GC/HRMS	EPA 8290/8290A/1613B	OCDF
GC/HRMS	EPA 8290/8290A/1613B	Total TCDD
GC/HRMS	EPA 8290/8290A/1613B	Total PeCDD
GC/HRMS	EPA 8290/8290A/1613B	Total HxCDD
GC/HRMS	EPA 8290/8290A/1613B	Total HpCDD
GC/HRMS	EPA 8290/8290A/1613B	Total TCDF
GC/HRMS	EPA 8290/8290A/1613B	Total PeCDF
GC/HRMS	EPA 8290/8290A/1613B	Total HxCDF
GC/HRMS	EPA 8290/8290A/1613B	Total HpCDF

Non-Potable Water		
Technology	**Method**	**Analyte**
HPLC/UV	EPA 8330A/8330B	2-Amino-4,6-dinitrotoluene
HPLC/UV	EPA 8330A/8330B	4-Amino-2,6-dinitrotoluene
HPLC/UV	EPA 8330A/8330B	3,5-Dinitroaniline
HPLC/UV	EPA 8330A/8330B	1,3-Dinitrobenzene
HPLC/UV	EPA 8330A/8330B	2,4-Dinitrotoluene
HPLC/UV	EPA 8330A/8330B	2,6-Dinitrotoluene
HPLC/UV	EPA 8330A/8330B	Glycerol trinitrate (Nitroglycerin)
HPLC/UV	EPA 8330A/8330B	Hexahydro-1,3,5-trinitro- 1,3,5-triazine (Hexogen)
HPLC/UV	EPA 8330A/8330B	Methyl-2,4,6- trinitrophenylnitramine
HPLC/UV	EPA 8330A/8330B	Nitrobenzene
HPLC/UV	EPA 8330A/8330B	2-Nitrotoluene (o-Nitrotoluene)
HPLC/UV	EPA 8330A/8330B	3-Nitrotoluene (m-Nitrotoluene)
HPLC/UV	EPA 8330A/8330B	4-Nitrotoluene (p-Nitrotoluene)
HPLC/UV	EPA 8330A/8330B	Octahydro-1,3,5,7- tetranitro1,3,5,7-tetracine (Octogen)
HPLC/UV	EPA 8330A/8330B	Picric acid
HPLC/UV	EPA 8330A/8330B	Pentaerythritol Tetranitrate
HPLC/UV	EPA 8330A/8330B	1,3,5-Trinitrobenzene
HPLC/UV	EPA 8330A/8330B	2,4,6-Trinitrotoluene
HPLC/UV	EPA 8330A/8330B	Hexahydro-1,3-dinitroso-5- nitro-1,3,5,triazine (DNX)
HPLC/UV	EPA 8330A/8330B	Hexahydro-1,3,5-trinitroso- 1,3,5-triazine (TNX)
HPLC/UV	EPA 8330A/8330B	1-Nitroso-3,5-dinitro-1,3,5- triazacyclohexane (MNX)
HPLC/UV	EPA 8330A Modified /WS-LC-0010	Nitroguanidine
GC-HRMS	EPA 8290 Modified / WS-ID-0021	2-(N-ethylperfluoro-1- octanesulfonamido)-ethanol (N-Et-FOSE)
GC-HRMS	EPA 8290 Modified / WS-ID-0021	2-(N-Methylperfluoro-1- octanesulfonamido)-ethanol (N-Me-FOSE)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	6:2 Fluorotelomer sulfonate (6:2 FTS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	8:2 Fluorotelomer sulfonate (8:2 FTS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	N-Ethyl perfluorooctane sulfonamide (EtFOSA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	N-Ethyl perfluorooctanesulfon amidacetic acid (EtFOSAA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	N-Methyl perfluorooctane sulfonamide (MeFOSA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	N-Methyl perfluorooctanesulfon amidoacetic acid (MeFOSAA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorooctanoic acid (PFOA)

Non-Potable Water		
Technology	**Method**	**Analyte**
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorooctane Sulfonic Acid (PFOS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorobutyric acid (PFBA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluoropentanoic acid (PFPA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorohexanoic acid (PFHxA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluoroheptanoic acid (PFHpA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorononanoic acid (PFNA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorodecanoic acid (PFDA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluoroundecanoic acid (PFUDA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorododecanoic acid (PFDoDA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorotridecanoic acid (PFTriA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorotetradecanoic acid (PDTeA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorobutane Sulfonic Acid (PFBS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorohexane Sulfonic Acid (PFHxS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluoroheptane Sulfonic Acid (PFHpS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorodecane Sulfonic Acid (PFDS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluoroocatane Sulfonamide (FOSA)
GC/HRMS	EPA 1668A/1668C	PCB 1
GC/HRMS	EPA 1668A/1668C	PCB 2
GC/HRMS	EPA 1668A/1668C	PCB 3
GC/HRMS	EPA 1668A/1668C	PCB 4
GC/HRMS	EPA 1668A/1668C	PCB 5
GC/HRMS	EPA 1668A/1668C	PCB 6
GC/HRMS	EPA 1668A/1668C	PCB 7
GC/HRMS	EPA 1668A/1668C	PCB 8
GC/HRMS	EPA 1668A/1668C	PCB 9
GC/HRMS	EPA 1668A/1668C	PCB 10
GC/HRMS	EPA 1668A/1668C	PCB 11
GC/HRMS	EPA 1668A/1668C	PCB 12

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 13
GC/HRMS	EPA 1668A/1668C	PCB 14
GC/HRMS	EPA 1668A/1668C	PCB 15
GC/HRMS	EPA 1668A/1668C	PCB 16
GC/HRMS	EPA 1668A/1668C	PCB 17
GC/HRMS	EPA 1668A/1668C	PCB 18
GC/HRMS	EPA 1668A/1668C	PCB 19
GC/HRMS	EPA 1668A/1668C	PCB 20
GC/HRMS	EPA 1668A/1668C	PCB 21
GC/HRMS	EPA 1668A/1668C	PCB 22
GC/HRMS	EPA 1668A/1668C	PCB 23
GC/HRMS	EPA 1668A/1668C	PCB 24
GC/HRMS	EPA 1668A/1668C	PCB 25
GC/HRMS	EPA 1668A/1668C	PCB 26
GC/HRMS	EPA 1668A/1668C	PCB 27
GC/HRMS	EPA 1668A/1668C	PCB 28
GC/HRMS	EPA 1668A/1668C	PCB 29
GC/HRMS	EPA 1668A/1668C	PCB 30
GC/HRMS	EPA 1668A/1668C	PCB 32
GC/HRMS	EPA 1668A/1668C	PCB 31
GC/HRMS	EPA 1668A/1668C	PCB 33
GC/HRMS	EPA 1668A/1668C	PCB 34
GC/HRMS	EPA 1668A/1668C	PCB 35
GC/HRMS	EPA 1668A/1668C	PCB 36
GC/HRMS	EPA 1668A/1668C	PCB 37
GC/HRMS	EPA 1668A/1668C	PCB 38
GC/HRMS	EPA 1668A/1668C	PCB 39
GC/HRMS	EPA 1668A/1668C	PCB 40
GC/HRMS	EPA 1668A/1668C	PCB 41
GC/HRMS	EPA 1668A/1668C	PCB 42
GC/HRMS	EPA 1668A/1668C	PCB 43
GC/HRMS	EPA 1668A/1668C	PCB 44
GC/HRMS	EPA 1668A/1668C	PCB 45
GC/HRMS	EPA 1668A/1668C	PCB 46
GC/HRMS	EPA 1668A/1668C	PCB 47
GC/HRMS	EPA 1668A/1668C	PCB 48
GC/HRMS	EPA 1668A/1668C	PCB 49
GC/HRMS	EPA 1668A/1668C	PCB 50
GC/HRMS	EPA 1668A/1668C	PCB 51

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 52
GC/HRMS	EPA 1668A/1668C	PCB 53
GC/HRMS	EPA 1668A/1668C	PCB 54
GC/HRMS	EPA 1668A/1668C	PCB 55
GC/HRMS	EPA 1668A/1668C	PCB 56
GC/HRMS	EPA 1668A/1668C	PCB 57
GC/HRMS	EPA 1668A/1668C	PCB 58
GC/HRMS	EPA 1668A/1668C	PCB 59
GC/HRMS	EPA 1668A/1668C	PCB 60
GC/HRMS	EPA 1668A/1668C	PCB 61
GC/HRMS	EPA 1668A/1668C	PCB 62
GC/HRMS	EPA 1668A/1668C	PCB 63
GC/HRMS	EPA 1668A/1668C	PCB 64
GC/HRMS	EPA 1668A/1668C	PCB 65
GC/HRMS	EPA 1668A/1668C	PCB 66
GC/HRMS	EPA 1668A/1668C	PCB 67
GC/HRMS	EPA 1668A/1668C	PCB 68
GC/HRMS	EPA 1668A/1668C	PCB 69
GC/HRMS	EPA 1668A/1668C	PCB 70
GC/HRMS	EPA 1668A/1668C	PCB 71
GC/HRMS	EPA 1668A/1668C	PCB 72
GC/HRMS	EPA 1668A/1668C	PCB 73
GC/HRMS	EPA 1668A/1668C	PCB 74
GC/HRMS	EPA 1668A/1668C	PCB 75
GC/HRMS	EPA 1668A/1668C	PCB 76
GC/HRMS	EPA 1668A/1668C	PCB 77
GC/HRMS	EPA 1668A/1668C	PCB 78
GC/HRMS	EPA 1668A/1668C	PCB 79
GC/HRMS	EPA 1668A/1668C	PCB 80
GC/HRMS	EPA 1668A/1668C	PCB 81
GC/HRMS	EPA 1668A/1668C	PCB 82
GC/HRMS	EPA 1668A/1668C	PCB 83
GC/HRMS	EPA 1668A/1668C	PCB 84
GC/HRMS	EPA 1668A/1668C	PCB 85
GC/HRMS	EPA 1668A/1668C	PCB 86
GC/HRMS	EPA 1668A/1668C	PCB 87
GC/HRMS	EPA 1668A/1668C	PCB 88
GC/HRMS	EPA 1668A/1668C	PCB 89
GC/HRMS	EPA 1668A/1668C	PCB 90

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 91
GC/HRMS	EPA 1668A/1668C	PCB 92
GC/HRMS	EPA 1668A/1668C	PCB 93
GC/HRMS	EPA 1668A/1668C	PCB 94
GC/HRMS	EPA 1668A/1668C	PCB 95
GC/HRMS	EPA 1668A/1668C	PCB 96
GC/HRMS	EPA 1668A/1668C	PCB 97
GC/HRMS	EPA 1668A/1668C	PCB 98
GC/HRMS	EPA 1668A/1668C	PCB 99
GC/HRMS	EPA 1668A/1668C	PCB 100
GC/HRMS	EPA 1668A/1668C	PCB 101
GC/HRMS	EPA 1668A/1668C	PCB 102
GC/HRMS	EPA 1668A/1668C	PCB 103
GC/HRMS	EPA 1668A/1668C	PCB 104
GC/HRMS	EPA 1668A/1668C	PCB 105
GC/HRMS	EPA 1668A/1668C	PCB 106
GC/HRMS	EPA 1668A/1668C	PCB 107
GC/HRMS	EPA 1668A/1668C	PCB 108
GC/HRMS	EPA 1668A/1668C	PCB 109
GC/HRMS	EPA 1668A/1668C	PCB 110
GC/HRMS	EPA 1668A/1668C	PCB 111
GC/HRMS	EPA 1668A/1668C	PCB 112
GC/HRMS	EPA 1668A/1668C	PCB 113
GC/HRMS	EPA 1668A/1668C	PCB 114
GC/HRMS	EPA 1668A/1668C	PCB 115
GC/HRMS	EPA 1668A/1668C	PCB 116
GC/HRMS	EPA 1668A/1668C	PCB 117
GC/HRMS	EPA 1668A/1668C	PCB 118
GC/HRMS	EPA 1668A/1668C	PCB 119
GC/HRMS	EPA 1668A/1668C	PCB 120
GC/HRMS	EPA 1668A/1668C	PCB 121
GC/HRMS	EPA 1668A/1668C	PCB 122
GC/HRMS	EPA 1668A/1668C	PCB 123
GC/HRMS	EPA 1668A/1668C	PCB 124
GC/HRMS	EPA 1668A/1668C	PCB 125
GC/HRMS	EPA 1668A/1668C	PCB 126
GC/HRMS	EPA 1668A/1668C	PCB 127
GC/HRMS	EPA 1668A/1668C	PCB 128
GC/HRMS	EPA 1668A/1668C	PCB 129

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 130
GC/HRMS	EPA 1668A/1668C	PCB 131
GC/HRMS	EPA 1668A/1668C	PCB 132
GC/HRMS	EPA 1668A/1668C	PCB 133
GC/HRMS	EPA 1668A/1668C	PCB 134
GC/HRMS	EPA 1668A/1668C	PCB 135
GC/HRMS	EPA 1668A/1668C	PCB 136
GC/HRMS	EPA 1668A/1668C	PCB 137
GC/HRMS	EPA 1668A/1668C	PCB 138
GC/HRMS	EPA 1668A/1668C	PCB 139
GC/HRMS	EPA 1668A/1668C	PCB 140
GC/HRMS	EPA 1668A/1668C	PCB 141
GC/HRMS	EPA 1668A/1668C	PCB 142
GC/HRMS	EPA 1668A/1668C	PCB 143
GC/HRMS	EPA 1668A/1668C	PCB 144
GC/HRMS	EPA 1668A/1668C	PCB 145
GC/HRMS	EPA 1668A/1668C	PCB 146
GC/HRMS	EPA 1668A/1668C	PCB 147
GC/HRMS	EPA 1668A/1668C	PCB 148
GC/HRMS	EPA 1668A/1668C	PCB 149
GC/HRMS	EPA 1668A/1668C	PCB 150
GC/HRMS	EPA 1668A/1668C	PCB 151
GC/HRMS	EPA 1668A/1668C	PCB 152
GC/HRMS	EPA 1668A/1668C	PCB 153
GC/HRMS	EPA 1668A/1668C	PCB 154
GC/HRMS	EPA 1668A/1668C	PCB 155
GC/HRMS	EPA 1668A/1668C	PCB 156
GC/HRMS	EPA 1668A/1668C	PCB 157
GC/HRMS	EPA 1668A/1668C	PCB 158
GC/HRMS	EPA 1668A/1668C	PCB 159
GC/HRMS	EPA 1668A/1668C	PCB 160
GC/HRMS	EPA 1668A/1668C	PCB 161
GC/HRMS	EPA 1668A/1668C	PCB 162
GC/HRMS	EPA 1668A/1668C	PCB 163
GC/HRMS	EPA 1668A/1668C	PCB 164
GC/HRMS	EPA 1668A/1668C	PCB 165
GC/HRMS	EPA 1668A/1668C	PCB 166
GC/HRMS	EPA 1668A/1668C	PCB 167
GC/HRMS	EPA 1668A/1668C	PCB 168

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 169
GC/HRMS	EPA 1668A/1668C	PCB 170
GC/HRMS	EPA 1668A/1668C	PCB 171
GC/HRMS	EPA 1668A/1668C	PCB 172
GC/HRMS	EPA 1668A/1668C	PCB 173
GC/HRMS	EPA 1668A/1668C	PCB 174
GC/HRMS	EPA 1668A/1668C	PCB 175
GC/HRMS	EPA 1668A/1668C	PCB 176
GC/HRMS	EPA 1668A/1668C	PCB 177
GC/HRMS	EPA 1668A/1668C	PCB 178
GC/HRMS	EPA 1668A/1668C	PCB 179
GC/HRMS	EPA 1668A/1668C	PCB 180
GC/HRMS	EPA 1668A/1668C	PCB 181
GC/HRMS	EPA 1668A/1668C	PCB 182
GC/HRMS	EPA 1668A/1668C	PCB 183
GC/HRMS	EPA 1668A/1668C	PCB 184
GC/HRMS	EPA 1668A/1668C	PCB 185
GC/HRMS	EPA 1668A/1668C	PCB 186
GC/HRMS	EPA 1668A/1668C	PCB 187
GC/HRMS	EPA 1668A/1668C	PCB 188
GC/HRMS	EPA 1668A/1668C	PCB 189
GC/HRMS	EPA 1668A/1668C	PCB 190
GC/HRMS	EPA 1668A/1668C	PCB 191
GC/HRMS	EPA 1668A/1668C	PCB 192
GC/HRMS	EPA 1668A/1668C	PCB 193
GC/HRMS	EPA 1668A/1668C	PCB 194
GC/HRMS	EPA 1668A/1668C	PCB 195
GC/HRMS	EPA 1668A/1668C	PCB 196
GC/HRMS	EPA 1668A/1668C	PCB 197
GC/HRMS	EPA 1668A/1668C	PCB 198
GC/HRMS	EPA 1668A/1668C	PCB 199
GC/HRMS	EPA 1668A/1668C	PCB 200
GC/HRMS	EPA 1668A/1668C	PCB 201
GC/HRMS	EPA 1668A/1668C	PCB 202
GC/HRMS	EPA 1668A/1668C	PCB 203
GC/HRMS	EPA 1668A/1668C	PCB 204
GC/HRMS	EPA 1668A/1668C	PCB 205
GC/HRMS	EPA 1668A/1668C	PCB 206
GC/HRMS	EPA 1668A/1668C	PCB 207

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 208
GC/HRMS	EPA 1668A/1668C	PCB 209
Preparation	**Method**	**Type**
Acid Digestion (Aqueous)	EPA 3005A/3010A	Inorganics
Separatory Funnel Liquid-Liquid Extraction	EPA 3510C	Semivolatile and Non-Volatile Organics
Solid Phase Extraction	EPA 3535A	Semivolatile and Non-Volatile Organics
Purge and Trap	EPA 5030B/5030C	Volatile Organic Compounds
Florisil Cleanup	EPA 3620B/3620C	Cleanup of pesticide residues and other chlorinated hydrocarbons
Sulfur Cleanup	EPA 3660A	Sulfur Cleanup
Sulfuric Acid Cleanup	EPA 3665A	Sulfuric Acid Cleanup for PCBs
Silica Gel Cleanup	EPA 3630C	Column Cleanup

Drinking Water		
Technology	**Method**	**Analyte**
LC/MS/MS	EPA 537	Perfluorobutane Sulfonic Acid (PFBS)
LC/MS/MS	EPA 537	Perfluoroheptanoic acid (PFHpA)
LC/MS/MS	EPA 537	Perfluorohexane Sulfonic Acid (PFHxS)
LC/MS/MS	EPA 537	Perfluorononanoic acid (PFNA)
LC/MS/MS	EPA 537	Perfluorooctanoic acid (PFOA)
LC/MS/MS	EPA 537	Perfluorooctane Sulfonic Acid(PFOS)
Preparation	**Method**	**Type**
Solid Phase Extraction	EPA 537	Perfluoro compounds in Drinking Water

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP-AES	EPA 6010B/6010C	Aluminum
ICP-AES	EPA 6010B/6010C	Antimony
ICP-AES	EPA 6010B/6010C	Arsenic
ICP-AES	EPA 6010B/6010C	Barium
ICP-AES	EPA 6010B/6010C	Beryllium
ICP-AES	EPA 6010B/6010C	Boron
ICP-AES	EPA 6010B/6010C	Cadmium
ICP-AES	EPA 6010B/6010C	Calcium

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP-AES	EPA 6010B/6010C	Chromium (Total)
ICP-AES	EPA 6010B/6010C	Cobalt
ICP-AES	EPA 6010B/6010C	Copper
ICP-AES	EPA 6010B/6010C	Iron
ICP-AES	EPA 6010B/6010C	Lead
ICP-AES	EPA 6010B/6010C	Magnesium
ICP-AES	EPA 6010B/6010C	Manganese
ICP-AES	EPA 6010B/6010C	Molybdenum
ICP-AES	EPA 6010B/6010C	Nickel
ICP-AES	EPA 6010B/6010C	Potassium
ICP-AES	EPA 6010B/6010C	Selenium
ICP-AES	EPA 6010B/6010C	Silver
ICP-AES	EPA 6010B/6010C	Sodium
ICP-AES	EPA 6010B/6010C	Thallium
ICP-AES	EPA 6010B/6010C	Tin
ICP-AES	EPA 6010B/6010C	Titanium
ICP-AES	EPA 6010B/6010C	Vanadium
ICP-AES	EPA 6010B/6010C	Zinc
ICP-MS	EPA 6020/6020A	Aluminum
ICP-MS	EPA 6020/6020A	Antimony
ICP-MS	EPA 6020/6020A	Arsenic
ICP-MS	EPA 6020/6020A	Barium
ICP-MS	EPA 6020/6020A	Beryllium
ICP-MS	EPA 6020/6020A	Cadmium
ICP-MS	EPA 6020/6020A	Calcium
ICP-MS	EPA 6020/6020A	Chromium (Total)
ICP-MS	EPA 6020/6020A	Cobalt
ICP-MS	EPA 6020/6020A	Copper
ICP-MS	EPA 6020/6020A	Iron
ICP-MS	EPA 6020/6020A	Lead
ICP-MS	EPA 6020/6020A	Magnesium
ICP-MS	EPA 6020/6020A	Manganese
ICP-MS	EPA 6020/6020A	Molybdenum
ICP-MS	EPA 6020/6020A	Nickel
ICP-MS	EPA 6020/6020A	Phosphorus
ICP-MS	EPA 6020/6020A	Potassium
ICP-MS	EPA 6020/6020A	Selenium
ICP-MS	EPA 6020/6020A	Silver
ICP-MS	EPA 6020/6020A	Sodium

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP-MS	EPA 6020/6020A	Strontium
ICP-MS	EPA 6020/6020A	Thallium
ICP-MS	EPA 6020/6020A	Tin
ICP-MS	EPA 6020/6020A	Titanium
ICP-MS	EPA 6020/6020A	Uranium
ICP-MS	EPA 6020/6020A	Vanadium
ICP-MS	EPA 6020/6020A	Zinc
CVAAS	EPA 7471A/7471B	Mercury
Colorimetric	EPA 353.2	Nitrate
Colorimetric	EPA 353.2	Nitrate-nitrite
Colorimetric	EPA 353.2	Nitrite
Colorimetric/Hydrolysis	EPA 353.2 Modified /WS-WC-0050	Nitrocellulose
LC/MS/MS	EPA 6850	Perchlorate
Probe	EPA 9045C/9045D	pH
Ion Chromatography	EPA 9056A/300.0	Bromide
Ion Chromatography	EPA 9056A/300.0	Chloride
Ion Chromatography	EPA 9056A/300.0	Fluoride
Ion Chromatography	EPA 9056A/300.0	Sulfate
Ion Chromatography	EPA 9056A/300.0	Nitrate
Ion Chromatography	EPA 9056A/300.0	Nitrite
Gravimetric	ASTM D2216	%Moisture
GC/MS	EPA 8260B/8260C	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B/8260C	1,1,1-Trichloroethane
GC/MS	EPA 8260B/8260C	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B/8260C	1,1,2-Trichloroethane
GC/MS	EPA 8260B/8260C	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA 8260B/8260C	1,1-Dichloroethane
GC/MS	EPA 8260B/8260C	1,1-Dichloroethene
GC/MS	EPA 8260B/8260C	1,1-Dichloropropene
GC/MS	EPA 8260B/8260C	1,2,3-Trichlorobenzene
GC/MS	EPA 8260B/8260C	1,2,3-Trichloropropane
GC/MS	EPA 8260B/8260C	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B/8260C	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B/8260C	1,2-Dibromo-3-chloropropane
GC/MS	EPA 8260B/8260C	1,2-Dibromoethane
GC/MS	EPA 8260B/8260C	1,2-Dichlorobenzene
GC/MS	EPA 8260B/8260C	1,2-Dichloroethane
GC/MS	EPA 8260B/8260C	1,2-Dichloropropane
GC/MS	EPA 8260B/8260C	1,3,5-Trimethylbenzene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/8260C	1,3-Dichlorobenzene
GC/MS	EPA 8260B/8260C	1,3-Dichloropropane
GC/MS	EPA 8260B/8260C	1,4-Dichlorobenzene
GC/MS	EPA 8260B/8260C	1-Chlorohexane
GC/MS	EPA 8260B/8260C	2,2-Dichloropropane
GC/MS	EPA 8260B/8260C	2-Butanone (MEK)
GC/MS	EPA 8260B/8260C	2-Chlorotoluene
GC/MS	EPA 8260B/8260C	2-Hexanone (MBK)
GC/MS	EPA 8260B/8260C	2-Methyl-2-propanol (tert- Butyl Alcohol, TBA)
GC/MS	EPA 8260B/8260C	4-Chlorotoluene
GC/MS	EPA 8260B/8260C	4-Isopropyltoluene
GC/MS	EPA 8260B/8260C	4-Methyl-2-pentanone (MIBK)
GC/MS	EPA 8260B/8260C	Acetone
GC/MS	EPA 8260B/8260C	Allyl Chloride
GC/MS	EPA 8260B/8260C	Benzene
GC/MS	EPA 8260B/8260C	Bromobenzene
GC/MS	EPA 8260B/8260C	Bromochloromethane
GC/MS	EPA 8260B/8260C	Bromodichloromethane
GC/MS	EPA 8260B/8260C	Bromoform
GC/MS	EPA 8260B/8260C	Bromomethane
GC/MS	EPA 8260B/8260C	Carbon Disulfide
GC/MS	EPA 8260B/8260C	Carbon Tetrachloride
GC/MS	EPA 8260B/8260C	Chlorobenzene
GC/MS	EPA 8260B/8260C	Chloroethane
GC/MS	EPA 8260B/8260C	Chloroform
GC/MS	EPA 8260B/8260C	Chloromethane
GC/MS	EPA 8260B/8260C	cis-1,2-Dichloroethene
GC/MS	EPA 8260B/8260C	cis-1,3-Dichloropropene
GC/MS	EPA 8260B/8260C	Cyclohexane
GC/MS	EPA 8260B/8260C	Dibromochloromethane
GC/MS	EPA 8260B/8260C	Dibromomethane
GC/MS	EPA 8260B/8260C	Dichlorodifluoromethane
GC/MS	EPA 8260B/8260C	Diisopropyl Ether (DIPE)
GC/MS	EPA 8260B/8260C	Ethylbenzene
GC/MS	EPA 8260B/8260C	Ethylmethacrylate
GC/MS	EPA 8260B/8260C	Ethyl tert-butyl Ether (ETBE)
GC/MS	EPA 8260B/8260C	Hexachlorobutadiene
GC/MS	EPA 8260B/8260C	Hexane
GC/MS	EPA 8260B/8260C	Iodomethane

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/8260C	Isobutanol (2-Methyl-1-propanol)
GC/MS	EPA 8260B/8260C	Isopropylbenzene
GC/MS	EPA 8260B/8260C	m & p Xylene
GC/MS	EPA 8260B/8260C	Methyl tert-butyl Ether (MTBE)
GC/MS	EPA 8260B/8260C	Methylene Chloride
GC/MS	EPA 8260B/8260C	Naphthalene
GC/MS	EPA 8260B/8260C	n-Butylbenzene
GC/MS	EPA 8260B/8260C	n-Propylbenzene
GC/MS	EPA 8260B/8260C	o-Xylene
GC/MS	EPA 8260B/8260C	sec-Butylbenzene
GC/MS	EPA 8260B/8260C	Styrene
GC/MS	EPA 8260B/8260C	t-Amyl methyl Ether (TAME)
GC/MS	EPA 8260B/8260C	t-1,4-Dichloro-2-Butene
GC/MS	EPA 8260B/8260C	tert-Butylbenzene
GC/MS	EPA 8260B/8260C	Tetrachloroethene
GC/MS	EPA 8260B/8260C	Toluene
GC/MS	EPA 8260B/8260C	trans-1,2-Dichloroethene
GC/MS	EPA 8260B/8260C	trans-1,3-Dichloropropene
GC/MS	EPA 8260B/8260C	Trichloroethene
GC/MS	EPA 8260B/8260C	Trichlorofluoromethane
GC/MS	EPA 8260B/8260C	Vinyl Acetate
GC/MS	EPA 8260B/8260C	Vinyl Chloride
GC/MS	EPA 8260B/8260C	Xylenes, Total
GC/MS	EPA 8260B/AK101MS	Gasoline Range Organics (GRO)
GC/MS	EPA 8270C/8270D	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270C/8270D	1,2,4-Trichlorobenzene
GC/MS	EPA 8270C/8270D	1,2-Dichlorobenzene
GC/MS	EPA 8270C/8270D	1,2-Diphenylhydrazine (as Azobenzene)
GC/MS	EPA 8270C/8270D	1,3-Dichlorobenzene
GC/MS	EPA 8270C/8270D	1,3-Dinitrobenzene
GC/MS	EPA 8270C/8270D	1,4-Dichlorobenzene
GC/MS	EPA 8270C/8270D	1-Methylnaphthalene
GC/MS	EPA 8270C/8270D	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270C/8270D	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/8270D	2,4,6-Trichlorophenol
GC/MS	EPA 8270C/8270D	2,4-Dichlorophenol
GC/MS	EPA 8270C/8270D	2,4-Dimethylphenol
GC/MS	EPA 8270C/8270D	2,4-Dinitrophenol
GC/MS	EPA 8270C/8270D	2,4-Dinitrotoluene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/8270D	2,6-Dichlorophenol
GC/MS	EPA 8270C/8270D	2,6-Dinitrotoluene
GC/MS	EPA 8270C/8270D	2-Chloronaphthalene
GC/MS	EPA 8270C/8270D	2-Chlorophenol
GC/MS	EPA 8270C/8270D	2-Methylnaphthalene
GC/MS	EPA 8270C/8270D	2-Methylphenol
GC/MS	EPA 8270C/8270D	2-Nitroaniline
GC/MS	EPA 8270C/8270D	2-Nitrophenol
GC/MS	EPA 8270C/8270D	3&4-Methylphenol
GC/MS	EPA 8270C/8270D	3,3'-Dichlorobenzidine
GC/MS	EPA 8270C/8270D	3-Nitroaniline
GC/MS	EPA 8270C/8270D	4,6-Dinitro-2-methylphenol
GC/MS	EPA 8270C/8270D	4-Bromophenyl phenyl ether
GC/MS	EPA 8270C/8270D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/8270D	4-Chloroaniline
GC/MS	EPA 8270C/8270D	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270C/8270D	4-Nitroaniline
GC/MS	EPA 8270C/8270D	4-Nitrophenol
GC/MS	EPA 8270C/8270D	Acenaphthene
GC/MS	EPA 8270C/8270D	Acenaphthylene
GC/MS	EPA 8270C/8270D	Aniline
GC/MS	EPA 8270C/8270D	Anthracene
GC/MS	EPA 8270C/8270D	Benzo(a)anthracene
GC/MS	EPA 8270C/8270D	Benzo(a)pyrene
GC/MS	EPA 8270C/8270D	Benzo(b)fluoranthene
GC/MS	EPA 8270C/8270D	Benzo(g,h,i)perylene
GC/MS	EPA 8270C/8270D	Benzo(k)fluoranthene
GC/MS	EPA 8270C/8270D	Benzoic Acid
GC/MS	EPA 8270C/8270D	Benzyl Alcohol
GC/MS	EPA 8270C/8270D	Benzyl butyl Phthalate
GC/MS	EPA 8270C/8270D	Biphenyl
GC/MS	EPA 8270C/8270D	Bis(2-chloroethoxy) Methane
GC/MS	EPA 8270C/8270D	Bis(2-chloroethyl) Ether
GC/MS	EPA 8270C/8270D	Bis(2-chloroisopropyl) Ether
GC/MS	EPA 8270C/8270D	Carbazole
GC/MS	EPA 8270C/8270D	Chrysene
GC/MS	EPA 8270C/8270D	Bis (2-ethylhexyl) Phthalate
GC/MS	EPA 8270C/8270D	Dibenz(a,h)anthracene
GC/MS	EPA 8270C/8270D	Dibenzofuran

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/8270D	Diethyl Phthalate
GC/MS	EPA 8270C/8270D	Dimethyl Phthalate
GC/MS	EPA 8270C/8270D	Di-n-butyl Phthalate
GC/MS	EPA 8270C/8270D	Di-n-octyl Phthalate
GC/MS	EPA 8270C/8270D	Fluoranthene
GC/MS	EPA 8270C/8270D	Fluorene
GC/MS	EPA 8270C/8270D	Hexachlorobenzene
GC/MS	EPA 8270C/8270D	Hexachlorobutadiene
GC/MS	EPA 8270C/8270D	Hexachlorocyclopentadiene
GC/MS	EPA 8270C/8270D	Hexachloroethane
GC/MS	EPA 8270C/8270D	Indeno(1,2,3-c,d) Pyrene
GC/MS	EPA 8270C/8270D	Isophorone
GC/MS	EPA 8270C/8270D	Naphthalene
GC/MS	EPA 8270C/8270D	Nitrobenzene
GC/MS	EPA 8270C/8270D	n-Nitrosodimethylamine
GC/MS	EPA 8270C/8270D	n-Nitrosodi-n-propylamine
GC/MS	EPA 8270C/8270D	n-Nitrosodiphenylamine
GC/MS	EPA 8270C/8270D	Pentachlorophenol
GC/MS	EPA 8270C/8270D	Phenanthrene
GC/MS	EPA 8270C/8270D	Phenol
GC/MS	EPA 8270C/8270D	Pyrene
GC/MS	EPA 8270C/8270D	Pyridine
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	1-Methylnaphthalene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	2-Methylnaphthalene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Acenaphthene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Acenaphthylene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Anthracene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(a)anthracene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(a)pyrene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(b)fluoranthene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(g,h,i)perylene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(k)fluoranthene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Chrysene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Dibenz(a,h)anthracene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Fluoranthene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Fluorene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Indeno(1,2,3-c,d) Pyrene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Naphthalene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Phenanthrene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Pyrene
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	1,4-Dithiane
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	Benzothiazole
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	p-Chlorophenyl methylsulfide
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	p-Chlorophenyl methylsulfoxide
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	p-Chlorophenyl methylsulfone
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	Chloropicrin
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	Acetophenone
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	2-Chloroacetophenone
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	1,4-Oxathiane
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	Dimethyl Disulfide
GC/MS SIM	EPA 521 Modified / WS-MS-0012	N-Nitrosodimethyl amine (NDMA)
GC-FID	EPA 8015B/8015C/8015D AK102	Diesel Range Organics (DRO)
GC-FID	AK103	Residual Range Organics
GC-FID	EPA 8015B/8015C/8015D	Motor Oil Range Organics (MRO)
GC-ECD	EPA 8081A/8081B	Aldrin
GC-ECD	EPA 8081A/8081B	a-BHC
GC-ECD	EPA 8081A/8081B	b-BHC

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC-ECD	EPA 8081A/8081B	d-BHC
GC-ECD	EPA 8081A/8081B	g-BHC (Lindane)
GC-ECD	EPA 8081A/8081B	a-Chlordane
GC-ECD	EPA 8081A/8081B	g-Chlordane
GC-ECD	EPA 8081A/8081B	4,4'-DDD
GC-ECD	EPA 8081A/8081B	4,4'-DDE
GC-ECD	EPA 8081A/8081B	4,4'-DDT
GC-ECD	EPA 8081A/8081B	Dieldrin
GC-ECD	EPA 8081A/8081B	Endosulfan I
GC-ECD	EPA 8081A/8081B	Endosulfan II
GC-ECD	EPA 8081A/8081B	Endosulfan sulfate
GC-ECD	EPA 8081A/8081B	Endrin
GC-ECD	EPA 8081A/8081B	Endrin Aldehyde
GC-ECD	EPA 8081A/8081B	Endrin Ketone
GC-ECD	EPA 8081A/8081B	Heptachlor
GC-ECD	EPA 8081A/8081B	Heptachlor Epoxide
GC-ECD	EPA 8081A/8081B	Methoxychlor
GC-ECD	EPA 8081A/8081B	Toxaphene
GC-ECD	EPA 8081A/8081B	Chlordane (technical)
GC-ECD	EPA 8082/8082A	PCB-1016
GC-ECD	EPA 8082/8082A	PCB-1221
GC-ECD	EPA 8082/8082A	PCB-1232
GC-ECD	EPA 8082/8082A	PCB-1242
GC-ECD	EPA 8082/8082A	PCB-1248
GC-ECD	EPA 8082/8082A	PCB-1254
GC-ECD	EPA 8082/8082A	PCB-1260
GC-ECD	EPA 8082/8082A	PCB-1262
GC-ECD	EPA 8082/8082A	PCB-1268
GC/MS	EPA 8280A/8280B	2,3,7,8-TeCDD
GC/MS	EPA 8280A/8280B	1,2,3,7,8-PeCDD
GC/MS	EPA 8280A/8280B	1,2,3,4,7,8-HxCDD
GC/MS	EPA 8280A/8280B	1,2,3,6,7,8-HxCDD
GC/MS	EPA 8280A/8280B	1,2,3,7,8,9-HxCDD
GC/MS	EPA 8280A/8280B	1,2,3,4,6,7,8-HpCDD
GC/MS	EPA 8280A/8280B	OCDD
GC/MS	EPA 8280A/8280B	2,3,7,8-TeCDF
GC/MS	EPA 8280A/8280B	1,2,3,7,8-PeCDF
GC/MS	EPA 8280A/8280B	2,3,4,7,8-PeCDF
GC/MS	EPA 8280A/8280B	1,2,3,4,7,8-HxCDF

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8280A/8280B	1,2,3,6,7,8-HxCDF
GC/MS	EPA 8280A/8280B	1,2,3,7,8,9-HxCDF
GC/MS	EPA 8280A/8280B	2,3,4,6,7,8-HxCDF
GC/MS	EPA 8280A/8280B	1,2,3,4,6,7,8-HpCDF
GC/MS	EPA 8280A/8280B	1,2,3,4,7,8,9-HpCDF
GC/MS	EPA 8280A/8280B	OCDF
GC/MS	EPA 8280A/8280B	Total TCDD
GC/MS	EPA 8280A/8280B	Total PeCDD
GC/MS	EPA 8280A/8280B	Total HxCDD
GC/MS	EPA 8280A/8280B	Total HeptaCDD
GC/MS	EPA 8280A/8280B	Total TCDF
GC/MS	EPA 8280A/8280B	Total PeCDF
GC/MS	EPA 8280A/8280B	Total HxCDF
GC/MS	EPA 8280A/8280B	Total HpCDF
GC/HRMS	EPA 8290/ 8290A/1613B	2,3,7,8-TeCDD
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,7,8-PeCDD
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,4,7,8-HxCDD
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,6,7,8-HxCDD
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,7,8,9-HxCDD
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,4,6,7,8-HpCDD
GC/HRMS	EPA 8290/ 8290A/1613B	OCDD
GC/HRMS	EPA 8290/ 8290A/1613B	2,3,7,8-TeCDF
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,7,8-PeCDF
GC/HRMS	EPA 8290/ 8290A/1613B	2,3,4,7,8-PeCDF
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,4,7,8-HxCDF
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,6,7,8-HxCDF
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,7,8,9-HxCDF
GC/HRMS	EPA 8290/ 8290A/1613B	2,3,4,6,7,8-HxCDF
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,4,6,7,8-HpCDF
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,4,7,8,9-HpCDF
GC/HRMS	EPA 8290/ 8290A/1613B	OCDF
GC/HRMS	EPA 8290/ 8290A/1613B	Total TCDD
GC/HRMS	EPA 8290/ 8290A/1613B	Total PeCDD
GC/HRMS	EPA 8290/ 8290A/1613B	Total HxCDD
GC/HRMS	EPA 8290/ 8290A/1613B	Total HpCDD
GC/HRMS	EPA 8290/ 8290A/1613B	Total TCDF
GC/HRMS	EPA 8290/ 8290A/1613B	Total PeCDF
GC/HRMS	EPA 8290/ 8290A/1613B	Total HxCDF
GC/HRMS	EPA 8290/ 8290A/1613B	Total HpCDF

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
HPLC/UV	EPA 8330A/8330B	2-Amino-4,6-dinitrotoluene
HPLC/UV	EPA 8330A/8330B	4-Amino-2,6-dinitrotoluene
HPLC/UV	EPA 8330A/8330B	3,5-Dinitroaniline
HPLC/UV	EPA 8330A/8330B	1,3-Dinitrobenzene
HPLC/UV	EPA 8330A/8330B	2,4-Dinitrotoluene
HPLC/UV	EPA 8330A/8330B	2,6-Dinitrotoluene
HPLC/UV	EPA 8330A/8330B	Glycerol trinitrate (Nitroglycerin)
HPLC/UV	EPA 8330A/8330B	Hexahydro-1,3,5-trinitro- 1,3,5-triazine (Hexogen)
HPLC/UV	EPA 8330A/8330B	Methyl-2,4,6- trinitrophenylnitramine
HPLC/UV	EPA 8330A/8330B	Nitrobenzene
HPLC/UV	EPA 8330A/8330B	2-Nitrotoluene (o-Nitrotoluene)
HPLC/UV	EPA 8330A/8330B	3-Nitrotoluene (m-Nitrotoluene)
HPLC/UV	EPA 8330A/8330B	4-Nitrotoluene (p-Nitrotoluene)
HPLC/UV	EPA 8330A/8330B	Octahydro-1,3,5,7- tetranitro1,3,5,7-tetracine (Octogen)
HPLC/UV	EPA 8330A/8330B	Picric acid
HPLC/UV	EPA 8330A/8330B	Pentaerythritol Tetranitrate
HPLC/UV	EPA 8330A/8330B	1,3,5-Trinitrobenzene
HPLC/UV	EPA 8330A/8330B	2,4,6-Trinitrotoluene
HPLC/UV	EPA 8330A/8330B	Hexahydro-1,3-dinitroso-5- nitro-1,3,5,triazine (DNX)
HPLC/UV	EPA 8330A/8330B	Hexahydro-1,3,5-trinitroso- 1,3,5-triazine (TNX)
HPLC/UV	EPA 8330A/8330B	1-Nitroso-3,5-dinitro-1,3,5- triazacyclohexane (MNX)
HPLC/UV	EPA 8330A Modified / WS-LC-0010	Nitroguanidine
GC-HRMS	EPA 8290 Modified / WS-ID-0021	2-(N-ethylperfluoro-1- octanesulfonamido)-ethanol [N-Et-FOSE]
GC-HRMS	EPA 8290 Modified / WS-ID-0021	2-(N-Methylperfluoro-1- octanesulfonamido)-ethanol [N-Me-FOSE]
LC/MS/MS	EPA 537 Modified / WS-LC-0025	6:2 Fluorotelomer sulfonate (6:2 FTS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	8:2 Fluorotelomer sulfonate (8:2 FTS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	N-Ethyl perfluorooctane sulfonamide (EtFOSA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	N-Ethyl perfluorooctanesulfon amidacetic acid (EtFOSAA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	N-Methyl perfluorooctane sulfonamide (MeFOSA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	N-Methyl perfluorooctanesulfon amidoacetic acide (MeFOSAA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorooctanoic acid (PFOA)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorooctane Sulfonic Acid (PFOS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorobutyric acid (PFBA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluoropentanoic acid (PFPA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorohexanoic acid (PFHxA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluoroheptanoic acid (PFHpA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorononanoic acid (PFNA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorodecanoic acid (PFDA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluoroundecanoic acid (PFUDA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorododecanoic acid (PFDoDA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorotridecanoic acid (PFTriA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorotetradecanoic acid (PDTeA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorobutane Sulfonic Acid (PFBS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorohexane Sulfonic Acid (PFHxS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluoroheptane Sulfonic Acid (PFHpS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorodecane Sulfonic Acid (PFDS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluoroocatane Sulfonamide (FOSA)
GC/HRMS	EPA 1668A/1668C	PCB 1
GC/HRMS	EPA 1668A/1668C	PCB 2
GC/HRMS	EPA 1668A/1668C	PCB 3
GC/HRMS	EPA 1668A/1668C	PCB 4
GC/HRMS	EPA 1668A/1668C	PCB 5
GC/HRMS	EPA 1668A/1668C	PCB 6
GC/HRMS	EPA 1668A/1668C	PCB 7
GC/HRMS	EPA 1668A/1668C	PCB 8
GC/HRMS	EPA 1668A/1668C	PCB 9
GC/HRMS	EPA 1668A/1668C	PCB 10
GC/HRMS	EPA 1668A/1668C	PCB 11
GC/HRMS	EPA 1668A/1668C	PCB 12

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 13
GC/HRMS	EPA 1668A/1668C	PCB 14
GC/HRMS	EPA 1668A/1668C	PCB 15
GC/HRMS	EPA 1668A/1668C	PCB 16
GC/HRMS	EPA 1668A/1668C	PCB 17
GC/HRMS	EPA 1668A/1668C	PCB 18
GC/HRMS	EPA 1668A/1668C	PCB 19
GC/HRMS	EPA 1668A/1668C	PCB 20
GC/HRMS	EPA 1668A/1668C	PCB 21
GC/HRMS	EPA 1668A/1668C	PCB 22
GC/HRMS	EPA 1668A/1668C	PCB 23
GC/HRMS	EPA 1668A/1668C	PCB 24
GC/HRMS	EPA 1668A/1668C	PCB 25
GC/HRMS	EPA 1668A/1668C	PCB 26
GC/HRMS	EPA 1668A/1668C	PCB 27
GC/HRMS	EPA 1668A/1668C	PCB 28
GC/HRMS	EPA 1668A/1668C	PCB 29
GC/HRMS	EPA 1668A/1668C	PCB 30
GC/HRMS	EPA 1668A/1668C	PCB 32
GC/HRMS	EPA 1668A/1668C	PCB 31
GC/HRMS	EPA 1668A/1668C	PCB 33
GC/HRMS	EPA 1668A/1668C	PCB 34
GC/HRMS	EPA 1668A/1668C	PCB 35
GC/HRMS	EPA 1668A/1668C	PCB 36
GC/HRMS	EPA 1668A/1668C	PCB 37
GC/HRMS	EPA 1668A/1668C	PCB 38
GC/HRMS	EPA 1668A/1668C	PCB 39
GC/HRMS	EPA 1668A/1668C	PCB 40
GC/HRMS	EPA 1668A/1668C	PCB 41
GC/HRMS	EPA 1668A/1668C	PCB 42
GC/HRMS	EPA 1668A/1668C	PCB 43
GC/HRMS	EPA 1668A/1668C	PCB 44
GC/HRMS	EPA 1668A/1668C	PCB 45
GC/HRMS	EPA 1668A/1668C	PCB 46
GC/HRMS	EPA 1668A/1668C	PCB 47
GC/HRMS	EPA 1668A/1668C	PCB 48
GC/HRMS	EPA 1668A/1668C	PCB 49
GC/HRMS	EPA 1668A/1668C	PCB 50
GC/HRMS	EPA 1668A/1668C	PCB 51

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 52
GC/HRMS	EPA 1668A/1668C	PCB 53
GC/HRMS	EPA 1668A/1668C	PCB 54
GC/HRMS	EPA 1668A/1668C	PCB 55
GC/HRMS	EPA 1668A/1668C	PCB 56
GC/HRMS	EPA 1668A/1668C	PCB 57
GC/HRMS	EPA 1668A/1668C	PCB 58
GC/HRMS	EPA 1668A/1668C	PCB 59
GC/HRMS	EPA 1668A/1668C	PCB 60
GC/HRMS	EPA 1668A/1668C	PCB 61
GC/HRMS	EPA 1668A/1668C	PCB 62
GC/HRMS	EPA 1668A/1668C	PCB 63
GC/HRMS	EPA 1668A/1668C	PCB 64
GC/HRMS	EPA 1668A/1668C	PCB 65
GC/HRMS	EPA 1668A/1668C	PCB 66
GC/HRMS	EPA 1668A/1668C	PCB 67
GC/HRMS	EPA 1668A/1668C	PCB 68
GC/HRMS	EPA 1668A/1668C	PCB 69
GC/HRMS	EPA 1668A/1668C	PCB 70
GC/HRMS	EPA 1668A/1668C	PCB 71
GC/HRMS	EPA 1668A/1668C	PCB 72
GC/HRMS	EPA 1668A/1668C	PCB 73
GC/HRMS	EPA 1668A/1668C	PCB 74
GC/HRMS	EPA 1668A/1668C	PCB 75
GC/HRMS	EPA 1668A/1668C	PCB 76
GC/HRMS	EPA 1668A/1668C	PCB 77
GC/HRMS	EPA 1668A/1668C	PCB 78
GC/HRMS	EPA 1668A/1668C	PCB 79
GC/HRMS	EPA 1668A/1668C	PCB 80
GC/HRMS	EPA 1668A/1668C	PCB 81
GC/HRMS	EPA 1668A/1668C	PCB 82
GC/HRMS	EPA 1668A/1668C	PCB 83
GC/HRMS	EPA 1668A/1668C	PCB 84
GC/HRMS	EPA 1668A/1668C	PCB 85
GC/HRMS	EPA 1668A/1668C	PCB 86
GC/HRMS	EPA 1668A/1668C	PCB 87
GC/HRMS	EPA 1668A/1668C	PCB 88
GC/HRMS	EPA 1668A/1668C	PCB 89
GC/HRMS	EPA 1668A/1668C	PCB 90

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 91
GC/HRMS	EPA 1668A/1668C	PCB 92
GC/HRMS	EPA 1668A/1668C	PCB 93
GC/HRMS	EPA 1668A/1668C	PCB 94
GC/HRMS	EPA 1668A/1668C	PCB 95
GC/HRMS	EPA 1668A/1668C	PCB 96
GC/HRMS	EPA 1668A/1668C	PCB 97
GC/HRMS	EPA 1668A/1668C	PCB 98
GC/HRMS	EPA 1668A/1668C	PCB 99
GC/HRMS	EPA 1668A/1668C	PCB 100
GC/HRMS	EPA 1668A/1668C	PCB 101
GC/HRMS	EPA 1668A/1668C	PCB 102
GC/HRMS	EPA 1668A/1668C	PCB 103
GC/HRMS	EPA 1668A/1668C	PCB 104
GC/HRMS	EPA 1668A/1668C	PCB 105
GC/HRMS	EPA 1668A/1668C	PCB 106
GC/HRMS	EPA 1668A/1668C	PCB 107
GC/HRMS	EPA 1668A/1668C	PCB 108
GC/HRMS	EPA 1668A/1668C	PCB 109
GC/HRMS	EPA 1668A/1668C	PCB 110
GC/HRMS	EPA 1668A/1668C	PCB 111
GC/HRMS	EPA 1668A/1668C	PCB 112
GC/HRMS	EPA 1668A/1668C	PCB 113
GC/HRMS	EPA 1668A/1668C	PCB 114
GC/HRMS	EPA 1668A/1668C	PCB 115
GC/HRMS	EPA 1668A/1668C	PCB 116
GC/HRMS	EPA 1668A/1668C	PCB 117
GC/HRMS	EPA 1668A/1668C	PCB 118
GC/HRMS	EPA 1668A/1668C	PCB 119
GC/HRMS	EPA 1668A/1668C	PCB 120
GC/HRMS	EPA 1668A/1668C	PCB 121
GC/HRMS	EPA 1668A/1668C	PCB 122
GC/HRMS	EPA 1668A/1668C	PCB 123
GC/HRMS	EPA 1668A/1668C	PCB 124
GC/HRMS	EPA 1668A/1668C	PCB 125
GC/HRMS	EPA 1668A/1668C	PCB 126
GC/HRMS	EPA 1668A/1668C	PCB 127
GC/HRMS	EPA 1668A/1668C	PCB 128
GC/HRMS	EPA 1668A/1668C	PCB 129

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 130
GC/HRMS	EPA 1668A/1668C	PCB 131
GC/HRMS	EPA 1668A/1668C	PCB 132
GC/HRMS	EPA 1668A/1668C	PCB 133
GC/HRMS	EPA 1668A/1668C	PCB 134
GC/HRMS	EPA 1668A/1668C	PCB 135
GC/HRMS	EPA 1668A/1668C	PCB 136
GC/HRMS	EPA 1668A/1668C	PCB 137
GC/HRMS	EPA 1668A/1668C	PCB 138
GC/HRMS	EPA 1668A/1668C	PCB 139
GC/HRMS	EPA 1668A/1668C	PCB 140
GC/HRMS	EPA 1668A/1668C	PCB 141
GC/HRMS	EPA 1668A/1668C	PCB 142
GC/HRMS	EPA 1668A/1668C	PCB 143
GC/HRMS	EPA 1668A/1668C	PCB 144
GC/HRMS	EPA 1668A/1668C	PCB 145
GC/HRMS	EPA 1668A/1668C	PCB 146
GC/HRMS	EPA 1668A/1668C	PCB 147
GC/HRMS	EPA 1668A/1668C	PCB 148
GC/HRMS	EPA 1668A/1668C	PCB 149
GC/HRMS	EPA 1668A/1668C	PCB 150
GC/HRMS	EPA 1668A/1668C	PCB 151
GC/HRMS	EPA 1668A/1668C	PCB 152
GC/HRMS	EPA 1668A/1668C	PCB 153
GC/HRMS	EPA 1668A/1668C	PCB 154
GC/HRMS	EPA 1668A/1668C	PCB 155
GC/HRMS	EPA 1668A/1668C	PCB 156
GC/HRMS	EPA 1668A/1668C	PCB 157
GC/HRMS	EPA 1668A/1668C	PCB 158
GC/HRMS	EPA 1668A/1668C	PCB 159
GC/HRMS	EPA 1668A/1668C	PCB 160
GC/HRMS	EPA 1668A/1668C	PCB 161
GC/HRMS	EPA 1668A/1668C	PCB 162
GC/HRMS	EPA 1668A/1668C	PCB 163
GC/HRMS	EPA 1668A/1668C	PCB 164
GC/HRMS	EPA 1668A/1668C	PCB 165
GC/HRMS	EPA 1668A/1668C	PCB 166
GC/HRMS	EPA 1668A/1668C	PCB 167
GC/HRMS	EPA 1668A/1668C	PCB 168

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 169
GC/HRMS	EPA 1668A/1668C	PCB 170
GC/HRMS	EPA 1668A/1668C	PCB 171
GC/HRMS	EPA 1668A/1668C	PCB 172
GC/HRMS	EPA 1668A/1668C	PCB 173
GC/HRMS	EPA 1668A/1668C	PCB 174
GC/HRMS	EPA 1668A/1668C	PCB 175
GC/HRMS	EPA 1668A/1668C	PCB 176
GC/HRMS	EPA 1668A/1668C	PCB 177
GC/HRMS	EPA 1668A/1668C	PCB 178
GC/HRMS	EPA 1668A/1668C	PCB 179
GC/HRMS	EPA 1668A/1668C	PCB 180
GC/HRMS	EPA 1668A/1668C	PCB 181
GC/HRMS	EPA 1668A/1668C	PCB 182
GC/HRMS	EPA 1668A/1668C	PCB 183
GC/HRMS	EPA 1668A/1668C	PCB 184
GC/HRMS	EPA 1668A/1668C	PCB 185
GC/HRMS	EPA 1668A/1668C	PCB 186
GC/HRMS	EPA 1668A/1668C	PCB 187
GC/HRMS	EPA 1668A/1668C	PCB 188
GC/HRMS	EPA 1668A/1668C	PCB 189
GC/HRMS	EPA 1668A/1668C	PCB 190
GC/HRMS	EPA 1668A/1668C	PCB 191
GC/HRMS	EPA 1668A/1668C	PCB 192
GC/HRMS	EPA 1668A/1668C	PCB 193
GC/HRMS	EPA 1668A/1668C	PCB 194
GC/HRMS	EPA 1668A/1668C	PCB 195
GC/HRMS	EPA 1668A/1668C	PCB 196
GC/HRMS	EPA 1668A/1668C	PCB 197
GC/HRMS	EPA 1668A/1668C	PCB 198
GC/HRMS	EPA 1668A/1668C	PCB 199
GC/HRMS	EPA 1668A/1668C	PCB 200
GC/HRMS	EPA 1668A/1668C	PCB 201
GC/HRMS	EPA 1668A/1668C	PCB 202
GC/HRMS	EPA 1668A/1668C	PCB 203
GC/HRMS	EPA 1668A/1668C	PCB 204
GC/HRMS	EPA 1668A/1668C	PCB 205
GC/HRMS	EPA 1668A/1668C	PCB 206
GC/HRMS	EPA 1668A/1668C	PCB 207

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 208
GC/HRMS	EPA 1668A/1668C	PCB 209
Preparation	**Method**	**Type**
Acid Digestion (Aqueous)	EPA 3005A/3010A	Inorganics
Acid Digestion (Solid)	EPA 3050B	Inorganics
Separatory Funnel Liquid-Liquid Extraction	EPA 3510C	Semivolatile and Non-Volatile Organics
Ultrasonic Extraction	EPA 3550B/3550C	Semivolatile and Non-Volatile Organics
Solvent Dilution	EPA 3580A	Semivolatile and Non-Volatile Organics
Purge and Trap	EPA 5030B	Volatile Organic Compounds
Purge and Trap	EPA 5035/5035A	Volatile Organic Compounds
Table / Jar Shake	WS-OP-0005	Chemical Warfare Degradates (in solid)
Microwave Extraction	EPA 3546	Semivolatile and Non-Volatile Organics
Florisil Cleanup	EPA 3620B/3620C	Cleanup of pesticide residues and other chlorinated hydrocarbons
Sulfur Cleanup	EPA 3660A	Sulfur Cleanup
Sulfuric Acid Cleanup	EPA 3665A	Sulfuric Acid Cleanup for PCBs
Silica Gel Cleanup	EPA 3630C	Column Cleanup
TCLP Extraction	EPA 1311	Toxicity Characteristic Leaching Procedure

Air and Emissions		
Technology	**Method**	**Analyte**
ICP-MS	EPA 6020/6020A	Aluminum
ICP-MS	EPA 6020/6020A	Antimony
ICP-MS	EPA 6020/6020A	Arsenic
ICP-MS	EPA 6020/6020A	Barium
ICP-MS	EPA 6020/6020A	Beryllium
ICP-MS	EPA 6020/6020A	Cadmium
ICP-MS	EPA 6020/6020A	Calcium
ICP-MS	EPA 6020/6020A	Chromium (Total)
ICP-MS	EPA 6020/6020A	Cobalt
ICP-MS	EPA 6020/6020A	Copper
ICP-MS	EPA 6020/6020A	Iron
ICP-MS	EPA 6020/6020A	Lead
ICP-MS	EPA 6020/6020A	Magnesium
ICP-MS	EPA 6020/6020A	Manganese
ICP-MS	EPA 6020/6020A	Molybdenum
ICP-MS	EPA 6020/6020A	Nickel

Air and Emissions		
Technology	**Method**	**Analyte**
ICP-MS	EPA 6020/6020A	Potassium
ICP-MS	EPA 6020/6020A	Selenium
ICP-MS	EPA 6020/6020A	Silver
ICP-MS	EPA 6020/6020A	Sodium
ICP-MS	EPA 6020/6020A	Thallium
ICP-MS	EPA 6020/6020A	Vanadium
ICP-MS	EPA 6020/6020A	Zinc
Gravimetric	40CFR Part 50 App B	TSP (Total Suspended Particulate)
Gravimetric	40CFR Part 50 App J	PM10
GC/MS	EPA TO14A/TO15	1,1,1-Trichloroethane
GC/MS	EPA TO14A/TO15	1,1,2,2-Tetrachloroethane
GC/MS	EPA TO14A/TO15	1,1,2-Trichloroethane
GC/MS	EPA TO14A/TO15	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA TO14A/TO15	1,1-Dichloroethane
GC/MS	EPA TO14A/TO15	1,1-Dichloroethene
GC/MS	EPA TO14A/TO15	1,2,3-Trichlorobenzene
GC/MS	EPA TO14A/TO15	1,2,3-Trichloropropane
GC/MS	EPA TO14A/TO15	1,2,4-Trichlorobenzene
GC/MS	EPA TO14A/TO15	1,2,4-Trimethylbenzene
GC/MS	EPA TO14A/TO15	1,2-Dibromoethane
GC/MS	EPA TO14A/TO15	1,2-Dichlorobenzene
GC/MS	EPA TO14A/TO15	1,2-Dichloroethane
GC/MS	EPA TO14A/TO15	1,2-Dichloropropane
GC/MS	EPA TO14A/TO15	1,3,5-Trimethylbenzene
GC/MS	EPA TO14A/TO15	1,3-Dichlorobenzene
GC/MS	EPA TO14A/TO15	1,4-Dichlorobenzene
GC/MS	EPA TO14A/TO15	1,4-Dioxane
GC/MS	EPA TO14A/TO15	2-Butanone (MEK)
GC/MS	EPA TO14A/TO15	2-Chlorotoluene
GC/MS	EPA TO14A/TO15	2-Hexanone (MBK)
GC/MS	EPA TO14A/TO15	2-Methyl-2-propanol (tert- Butyl Alcohol, TBA)
GC/MS	EPA TO14A/TO15	4-Ethyltoluene
GC/MS	EPA TO14A/TO15	4-Isopropyltoluene
GC/MS	EPA TO14A/TO15	4-Methyl-2-pentanone (MIBK)
GC/MS	EPA TO14A/TO15	Acetone
GC/MS	EPA TO14A/TO15	Acrolein
GC/MS	EPA TO14A/TO15	Allyl Chloride
GC/MS	EPA TO14A/TO15	Alpha Methyl Styrene
GC/MS	EPA TO14A/TO15	Benzene

Air and Emissions		
Technology	**Method**	**Analyte**
GC/MS	EPA TO14A/TO15	Benzyl Chloride
GC/MS	EPA TO14A/TO15	Bromodichloromethane
GC/MS	EPA TO14A/TO15	Bromoform
GC/MS	EPA TO14A/TO15	Bromomethane
GC/MS	EPA TO14A/TO15	Butadiene (1,3-Butadiene)
GC/MS	EPA TO14A/TO15	Butane
GC/MS	EPA TO14A/TO15	Carbon Disulfide
GC/MS	EPA TO14A/TO15	Carbon Tetrachloride
GC/MS	EPA TO14A/TO15	Chlorobenzene
GC/MS	EPA TO14A/TO15	Chlorodifluoromethane
GC/MS	EPA TO14A/TO15	Chloroethane
GC/MS	EPA TO14A/TO15	Chloroform
GC/MS	EPA TO14A/TO15	Chloromethane
GC/MS	EPA TO14A/TO15	cis-1,2-Dichloroethene
GC/MS	EPA TO14A/TO15	cis-1,3-Dichloropropene
GC/MS	EPA TO14A/TO15	Cyclohexane
GC/MS	EPA TO14A/TO15	Dibromochloromethane
GC/MS	EPA TO14A/TO15	Dibromomethane
GC/MS	EPA TO14A/TO15	Dichlorodifluoromethane
GC/MS	EPA TO14A/TO15	Ethyl Acetate
GC/MS	EPA TO14A/TO15	Ethylbenzene
GC/MS	EPA TO14A/TO15	Hexachlorobutadiene
GC/MS	EPA TO14A/TO15	Hexane
GC/MS	EPA TO14A/TO15	Isooctane (2,2,4- Trimethylpentane)
GC/MS	EPA TO14A/TO15	Isopropyl Alcohol
GC/MS	EPA TO14A/TO15	Isopropylbenzene
GC/MS	EPA TO14A/TO15	m & p Xylene
GC/MS	EPA TO14A/TO15	Methyl tert-butyl Ether (MTBE)
GC/MS	EPA TO14A/TO15	Methylene Chloride
GC/MS	EPA TO14A/TO15	Naphthalene
GC/MS	EPA TO14A/TO15	n-Butanol
GC/MS	EPA TO14A/TO15	n-Butylbenzene
GC/MS	EPA TO14A/TO15	n-Heptane
GC/MS	EPA TO14A/TO15	n-Nonane
GC/MS	EPA TO14A/TO15	n-Octane
GC/MS	EPA TO14A/TO15	n-Propylbenzene
GC/MS	EPA TO14A/TO15	o-Xylene
GC/MS	EPA TO14A/TO15	Pentane
GC/MS	EPA TO14A/TO15	Propene

Air and Emissions		
Technology	**Method**	**Analyte**
GC/MS	EPA TO14A/TO15	sec-Butylbenzene
GC/MS	EPA TO14A/TO15	Styrene
GC/MS	EPA TO14A/TO15	tert-Butylbenzene
GC/MS	EPA TO14A/TO15	Tetrachloroethene
GC/MS	EPA TO14A/TO15	Tetrahydrofuran
GC/MS	EPA TO14A/TO15	Toluene
GC/MS	EPA TO14A/TO15	trans-1,2-Dichloroethene
GC/MS	EPA TO14A/TO15	trans-1,3-Dichloropropene
GC/MS	EPA TO14A/TO15	Trichloroethene
GC/MS	EPA TO14A/TO15	Trichlorofluoromethane
GC/MS	EPA TO14A/TO15	Vinyl Acetate
GC/MS	EPA TO14A/TO15	Vinyl Bromide
GC/MS	EPA TO14A/TO15	Vinyl Chloride
GC/MS	EPA TO14A/TO15	Xylenes, Total
GC-FID/TCD	ASTM1946D / EPA 3C	Carbon Dioxide
GC-FID/TCD	ASTM1946D / EPA 3C	Nitrogen
GC-FID/TCD	ASTM1946D / EPA 3C	Oxygen
GC-FID/TCD	ASTM1946D / EPA 3C	Helium
GC-FID/TCD	ASTM1946D / EPA 3C	Hydrogen
GC-FID/TCD	ASTM1946D / EPA 3C	Methane
GC-FID/TCD	ASTM1946D / EPA 3C	Carbon Monoxide[A1]
GC/MS	EPA TO14A/TO15	Gasoline Range Organics (GRO)
GC/MS	EPA TO14A/TO15	TPH as Gasoline
GC/MS SIM	EPA TO15 SIM	1,1,1-Trichloroethane
GC/MS SIM	EPA TO15 SIM	1,1,2,2-Tetrachloroethane
GC/MS SIM	EPA TO15 SIM	1,1,2-Trichloroethane
GC/MS SIM	EPA TO15 SIM	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS SIM	EPA TO15 SIM	1,1-Dichloroethane
GC/MS SIM	EPA TO15 SIM	1,1-Dichloroethene
GC/MS SIM	EPA TO15 SIM	1,2,3-Trichloropropane
GC/MS SIM	EPA TO15 SIM	1,2,4-Trichlorobenzene
GC/MS SIM	EPA TO15 SIM	1,2-Dibromoethane
GC/MS SIM	EPA TO15 SIM	1,2-Dichlorobenzene
GC/MS SIM	EPA TO15 SIM	1,2-Dichloroethane
GC/MS SIM	EPA TO15 SIM	1,2-Dichloropropane
GC/MS SIM	EPA TO15 SIM	1,3-Dichlorobenzene
GC/MS SIM	EPA TO15 SIM	1,4-Dichlorobenzene
GC/MS SIM	EPA TO15 SIM	1,4-Dioxane
GC/MS SIM	EPA TO15 SIM	Acrolein

Air and Emissions		
Technology	**Method**	**Analyte**
GC/MS SIM	EPA TO15 SIM	Benzene
GC/MS SIM	EPA TO15 SIM	Benzyl Chloride
GC/MS SIM	EPA TO15 SIM	Bromodichloromethane
GC/MS SIM	EPA TO15 SIM	Butadiene (1,3-Butadiene)
GC/MS SIM	EPA TO15 SIM	Carbon Tetrachloride
GC/MS SIM	EPA TO15 SIM	Chlorobenzene
GC/MS SIM	EPA TO15 SIM	Chloroethane
GC/MS SIM	EPA TO15 SIM	Chloroform
GC/MS SIM	EPA TO15 SIM	Chloromethane
GC/MS SIM	EPA TO15 SIM	cis-1,2-Dichloroethene
GC/MS SIM	EPA TO15 SIM	cis-1,3-Dichloropropene
GC/MS SIM	EPA TO15 SIM	Dibromochloromethane
GC/MS SIM	EPA TO15 SIM	Dichlorodifluoromethane
GC/MS SIM	EPA TO15 SIM	Ethylbenzene
GC/MS SIM	EPA TO15 SIM	Hexachlorobutadiene
GC/MS SIM	EPA TO15 SIM	m & p Xylene
GC/MS SIM	EPA TO15 SIM	Methyl tert-butyl Ether (MTBE)
GC/MS SIM	EPA TO15 SIM	Methylene Chloride
GC/MS SIM	EPA TO15 SIM	Naphthalene
GC/MS SIM	EPA TO15 SIM	o-Xylene
GC/MS SIM	EPA TO15 SIM	Styrene
GC/MS SIM	EPA TO15 SIM	Tetrachloroethene
GC/MS SIM	EPA TO15 SIM	Toluene
GC/MS SIM	EPA TO15 SIM	trans-1,2-Dichloroethene
GC/MS SIM	EPA TO15 SIM	trans-1,3-Dichloropropene
GC/MS SIM	EPA TO15 SIM	Trichloroethene
GC/MS SIM	EPA TO15 SIM	Trichlorofluoromethane
GC/MS SIM	EPA TO15 SIM	Vinyl Chloride
GC/MS SIM	EPA TO15 SIM	Xylenes, Total
GC/MS	EPA TO-13A	1,2,4-Trichlorobenzene
GC/MS	EPA TO-13A	1,2-Dichlorobenzene
GC/MS	EPA TO-13A	1,3-Dichlorobenzene
GC/MS	EPA TO-13A	1,3-Dinitrobenzene
GC/MS	EPA TO-13A	1,4-Dichlorobenzene
GC/MS	EPA TO-13A	1-Methylnaphthalene
GC/MS	EPA TO-13A	2,3,4,6-Tetrachlorophenol
GC/MS	EPA TO-13A	2,4,5-Trichlorophenol
GC/MS	EPA TO-13A	2,4,6-Trichlorophenol
GC/MS	EPA TO-13A	2,4-Dichlorophenol

Air and Emissions		
Technology	**Method**	**Analyte**
GC/MS	EPA TO-13A	2,4-Dimethylphenol
GC/MS	EPA TO-13A	2,4-Dinitrophenol
GC/MS	EPA TO-13A	2,4-Dinitrotoluene
GC/MS	EPA TO-13A	2,6-Dichlorophenol
GC/MS	EPA TO-13A	2,6-Dinitrotoluene
GC/MS	EPA TO-13A	2-Chloronaphthalene
GC/MS	EPA TO-13A	2-Chlorophenol
GC/MS	EPA TO-13A	2-Methylnaphthalene
GC/MS	EPA TO-13A	2-Methylphenol
GC/MS	EPA TO-13A	2-Nitroaniline
GC/MS	EPA TO-13A	2-Nitrophenol
GC/MS	EPA TO-13A	3&4-Methylphenol
GC/MS	EPA TO-13A	3,3'-Dichlorobenzidine
GC/MS	EPA TO-13A	3-Nitroaniline
GC/MS	EPA TO-13A	4,6-Dinitro-2-methylphenol
GC/MS	EPA TO-13A	4-Bromophenyl phenyl ether
GC/MS	EPA TO-13A	4-Chloro-3-methylphenol
GC/MS	EPA TO-13A	4-Chloroaniline
GC/MS	EPA TO-13A	4-Chlorophenyl phenyl ether
GC/MS	EPA TO-13A	4-Nitroaniline
GC/MS	EPA TO-13A	4-Nitrophenol
GC/MS	EPA TO-13A	Acenaphthene
GC/MS	EPA TO-13A	Acenaphthylene
GC/MS	EPA TO-13A	Aniline
GC/MS	EPA TO-13A	Anthracene
GC/MS	EPA TO-13A	Benzo(a)anthracene
GC/MS	EPA TO-13A	Benzo(a)pyrene
GC/MS	EPA TO-13A	Benzo(b)fluoranthene
GC/MS	EPA TO-13A	Benzo(g,h,i)perylene
GC/MS	EPA TO-13A	Benzo(k)fluoranthene
GC/MS	EPA TO-13A	Benzoic Acid
GC/MS	EPA TO-13A	Benzyl Alcohol
GC/MS	EPA TO-13A	Benzyl butyl Phthalate
GC/MS	EPA TO-13A	Biphenyl
GC/MS	EPA TO-13A	Bis(2-chloroethoxy) Methane
GC/MS	EPA TO-13A	Bis(2-chloroethyl) Ether
GC/MS	EPA TO-13A	Bis(2-chloroisopropyl) Ether
GC/MS	EPA TO-13A	Carbazole
GC/MS	EPA TO-13A	Chrysene

Air and Emissions		
Technology	**Method**	**Analyte**
GC/MS	EPA TO-13A	Bis (2-ethylhexyl) Phthalate
GC/MS	EPA TO-13A	Dibenz(a,h)anthracene
GC/MS	EPA TO-13A	Dibenzofuran
GC/MS	EPA TO-13A	Diethyl Phthalate
GC/MS	EPA TO-13A	Dimethyl Phthalate
GC/MS	EPA TO-13A	Di-n-butyl Phthalate
GC/MS	EPA TO-13A	Di-n-octyl Phthalate
GC/MS	EPA TO-13A	Fluoranthene
GC/MS	EPA TO-13A	Fluorene
GC/MS	EPA TO-13A	Hexachlorobenzene
GC/MS	EPA TO-13A	Hexachlorobutadiene
GC/MS	EPA TO-13A	Hexachlorocyclopentadiene
GC/MS	EPA TO-13A	Hexachloroethane
GC/MS	EPA TO-13A	Indeno(1,2,3-c,d) Pyrene
GC/MS	EPA TO-13A	Isophorone
GC/MS	EPA TO-13A	Naphthalene
GC/MS	EPA TO-13A	Nitrobenzene
GC/MS	EPA TO-13A	n-Nitrosodimethylamine
GC/MS	EPA TO-13A	n-Nitrosodi-n-propylamine
GC/MS	EPA TO-13A	n-Nitrosodiphenylamine
GC/MS	EPA TO-13A	Pentachlorophenol
GC/MS	EPA TO-13A	Phenanthrene
GC/MS	EPA TO-13A	Phenol
GC/MS	EPA TO-13A	Pyrene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	1-Methylnaphthalene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	2-Methylnaphthalene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Acenaphthene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Acenaphthylene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Anthracene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Benzo(a)anthracene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Benzo(a)pyrene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Benzo(b)fluoranthene

Air and Emissions		
Technology	**Method**	**Analyte**
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Benzo(g,h,i)perylene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Benzo(k)fluoranthene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Chrysene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Fluoranthene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Fluorene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Indeno(1,2,3-c,d) Pyrene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Naphthalene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Phenanthrene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Pyrene
GC-ECD	EPA TO-4A/TO-10A	PCB-1016
GC-ECD	EPA TO-4A/TO-10A	PCB-1221
GC-ECD	EPA TO-4A/TO-10A	PCB-1232
GC-ECD	EPA TO-4A/TO-10A	PCB-1242
GC-ECD	EPA TO-4A/TO-10A	PCB-1248
GC-ECD	EPA TO-4A/TO-10A	PCB-1254
GC-ECD	EPA TO-4A/TO-10A	PCB-1260
GC-ECD	EPA TO-4A/TO-10A	PCB-1262
GC-ECD	EPA TO-4A/TO-10A	PCB-1268
Preparation	**Method**	**Type**
Acid Digestion (Filters, Solid)	EPA 3050B	Inorganics
Soxhlet extraction of PUF	TO-4A/TO-10A	PCBs in Air
Soxhlet extraction of PUF/XAD	TO-13	Semivolatiles in Air
Florisil Cleanup	EPA 3620B/3620C	Cleanup of pesticide residues and other chlorinated hydrocarbons
Sulfur Cleanup	EPA 3660A	Sulfur Cleanup
Sulfuric Acid Cleanup	EPA 3665A	Sulfuric Acid Cleanup for PCBs

Notes:

1) This laboratory offers commercial testing service.

Approved by: _____ Date: January 20, 2017

R. Douglas Leonard
Chief Technical Officer

Issued: 1/20/17

APPENDIX D

LABORATORY STANDARD OPERATING PROCEDURES

Title: Sample Receiving and Identification

QA Officer: _____ Date: 4/21/16

Laboratory Director: _____ Date: 4/21/16

Author: _____ Date: 4/22/16

Analyst: _____ Date: _____

By signing this SOP, the analyst has read and understood the latest version of the test method; has read and understood this SOP; acknowledges receipt of this SOP; and is responsible for the implementation of this SOP.

Revision History:

Revision	Changes	Date
11	Updated to include subcontracting notification	3/02
12	New COC form, temperature instructions, format and spelling updates	10/02
13	Sample Receipt Policy and Notifications	7/03
14	Added detail to signature requirements, subcontracting, and verification	1/04
15	Aspen Login Instructions	9/05
16	Residual Chlorine Check	4/06
17	Composite Sample Date Add notes for MS/MSD login Internal COC Information & attachment, Updated Subcontract Labs	8/07
18	Minor Procedural changes, Composite sample hold time, removed chlorine test for Bacteria samples(done by analyst),5.3.2 Sample Acceptance Policy added. Reference to SOP 801, 5.3.2 accepting Hazardous materials.	12/10
19	CoC name updated, 5.4.4 container ID for duplicates, label on cap and bottle. 5.7.21 Added Sample Collection Time Requirement	7/11
20	5.2.6.7 Updated to specify ABN 625 and Pest 608 js, 5.1.1 and 5.2.10 O-phos filtration requrmnt updated, Updated COC and SRCR	04/14
21	Preservation check clarification and compliance samples5.2.8-9, revised sub lab section, added reference to SOP- 610, updated Appendices, updated storage.	4/16

1.0 Purpose and Applicability
- 1.1 The purpose of this procedure is to ensure the quality control of sample receipt, and includes provisions for documenting sample integrity, chain-of-custody and the initiation of interfaces with sample storage, and laboratory analyses.

2.0 Definitions
- 2.1 Sample: In general, a unit of matrix enclosed in a single container or multiple containers with the same client's field identification.
- 2.2 Sample Group: One or more samples comprising a set as defined by a client's request. This is received and logged-in under one lab number.
- 2.3 Lab Number: A unique serialized number which identifies a sample group. This is also the number which is used for reporting and tracking purposes.
- 2.4 Sample ID Number: A unique number assigned to each sample during the receiving process. This number consists of the ARA Lab Number followed by a numerical suffix serialized to account for the number of samples in a sample group. Single-letter suffixes are added when interchangeable replicates are received.
- 2.5 Sample Receipt Condition Report (SRCR): A form initiated during the receiving process which identifies information about the condition that the samples are received in, the chain-of-custody, and if the samples were received under method specific requirements.
- 2.6 Chain-of-Custody Form (COC): A form which accompanies samples during delivery. It serves to document client information, field collection information, and transfer of samples between the client and ARA personnel.
- 2.7 Internal Chain-of-Custody Form (ICOC): A form which accompanies samples from receipt at ARALLC through the analysis process and to the point of sample disposal. It serves to document the movement of the sample throughout the lab, and records who is responsible for the sample.
- 2.8 Project Folder: The folder which is used to hold all information of a sample group. This includes raw data, final reports, chain-of-custody, invoices, and anything else pertinent to the project, i.e. any communication between the client and ARA personnel.
- 2.9 Custody Seal: A clearly labeled, signed, and dated closure of a sample shipping container. Custody Seals are used only when requested.
- 2.10 Sample Receipt Notification: A form which is used to communicate discrepancies and sample integrity issues noted during the login process as indicated in the sample acceptance policy.
- 2.11 Sample Label: A water resistant label which adheres to the sample containers and can be written on with indelible ink.

3.0 Applicable Documents/References
- 3.1 ARALLC Quality Assurance Manual
- 3.2 TNI- current standard
- 3.3 DoD QSM – current standard
- 3.4 USACE, “Shell for Analytical Chemistry Requirements”, EM 200-1-3

4.0 Materials and Apparatus
- 4.1 File folders
- 4.2 Photocopier

4.3 Designated Log book for Log-in
4.4 pH paper
4.5 Preservatives
4.5.1 1:1 Nitric Acid Trace Metals Grade (HNO3)
4.5.2 1:1 Hydrochloric Acid (HCl)
4.5.3 1:1 Sulfuric Acid (H2SO4)
4.5.4 10N Sodium Hydroxide
4.5.5 2N Zinc Acetate: 22 grams Zinc Acetate dissolved in 87 mL of de-ionized water.
4.5.6 Sodium Thiosulfate (Na2S2O3)
4.5.7 Potassium Sulfate/Ammonium Hydroxide Buffer ((NH4)2SO4)
4.5.8 Purge & Trap Grade Methanol

Method Summary

4.6 This method of sample receiving, documenting, custody, and integrity will ensure an organized approach to keeping track of the samples brought in for analysis. The samples are checked and noted on a form as to their condition (SRCR). The samples are then "logged in" into a log book which contains general information. The date and time received, date sampled, employee initials, and project name. A unique laboratory number is assigned to the project. The project file is made. Samples are placed in appropriate storage locations. The detailed log-in information is then logged into the computer database.

5.0 Procedure

5.1 Sample Receipt and Inspection

5.1.1 COC: The customer must relinquish custody to the laboratory by signing the COC and indicating the date and time that this occurred. Absolute Resource Associates must accept receipt of samples by signing the COC, indicating the date and time, in the location on the COC noted “Received by Laboratory.” Inspect the content of the cooler to be sure that all the samples are there, record the sample temperature (see 5.2.1), sign the COC, and give the client the yellow copy of the COC.

5.1.1.1 If the following discrepancies exist, they should be discussed with the customer at this time and recorded on the COC.

5.1.1.1.1 Temperature of samples is >6°C and sampled >24hours ago.
5.1.1.1.2 Inappropriate sample containers
5.1.1.1.3 Holding time issues
5.1.1.1.4 Missing information on the COC
5.1.1.1.5 Dissolved analyses not documented as field filtered.
5.1.1.1.6 O-phos not documented as field filtered.

5.1.2 If no COC is present, sign any air bills, FedEx documents or other paperwork that exists. The client must be contacted to acquire the necessary information on the COC. In these circumstances a COC may be completed by the laboratory, and then faxed to the customer for approval. A copy of the COC is placed in the project folder as a permanent record.

5.1.3 Courier: When an ARA courier receives the samples from the customer at a location other than the laboratory, they will ask the customer to sign with date

and time on the COC to relinquish the samples to them. The courier will sign with date and time to take custody of the samples. The courier will notify the laboratory of any discrepancies and ensure that there is ice on the samples. Upon returning to the laboratory, they will relinquish the samples to the login personnel who will sign in the "Received by Laboratory" location.

5.1.4 Shipping: Examine the shipping container. Note on the SRCR if there are any custody seals. If present, are they intact? If no custody seals are used, check NA (Not Applicable) on the SRCR. Check the delivery type which applies on the SRCR. Make any notes regarding the shipping on the SRCR.

5.1.5 Compare the sample identification and sampling dates and times located on the sample bottles with the information provided on the COC. Note any discrepancies and resolve them with the customer.

5.2 Sample Condition

5.2.1 Take the temperature of the samples with the infrared temperature gun, which is NIST calibrated. Do this while the samples are still in the cooler. Aim the temperature gun in the cooler, pointed at the samples. Find a representative spot in the cooler or temperature blank if included, and pull the trigger (The temperature scan should occur where the samples are packed). Scan the sample(s), noting the lowest temperature reading while you are scanning. Note on the SRCR the lowest temperature measured and whether it was received on ice or not. This is important at ARA because some of our clients come directly from the site and the samples have not had time to be cooled to 4 C. If it was received on ice but is not 4 C, the ice is evidence that the chilling process has been initiated. If the temperature is >6 C, and the samples were collected >24 hours ago, immediately notify the Lab Director, or designee, for client contact to be made.

5.2.2 Inspect the contents of the cooler to make sure the samples are not broken. If there is a broken sample, determine whether we have sufficient sample volume in other containers to perform the analysis. Contact the client and discuss options immediately. Note on the SRCR that samples were intact or not. Describe any discrepancies.

5.2.3 Fill in the numbers and types of bottles received. For each preservative type received enter the total number of containers of each size received.

5.2.4 Inspect to make sure there are adequate sample bottles for all analyses requested on the COC and that there is enough volume for each analysis. Note: for projects requiring (1) liter amber containers, a duplicate container is generally needed for one sample in the project, if possible. Note on the SRCR.

5.2.5 Make sure the aqueous volatile organic compound samples are free of "head space". Head space is any air that is present in the volatiles vial. This is done by turning the VOC vial upside down, tapping lightly, and checking for air bubbles. If bubbles are found in each container and the bubble is greater than the size of a pea, the customer must be contacted to receive approval to proceed with the analysis. Note these discrepancies and customer communication on the SRCR. The soil in solid VOC samples must be completely covered with MeOH, with no signs of leakage.

5.2.6 Check preservation. Each preserved sample needs to be checked. Document on the SRCR that all samples were checked and found to be sufficiently preserved as required by noting "all pH<2" or "all >10" etc. For any samples found to be insufficiently preserved, indicate which samples were unacceptable and what actions were taken.

5.2.6.1 DO NOT OPEN VOC, GASES, or TOC SAMPLES TO CHECK FOR PRESERVATION. THESE METHODS CHECK pH DURING THE ANALYSIS PROCEDURE.

5.2.6.2 DO NOT CHECK O&G SAMPLES FOR PRESERVATION. THESE METHODS CHECK pH DURING THE ANALYSIS PROCEDURE.

5.2.6.3 To check preservation take the lid off the sample and check the pH on the lid. Do not stick the paper into the sample. Note on the SRCR all preservations that apply. If a sample was not preserved, add the necessary preservation after making a note on the SRCR.

5.2.6.4 Metals Preservation: If the samples for metals are not preserved at the time of receipt in the laboratory (within 2 weeks of sampling), the laboratory is to preserve the samples with 1:1 nitric acid to pH <2. The sample is to be held for 16 hours, the pH verified to be a <2, then the samples are ready for analysis. Note this on the SRCR, the sample bottle and notify the analyst.

5.2.6.5 Pesticides: If water samples are received for Pesticide analysis the pH of the non-preserved sample must be measured at login. If the sample is not between pH of 5-9, notify extractions personnel to adjust the sample pH as required in the method.

5.2.6.6 Acid Extractables/Base Neutrals (625) and Pesticides (608) Samples: Samples must be checked for residual chlorine upon sample receipt using the test paper in login. If residual chlorine is present, notify the analyst immediately so that sodium thiosulfate can be added to the samples per the associated method.

5.2.7 Check to make sure that all samples arrived within method holding time. Note on the SRCR. If anything has a short holding time, <72 hours, notify the analyst who performs that particular parameter. If a sample is beyond holding time, make a note on the SRCR and contact the client immediately to get authorization to proceed with analysis.

5.2.8 Check the COC for a due date. Notify analysts of any RUSH samples where results are needed in 72 hours or less. Document all pertinent project information in writing and provide this information to all analysts included in the work of the project.

5.2.9 Determine whether the samples are required to achieve compliance for any agency such as NHDES, MADEP, DOD, etc. The requirement for compliance should be clearly noted on the COC or the COC will be specific to compliance samples. If compliance analysis is identified, alert the sample receipt supervisor and note the requirement on the SRCR. In the case of NHDES OneStop samples, the sample receipt supervisor will provide an email notification to the appropriate personnel for the uploading requirement.

5.2.10 If there are any other discrepancies with the received sample group, make notes on the SRCR and be sure to contact the client when necessary.

5.2.11 Ortho Phosphate: Samples for O-Phosphate must be filtered in the field (within 15 minutes of sampling) and this should be noted on the COC. If O-phosphate samples are not field filtered, the customer must be contacted for authorization to proceed with analysis. The samples will be filtered prior to analysis at ARA and footnoted on the report.

5.2.12 "First Draw" metals should be collected in a wide mouth bottle.

5.2.13 Dissolved Metals: Samples for dissolved metals should be filtered in the field immediately after sampling. In the event that a customer requests that the laboratory filter the samples upon receipt, this is to be handled as an immediate analysis request. The filtration must be done immediately upon receipt in the laboratory, using non preserved sample. For samples in the MADEP MCP program, the samples are to be filtered, in the lab or in the field, within 24 hours of sample collection. The filtration is through a 0.45um filter and the filtrate is immediately preserved with nitric acid to a pH <2. Record the date, time, final pH and analyst initials for the filtration steps on the SRCR

5.2.14 Once all of this is completed, initial on the "inspected and received by" line and write in the date and time at the bottom of the SRCR.

5.3 Customer Notification and Sample Acceptance Policy

5.3.1 Review the SRCR notes for any discrepancies which require customer notification. Absolute Resource Associates maintains a Sample Receipt Policy which outlines the requirements and information necessary for sample acceptance. This policy is posted in the Login area and is also available on our website. Any discrepancies are documented and discussed with the customer.

5.3.2 We reserve the right to reject any hazardous sample. Any time there is a concern regarding the composition of a sample, it is the responsibility of the sample receiving personnel to request information from the client about any known hazards. Concerns must be discussed with the Lab Director before accepting the sample.

5.3.3 The Sample Acceptance Policy at Absolute Resource Associates, LLC requires the following (see Sample Acceptance Policy QSD-50 for specific information):

5.3.3.1 As per the requirements of TNI accreditation, any samples which are received by the laboratory that are missing information or have sample integrity discrepancies are rejected unless we receive authorization to proceed. Please make sure the following information is complete when submitted with your samples and that sample integrity is maintained by using the correct sample containers and preservation for the analyses required.

- Sample Identification
- Sample Location
- Date and time of collection
- Sample Collector's name

- Preservation Type
- Unique ID on the sample labels
- Proper sample containers
- Sufficient sample volume
- Adequate sample hold time
- Proper sample temperature

5.3.4 The customer must be notified of the discrepancies and authorize the laboratory to proceed with the analysis. If the customer is notified verbally- either in person or by phone- the conversation must be documented on the COC or SRCR. The customer may be notified in writing by completing the Sample Receipt Notification Form, and faxing it to them, along with a copy of the COC. As indicated on the form, if the customer does not respond to the notification, the laboratory will proceed with analysis and note the discrepancies in the report.

5.3.4.1 Missing Preservation Type
5.3.4.2 Sample ID
5.3.4.3 Sample Location (when applicable)
5.3.4.4 Date and Time of Sampling
5.3.4.5 Sample Collector's Name
5.3.4.6 Improper Sample Containers
5.3.4.7 Sample Holding Times Expired Upon Receipt
5.3.4.8 Inadequate Sample Volume Received
5.3.4.9 Sample Receipt Temperature

5.3.5 Samples that cannot be confirmed as to who the customer is, what parameters are needed, what the sample identification is, the nature of the sample to insure safe handling and disposal and the status of the integrity of the sample, are rejected by the laboratory. Explosive materials are rejected.

5.3.6 The laboratory report must unambiguously indicate any unresolved discrepancies noted during the sample receiving process as indicated on the Sample Receipt documents.

5.4 Sample Numbering

5.4.1 Enter the project in the Sample Receiving Logbook. Enter today's date and time, date of sampling, the customer name, project name and initial the entry. Assign the next sequential number in the Sample Login book to the sample group. This is the Absolute Resource Associates, LLC laboratory ID number.

5.4.2 Affix a sticker with the lab number just assigned to the COC, an SRCR, and a tab for the project folder.

5.4.3 On the COC, in the Lab Sample ID column, assign a two digit extension number (beginning with -01) to the laboratory ID number just assigned for each sample. Sample numbers are assigned so that each sample container is uniquely identified by the ARA Lab #, the sequential number assigned per field ID and the test/analysis written on the bottle.

5.4.4 Sample ID Numbering: Once each sample has been assigned an ID number on the COC, label the corresponding sample container. Record the appropriate laboratory sample ID on every bottle received. It may be written

in permanent ink or adhered to the bottle with a "weatherproof" type label. Make sure the container is dry and then label the seven digit sample number (_ _ _ _ _-_ _) on the cap and on the side of the sample bottle itself. Each container in a sample group is uniquely identified by the analysis written on the sample label. Any containers received with the same sample ID and analysis label are distinguished by adding a letter to the Lab Sample number. For example, VOC vials received in duplicate would be labeled 20000-01A and 20000-01B.

5.4.5 Note: If the same sample is to be analyzed both for total and dissolved parameters, such as total and dissolved metals, the sample is given a separate ("dash") number for each test. Similarly, if a sample is received with different preservation techniques for a single parameter, such as HCl preserved VOCs and sodium thiosulfate preserved VOCs, the sample is given a separate ("dash") number for each test.

5.4.6 Note: if only one vial is received for VOC analysis, write "only 1 vial" on the cap of the vial AND enter that into the comments for that sample in login.

5.5 Project Folder

5.5.1 Make up the project folder using the tab which has the corresponding ARA Lab Number on it. Write the client's ID code (three letters) on the tab. The current customer listing can be found in login. The following paperwork is compiled in the project folder: the SRCR, the COC, any air bills, shipping receipts, any sample collection information provided by the customer, memos, corrective actions initiated by either ARA or the client and the Sample Receipt Notification form, if applicable.

5.6 Corrective Action to any Discrepancies With a Sample Group

5.6.1 All customer communications regarding the resolution of issues noted during login are documented on the SRCR.

5.6.2 Any changes to the COC must be dated and initialed by the customer. If the customer requests the laboratory to make the changes, an explicit description of that communication must be found on the SRCR. A copy of the revised COC may be faxed to the customer for their approval. If at all possible, attempt to get the customer to send faxes of revised COC information.

5.6.3 Write initials on the SRCR next to the corrective action instructions along with the date and the name of whom gave the instructions.

5.6.4 If necessary notify the appropriate laboratory personnel detailing the problem and recommended course of action.

5.6.5 If the client cannot be reached, assign the samples in question to "on-hold" status and store them at 4 C in an appropriate refrigerator. Take no further action until the problem can be resolved via communication with the client. Take extreme care and be proactive to insure that every effort is made to get the resolution prior to sample expiration.

5.7 Logging Samples into the Computer: Enter the Aspen data base to log the samples in to the computer. Click on the Aspen leaf icon to open the data base.

5.7.1 Lab #: Under "Active Samples" select "Login Edit," then "Modify Previous Login". This will open the sample login screen. On the bottom left side of the page select "New Sample", then "Create New Batch". This will

automatically assign the next sequential number in the data base, which should match the Lab Number on the project you are preparing to log in. If this is not the case, you must find the missing project prior to entering data in the computer and resolve any discrepancy.

5.7.2 Reference: Review the COC to check for parameters that have combination prices, are set up as project references, or have a test reference. Some tests can be logged in automatically. To review the options for automatic login, click on the pull down menu under "Reference ID". For example if the project to be logged in requests RCRA Metals, select "R RCRA 8 Metals" and automatically the 8 metals, plus the digestion code will be populated in the requested analysis section of the login screen. Some customers also have routine samples that are received and these may also be set up as a reference to log in automatically, review these options prior to logging in the samples. It is important to have any reference codes entered prior to entering any other sample information.

5.7.3 Client ID: Move the cursor to the Client ID box. From the pull down list, select the customer ID. Click on the detail button and select the contact from the pull down list, to match the customer listed on the COC form. If the name is not on the list, type in the name. Check the bill to address information on this same page and make any necessary changes as noted on the COC form. Close this screen to return to the login page. On the right side of the page notice the client project list. This list will include any project specific information for this client. Review these notes and apply any that apply to the customer or project.

5.7.4 Client Project #: Enter the project number, if any, as provided on the COC.

5.7.5 Client Project Name: Enter the project name, if any, as provided on the COC.

5.7.6 State of Origin: Enter the state from which the samples were collected.

5.7.7 Program/Protocol: Select the program/protocol as specified on the COC. For MA work, select MCP QC Report. For projects that are governed by a QA Project Plan and require a QC Report, select QC report. Wastewater projects are to be logged in as NPDES protocol.

5.7.8 Quant Limit Standards: Select the standards as specified on the COC. If none are specified, select None Specified.

5.7.9 EDD Requirement: Select the EDD as requested on the COC. See the Client Project List for customers that routinely request EDD.

5.7.10 PO Number: Enter the PO #, if any, as per the COC or client's project notes.

5.7.11 Received Date: Enter the date received.

5.7.12 Received Time: Enter the time, in military time, received.

5.7.13 Received By: Select or enter the initials of the first person receiving the samples for the lab.

5.7.14 Turnaround: Enter the TAT as requested on the COC or as per the project notes. If there is a due date listed on the COC by the customer, enter the date and select the due date as Firm. All other samples get logged in for 5 day TAT, and select the due date as Internal. Review the customer project list for customer specific due dates.

5.7.15 Due Date: This date will be automatically filled in based on the turnaround requested. Review this to make sure it matches the date requested on the COC.

5.7.16 Received on Ice: Select Yes or No to indicate if the samples were received on ice or not

5.7.17 Received Temperature: Enter the temperature at receipt.

5.7.18 Sample ID: Enter the sample ID. TIP- if you have multiple samples to log in for the lab number, leave this blank until you have logged in all the tests and other information that is the same on other samples in the batch.

5.7.19 Sample Matrix: Enter the sample matrix. Refer to TIP in 5.7.18.

5.7.20 Collect Date: Enter the sample collection date. For composite samples, enter the date the composite ended.

5.7.21 Collect Time: Enter the sample collection time in military time. If no sample time is listed, the earliest time of day will be entered.

5.7.22 Requested Analysis: The analyses as requested on the COC are entered by using the pull down list under the Test Group Library. Review the tests requested on the COC and select the matrix specific tests from the pull down list. Select add test group after each test to enter the test for that sample. Some special notes:

5.7.22.1 Metals: An ICP_Digestion request is needed for all total metals samples.

5.7.22.2 Metals: Wastewater discharge samples (sometimes labelled as wastewater, influent, effluent, discharge, or WW) and residential water samples are to be logged in using method 200.7. If the customer is an industry or a town, the samples are generally wastewater samples.

5.7.22.3 Metals: Groundwater samples, solid samples and waste samples must be logged in using method 6010.

5.7.22.4 PCDry request is needed for all solid samples

5.7.22.5 Use MA MCP codes for all MA work.

5.7.22.6 Review and use Combination codes as needed. If combinations codes are used, the individual tests still need to be entered. Remember to check the fees form and enter $0.00 for the individual tests for any combination tests used.

5.7.23 Prices: Open “Fees Form” tab at the bottom of the screen to check/correct analysis prices. Prices are specific to each customer. This information is contained in several places. Look through the pages of the “Client Project Notes” for either general or project specific pricing information. Open the “View Quotes” tab, and scan for the client code and project name. The quote details show the prices. If no information is available in the notes or the quotes, open I/data/fee schedule and look for a client specific fee schedule. Every effort should be made to correct prices before replicating the sample.

5.7.24 If more than one sample is received and the analyses requests are similar, the first sample entered can be replicated by selecting “Replicate Sample”. Review the options in the box to select replicating only the sample

information or replicating the sample and the tests. Other options exist to copy the information to an existing batch or a new batch. Caution: If you select new batch, a new lab number will be assigned.

5.7.25 If a mistake is made, DO NOT ever delete a sample. Check with the Technical Director, Lab Director or Assistant Lab Director first.

5.7.26 If samples have been replicated, remember to go back to each sample and enter the sample specific information such as sample ID, sample date and time.

5.7.27 Fees Form: Remember to check the fees form and enter $0.00 for the individual tests for any combination tests used.

5.7.28 If rush services are requested, calculate the appropriate surcharge and select the appropriate test code for the surcharge on only one sample in the login. On the fees form, enter the surcharge.

5.7.29 Note: Special notes on the COC, such as “only 1 vial” or “samples on Hold”, or “samples HOT” should be entered on the first sample in the batch, AFTER the replicates have been made. If there are specific notes for a sample, enter the note on that particular sample only.

5.7.30 Once all the information is logged in, double check the information.

5.7.31 Click on the Print button on the bottom of the login screen. Select the LabID Summary and print. Add these pages to the folder.

5.7.32 Take out the COC from the folder and scan it. Use the name “Z COC XXXXX”, the X being the labID number. Move the scanned copy of the COC to the project folder in “O”/data/Reports.

5.7.33 Put the folder in the bin near the "In Progress Project" drawer for secondary review.

5.8 Sample Storage

5.8.1 Samples are stored in a location that meets their thermal preservation requirements and minimizes the chance of sample contamination. Storage temperatures are maintained +/-2 degrees of the specified temperature. Target temperatures of 4 degrees C are maintained above the freezing point of water to 6 degrees C.

5.8.2 VOC Samples: The VOC samples are stored in the VOC sample refrigerator on the racks labeled “to be run”. The Trip Blank, if present is stored with these samples. The duplicate VOC vials are stored in the VOA refrigerator in boxes labeled “Duplicates”, in consecutive order. High concentrations samples are stored in the refrigerator used for Wetlab samples.

5.8.3 Inorganic Samples: Inorganic samples are stored in the Wetlab refrigerator.

5.8.4 Metals Samples: Aqueous metals samples are placed on the shelf in login.

5.8.5 Semi-volatile Organic Samples (non VOC): SVOC samples are stored in Semi-VOA/Extractables refrigerator. O&G samples and samples for surfactants are also stored in this refrigerator.

5.8.6 Solid Samples (non VOC): Solid samples are to be stored in Semi-VOA/Extractables refrigerator. If a large job of solid samples come in, for ease of use store them together in a box. The same is true for large jobs of water samples- combine similar test method bottles in the same box.

5.8.7 If samples are on hold, put a note directly on the bottle so that it is clear to the analyst.

5.9 Internal Chain of Custody

5.9.1 An internal chain of custody is a legal record, documenting the transfer of samples within the laboratory. Based on client specific Quality Assurance Project Plans (QAPPs), samples may require internal tracking throughout the analytical process. An internal COC is completed during the login process and affixed to a box containing the samples. The samples are then stored in the appropriate sample storage area. Names, dates and sample IDs are recorded anytime the samples are relinquished to lab personnel and then received back into storage. The level of security required is specified by the QAAP and followed by ARA LLC.

5.10 Other Notes:

5.10.1 If a sample is modified in any nonstandard way, document this modification on the SRCR. For example, if the samples are split and preserved in login.

5.10.2 Procedures for performing steps necessary to prepare samples for analysis are documented in the "Sample Readiness SOP QA-801". This includes requirements such has compositing, filtering, decanting, sub sampling, particle size reduction and handing multi-phase samples.

5.10.3 If the client initiates any changes after the samples have been logged in, make the appropriate changes on the COC. Record the customer's request on the SRCR. Ask the customer to note the changes on their COC and fax a copy to the laboratory. Notify the analyst of the changes and make a note on the sample container and make the necessary changes in the login database.

5.10.4 If the client requests a MS/MSD on their samples, assign the same number to the MS/MSD samples as has been assigned to the original sample. On the sample bottles, put the sample number and the extension MS on one bottle and MSD on the other bottle. In Aspen, make a note in the notes field on the original sample indicating that this sample is to have an MS/MSD run.

5.11 Subcontracting Samples

5.11.1 The following procedure is for all samples that require sub-contracting laboratories. A note must be left on the communication board informing login personnel of the specific sample ID numbers that need to subcontracted. Certification status must be verified prior to subcontracting samples. If we are subcontracting a parameter for a job that requires an accreditation, we must use a laboratory that holds certification for that parameter (Ie. State certification, TNI or DoD). In addition, the customer must be notified in writing of our intention to subcontract their samples. A copy of that notification must be filed in the project folder or in the customer file. If the subcontracting arrangements are made during the project set up phase, the notation can be included in the quotation for the project. If the samples are dropped off by the customer, write on the COC in the comments section which samples are to be subcontracted. Have the customer sign the COC as usual and keep the yellow copy for their records.

5.11.2 See Subcontracting SOP 610 for detailed information regarding procedures and requirements.

6.0 Secondary Review: Any employee who has been trained in login is approved for secondary review of the login function. The checklist for review can be found on the bottom of the SRCR. A review of all information that has been input into the computer and follow up on discrepancies/customer communication is critical at this point. Any requested changes to the login information are discussed with the person who logged samples into the computer.

7.0 Quality Control Requirements

The quality control requirements of this SOP entail an in-depth review of data input into the computer and a review of the chain of custody documents for discrepancies. Any personnel who is trained in log-in procedures may review another staff member.

8.0 Calculations/Reporting

N/A

9.0 Corrective Actions

9.1 Corrective actions under this SOP may entail contacting clients - where appropriate - to advise them concerning sample volume and container discrepancies or holding time violations. When contacting the client describe the problem, advise them on the protocol/method requirements, and allow the client to make a decision on how to proceed.
9.2 Refer to the ARA QAM, Corrective Action Section and QSD-08 CA Report.

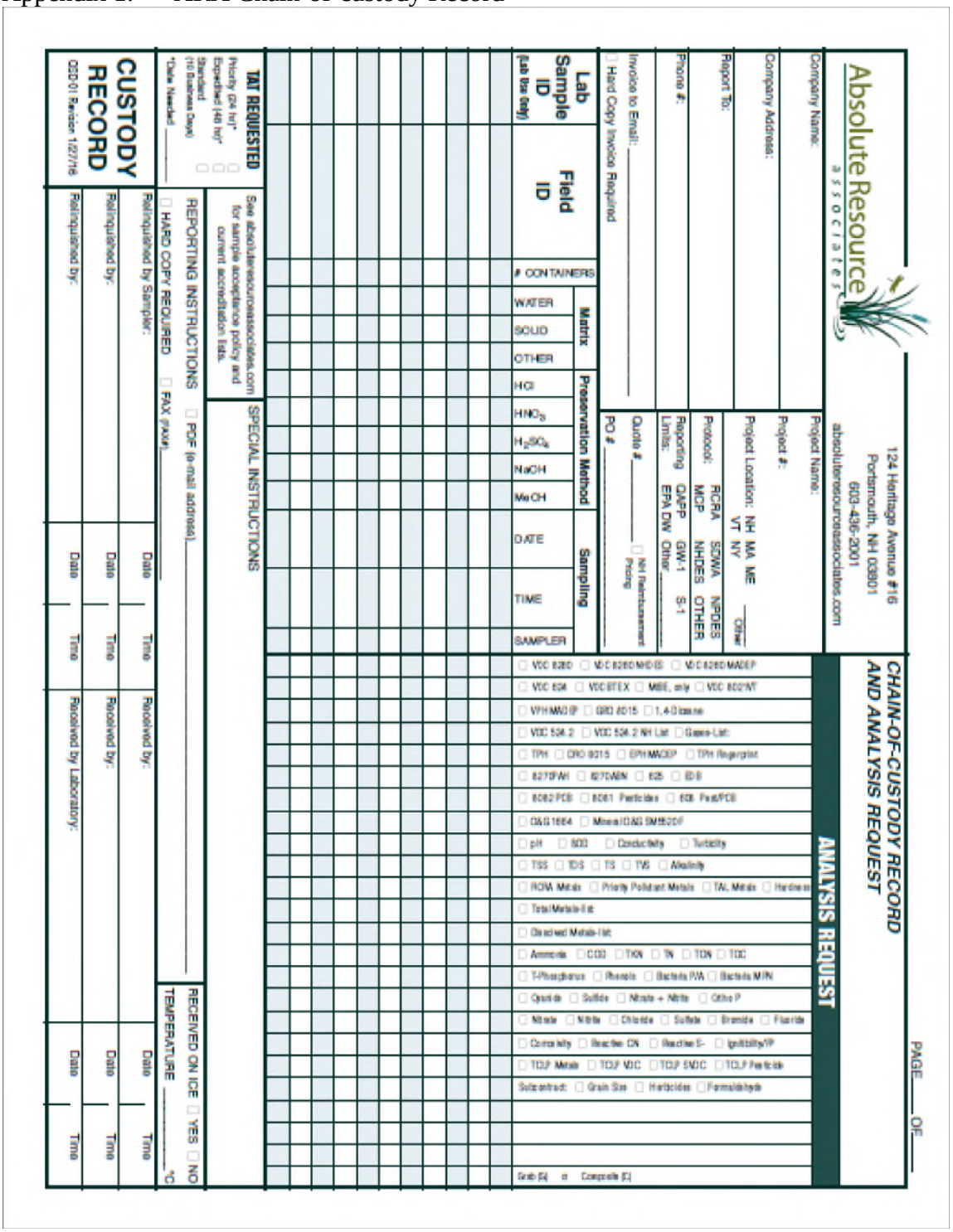
10.0 Responsibilities

It is the responsibility of all personnel trained in sample receipt to follow this SOP. It is also the responsibility for all personnel trained in sample receipt to evaluate all samples for suitability and to be sure there are no discrepancies.

11.0 Health and Safety

All samples are to be treated as unknowns and as potentially hazardous. Samples are to be handled with gloves and goggles or safety glasses. If any unusual sample matrices are noted during login (soaked rags, gasoline odor, colored liquids- a few examples) request assistance from the Lab Director or designee for proper storage and handling guidance.

Appendix 1: ARA Chain-of-custody Record



Absolute Resource associates

124 Heritage Avenue #16
Portsmouth, NH 03801
603-436-2001
absoluteresourceassociates.com

CHAIN-OF-CUSTODY RECORD AND ANALYSIS REQUEST

PAGE ___ OF ___

Company Name:
Company Address:
Report To:
Phone #:
Invoice to Email:
Hard Copy Invoice Required

Project Name:
Project #:
Project Location: NH MA ME VT NY Other
Protocol: RCRA SDWA NPDES MCP NHDES OTHER
Reporting Limits: GAPP GW-1 S-1 EPA DW Other
Quote #
NH Reimbursement Pricing
PO #

Lab Sample ID (Lab Use Only)
Field ID
Matrix: # CONTAINERS, WATER, SOLID, OTHER
Preservation Method: HCl, HNO_3, H_2SO_4, NaOH, MeOH
Sampling: DATE, TIME, SAMPLER

ANALYSIS REQUEST

- VOC 8260 MDC 8260 MADEP
- VOC 624 VOC BTEX MBE, only VOC 602/MT
- VPH MADEP GRO 8015 1,4-Dioxane
- VOC 524.2 VOC 524.2 NH List Comp-List:
- TPH DRO 8015 EPH MADEP TPH Fingerprint
- 8270 PAH 8270 MBN 625 ID:
- 8082 PCB 8081 Pesticides 608 Pest/PCB
- OAG 1664 Mineral OAG 5W5520
- pH BOD Conductivity Turbidity
- TSS TDS TS TVS Alkalinity
- RCRA Metals Priority Pollutant Metals TAL Metals Hardness
- Total Metals-list:
- Dissolved Metals-list:
- Ammonia COD TKN TN TDN TOC
- T-Phosphorus Phenols Bacteria P/A Bacteria M/N
- Cyanide Sulfide Nitrate + Nitrite Ortho P
- Nitrate Nitrite Chloride Sulfate Bromide Fluoride
- Corrosivity Reactive CN Reactive S- Ignitibility/FP
- TCLP Metals TCLP VOC TCLP SVOC TCLP Pesticide
- Subcontract: Grain Size Herbicides Formaldehyde

Grab (G) or Composite (C)

TAT REQUESTED
Priority (24 hr)* Expedited (48 hr)* Standard (10 Business Days)
*Date Needed: _____

See absoluteresourceassociates.com for sample acceptance policy and current accreditation lists.

SPECIAL INSTRUCTIONS

REPORTING INSTRUCTIONS ☐ PDF (e-mail address)_____
☐ HARD COPY REQUIRED ☐ FAX (FAX#)_____

RECEIVED ON ICE ☐ YES ☐ NO
TEMPERATURE _____ °C

CUSTODY RECORD
CDD01 Revision 1/27/16

Relinquished by Sampler:	Date	Time	Received by:	Date	Time
Relinquished by:	Date	Time	Received by:	Date	Time
Relinquished by:	Date	Time	Received by Laboratory:	Date	Time

Appendix 2

Sample Receipt Condition Report

Absolute Resource Associates **Job Number:** _____

Samples Shipped: ☐-UPS ☐-FedEx ☐-USPS ☐-Other _____
Samples Hand Delivered: ☐-Client ☐-Lab Courier ☐-Other _____
Custody Seals - present and intact: ☐-Yes ☐-No ☐-N/A Comments _____
CoC signed: ☐-Yes ☐-No ☐-N/A Comments _____
Receipt Temp: _____ °C On Ice: ☐-Yes ☐-No ☐-N/A If >6°C, sampled < 24 hr? ☐-Yes ☐-No _____

Chemistry Lab

Preservation	Bottle Size/Type & Quantity				Check pH for ALL samples and document:
HCl	40mL(G)	250mL(P)	500mL(P)	1L(G)	
HNO_3	125mL(P)	250mL(P)	500mL(P)		
H_2SO_4	40mL(G)	60mL(P)	125mL(P)	250mL(P) 500mL(P)	
NaOH	125mL(P)	250mL(P)			
$(NH_4)_2SO_4$	60mL(P)	125mL(P)	250mL(P)		
ZnAc/NaOH	125mL(P)	250mL(P)			
NaS_2O_3	40mL(G)	120mL(P)			Residual Cl:
None (W)	60mL(P)	125mL(P)	250mL(P)	500mL(P) 1L(G)	Bacteria ✓by analyst
MeOH	20mL(G)	40mL(G)			ABN625 _____
None (S)	2oz(G)	4oz(G)	8oz(G)	Syringe	Pest 608 _____

IAQ Lab

Analysis	Type & Quantity				Comments
Mold	Cassette	Bulk	Plate	Tape Lift	
Asbestos	Cassette	Bulk			
Lead	Cassette	Bulk	Wipe		
Air	Tube	SUMMA			

Login Review	Yes	No	N/A	Comments
Proper sample containers				
Enough sample/correct preservative				
VOC Integrity W-no headspace. S-MeOH covers solid in jar, no leaks.				
Samples within holding time				
Immediate tests communicated in writing NO_3, NO_2,o-PO_4, pH, BOD, Bacteria, Surfactants, Turbidity, Odor				
Ortho Phos field filtered & noted on COC?				
Date, time & ID on samples match CoC				
Rushes communicated to analyst in writing?				
Work subcontracted (note on login board)				
Pesticides (608) pH Check (pH5-9)				
Log-in Supervisor notified immediately of following items:				Discrepancies, compliance samples (NHDES, MADEP, DOD etc.)

Inspected and Received By: _____ Date/Time: _____

Reviewer's Checklist

☐ Client ID/Project Manager ☐ TAT/Rushes Communicated ☐ Sample IDs ☐ Analyses in Correctly
☐ Project Name ☐ Received Date/Time ☐ Matrix -references
☐ QC Report Required ☐ On Ice, Temperature? ☐ Date/Time Collected -Wastewater Methods
☐ EDD ☐ Sub'd samples sent? (CoC in folder) ☐ Short HT's Communicated ☐ Notes from CoC in LIMS

Reviewed By: _____ Date: _____

Notes: (continue on back as needed)

	Initials	Date	What was sent?
Uploaded / PDF / Fax	_____	_____	Report / Data / _____
Uploaded / PDF / Fax	_____	_____	Report / Data / _____
Uploaded / PDF / Excel	_____	_____	Report / Data / _____

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Appendix 3

Absolute Resource Associates

Preservation, Bottle Type and Holding Time Chart- **Aqueous**

Analysis- AQUEOUS	Preservation	Glass(G)/ Plastic(P)	Size(mL)	Holding Time	Combinations/Notes	Analyst
ABN/PAH 8270/625	None	G-Amber	1000	7days	Extra bottles may be sent for QC	Allison&Chuck
Acidity	None	P	125	Jan-00	Chlorophyll, color, pH	Ann, Alison
Alkalinity	None	P	125	14days	Needs separate container	Ann
Ammonia (NH3)	H2SO4 (pH<2)	P	250	28days	COD, Total Phosphorus, TKN	Ann
Anions(NO2, NO3,Cl, Br, O-PO4*, SO4)	None	P	60	NO2/NO3-O-PO48hr--Cl, Br,SO4,F-28days	All anions can be performed with one 60mL plastic container.	Anny
Note:*Anions O-PO4 (if filtration is required)	None	P	60	Filter within 15 mins	Syringe & Filter included upon Request	Anny
Anions NO2+NO3	H2SO4 (pH<2)	P	60	28days	3 drop H2SO4 into 60mL Bottle	Anny
BOD	None	P	500	48 hours	pH, Conductivity	Ann, Alison
Chlorophyll	None	G	1L G-Amb	Filter Promptly	Acidity, Color, pH	Alex, Alison
COD	H2SO4 (pH<2)	P	60	28 days	NH3, T-Phos, TKN (in 250mL)	Ann
Bacteria	Sodium Thiosulfate	P-Sterile Container	100	6 hr- ww, 30hr-dw	ww-waste water, dw-drinking water	Andy, AJD
Conductivity	None	P	125	28days	BOD, pH	Ann
Cyanide	NaOH (pH>12)	P	125	14days		Ann
1,4 Dioxane	None	G	2*40	14days	No headspace, HCl preserved vials are acceptable	Larry
DMF	None	G	2*40	14days		Aaron
EDB/ DBCP (504.1)	Sodium Thiosulfate	G	2*40	14days	3 mg sodium thiosulfate per vial	Larry, Andy
EPH	HCl (pH<2)	G-Amber	1000	14days		Allison+Andy+ Chuck
Flashpoint	None	G	250 or 4oz		Must be separate container	Andy
Gases	HCl (pH<2)	G	2*40	14days	Includes methane, ethane, ethene	Chuck
Hexavalent Chromium	(NH4)2SO4 Buffer	P	125	28 days	Unpreserved= 24hr holding time!	Ann, Aaron, Chuck
Metals	HNO3 (pH<2)	P	250	6 months Hg: 28 days		Anny
Odor	None	G-Amber	1L WM	24 hours		Ann, Aaron
Oil & Grease	HCl (pH<2)	G	1000	28 days	Extra bottles may be sent for QC	Allison
Pesticides 608/8081/8082	None	G	1000	7days	Must be pH 5-9 at receipt, Extra bottles may be sent for QC	Allison&Andy
PCB 608/8081/8082	None	G	1000	7days	Extra bottles may be sent for QC	Allison&Andy
pH	None	P	125	15 minutes	BOD, Conductivity	Ann/Alison/ Alex/Chuck
Propylene/Ethylene Glycol	None	G vial	1*40	14 days	No headspace	Larry
Total Phenol	H2SO4 (pH<2)	G-amber	250	28 days		Ann
Sulfide	Zinc Acetate, NaOH (pH>12)	P	125	7 days		Ann
Surfactant	None	G-amber	250	48 hours		Allison
TDS/TSS/TS	None	P	250(500 for TSS)	7days	Must be separate container	Ann
TOC	H2SO4 (pH<2)	G-amber	2*40	28days	No Headspace, Subcontracted	Chuck
TKN	H2SO4 (pH<2)	P	250	28days	COD, T-Phos, NH3	Ann, Alison
TPH 8100/DRO 8015	None	G	1000	7days		Allison&Andy
TPH ME DRO	HCl (pH<2)	G	1000	7days		Allison&Andy
Total Phosphorus	H2SO4 (pH<2)	P	125	28days	TKN, COD, NH3 (in 250mL)	Ann
Turbidity	None	P	125	48 hours		Andy
VOC 624/8260/524.2/VPH/ 8021/8015GRO/MEGRO	HCl (pH<2)	G vial	2*40	14 days	No headspace	Larry
DO	none	Glass BOD Bottle with Glass stopper	300	15 minutes		Ann

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Preservation, Bottle Type and Holding Time Chart- **Solids**

Analysis - SOLID	Preservation	Glass(G)/Plastic(P)	Size	Holding Time	Combinations/Notes	Analyst
ABN/PAH 8270/625	None	G-Amber	4oz	14 days	Can be combined in one 4oz amber jar	Allison & Chuck
TPH 8100/DRO 8015	None	G-Amber	4oz	14 days		Allison & Andy
TPH ME DRO	None	G-Amber	4oz	14 days	*Analyses which can go into clear jars, may also share a 4oz Amber jar with the parameters to the left.	Allison & Andy
Pesticides 8081/8082	None	G-Amber	4oz	14 days		Allison & Andy
EPH	None	G-Amber	4oz	14 days		Allison & Andy
Hexavalent Chromium	None	G-Clear	4oz	30 days		Ann
Ignitability	None	G-Clear	4oz	14 days	Must have it's own container	Andy
PCB 8081/8082	None	G-Clear	4oz	14 days		Allison & Andy
Anions(NO2, NO3,Cl, Br, O-PO4, SO4)	None	G-Clear	4oz	NO2,NO3,O-PO4 7days All other anions 28days		Amy
COD	None	G-Clear	4oz	28 days	All of these parameters can share one 4oz clear jar	Ann
Conductivity	None	G-Clear	4oz	28 days		Ann
Cyanide	None	G-Clear	4oz	14 days		Ann
Metals	None	G-Clear	4oz	180 days, Hg 28 days		Amy
pH	None	G-Clear	4oz	7 days	*If TCLP is required an additional 4oz jar is needed	Ann/Alison/ Alex/Aaron
Sulfide	None	G-Clear	4oz	7 days		Ann
TS	None	G-Clear	4oz	7 days		Ann
TKN	None	G-Clear	4oz	28 days	*Analyses which require amber jars cannot be taken from clear jars	Ann/Alison
Ammonia (NH3)	None	G-Clear	4oz	28 days		Ann
Total Phosphorus	None	G-Clear	4oz	28 days		Ann
VOC VPH	MeOH (10mL)	G-Clear	40mL	28 days	Multiple VOC methods, and GRO can share one MeOH preserved vial	Larry
VOC 8260/8021/8015GRO/MEGRO	MeOH (10mL)	G-Clear	40mL	14 days		Larry

124 Heritage Avenue #16 | Portsmouth, NH 03801 | 603.436.2001
absoluteresourceassociates.com

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Appendix 4

Sample Receipt Notification

The following discrepancies were noted upon receipt of your samples at Absolute Resource Associates, LLC (see attached COC). The laboratory will proceed with analysis unless we are notified to do otherwise. Please note that the laboratory is required to include notations in the report for any samples which have the following discrepancies. Thank you for your attention to this matter.

Missing Information	Comments
Missing Preservation Type	
Sample ID	
Sample Location (State)	
Date and Time of Sampling	
Sample Collector's Name	

Sample Integrity	Comments
Improper Sample Containers	
Sample Holding Times Expired Upon Receipt	
Inadequate Sample Volume Received	
Sample Temperature not at 4C +/- 2C	

Customer Response: Please complete and fax back to (603)430-2100

O Proceed with analysis as requested.

O Do not proceed with analysis until notified

Signed: _____

Date: _____

Lab Number: Date Faxed:

Appendix 5

Absolute Resource associates

NOTICE TO SAMPLE COLLECTION PERSONNEL

The Sample Acceptance Policy at Absolute Resource Associates is as follows:

Samples are accepted at the discretion of ARA. Safe handling protocol and disposal guidelines for potential hazards, from the sample matrix or contaminants, must be outlined in the ARA Chemical Hygiene Plan and ARA Waste Characterization and Disposal Procedures.

Samples that will not be accepted include, but are not limited to, those containing explosive or radioactive materials.

As per the requirements of TNI accreditation, any samples which are received by the laboratory that are missing information, have discrepancies or integrity issues must be rejected unless we receive authorization to proceed.

Please make sure the following information is complete when submitting your samples and that sample integrity is maintained by using the correct sample containers and preservations for the analyses required. We will contact you to obtain missing information or to advise you of any issues that could potentially impact data quality.

Sampling location
Date and time of collection
Initials of sample collector
Unique sample identification on the sample labels, consistent with COC.
Proper sample containers and preservations
Adequate holding time to complete analysis
Sufficient sample volume
Proper temperature

If you have any questions, please contact us at 603-436-2001.

Thank you for your help.

Appendix 6

Internal Chain of Custody Form

Project number:_____
Customer name:_____
Date Received:_____

INTERNAL CHAIN OF CUSTODY

Sample #	Storage Location	Date Stored	Stored by	# of Bottles

Sample #	Transferred To	By	Date	Time

Instructions: When taking the samples for analysis, please enter the sample numbers, the location that samples are transferred to, enter your initials and the date. If the reason for the transfer is analysis, enter the word **analysis** in the **Transferred To** section. When you are done with the samples, again record the sample numbers, the location **Transferred To**, your initials and the date. If the sample is completely used during the analysis, enter the word **empty** in the **Transferred To** section.

Title: Laboratory and Sample Waste Characterization and Disposal

QA Officer: Jennifer Guerette Date: 2-1-13

Laboratory Director: [signature] Date: 2-1-13

Author: Ann M Plante-Alexander Date: 2-6-13

Analyst: _____ Date: _____

By signing this SOP, the analyst has read and understood the latest version of the test method; has read and understood this SOP; acknowledges receipt of this SOP; and is responsible for the implementation of this SOP.

Revision History:

Revision	Changes	Date
2	Updated Co. Name	5/11
3	Added waste streams	1/13

Title: Laboratory Waste Disposal

1.0 Purpose and Applicability

This standard operating procedure describes how laboratory waste and client samples are characterized for waste disposal, transferred for disposal, and how they are disposed of. It should be noted this company does not accept samples known to be explosive, radioactive, bio-hazardous, or that contain dioxins or furans. ARA samples must be disposed compliant to all DOT, EPA and New Hampshire State regulations.

2.0 Definitions

Basic Waste This is an aqueous waste that is caustic. Its main components are water, sodium hydroxide, and generally waste has a pH >9.

COD Waste This waste stream is generated during the COD analysis. It is very acidic and contains hazardous levels of mercury.

Cyanide Liquids This aqueous waste stream is caustic. Its main components are water, sodium hydroxide, and pyridine.

Cyanide Solids This solid waste stream contains solid samples containing cyanides.

Dilute Metals and Acids This aqueous waste stream is acidic containing concentrated levels of nitric and hydrochloric acid. pH <2.

TCLP Metals Waste This waste stream contains the TCLP leachate and the solid sample with hazardous levels of TCLP metals (arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver).

TCLP Non Metals Waste: Samples containing hazardous levels of other TCLP-related test compounds (VOC, Pesticides, ABN) are segregated into another waste stream if the results should exceed the TCLP Limits. As this is an unusual circumstance, there is no regular waste stream set up for these wastes. This waste stream will be handled on a case by case basis between the Waste Management personnel and our waste disposal vendor.

Flammable Waste This liquid waste stream contains hexane, methanol, and lesser amounts of diethyl ether, hexanes, toluene, acetone, and oils.

Flammable Waste with Solids This waste stream contains methanol, and lesser amounts

of diethyl ether, hexanes, toluene, acetone, and oils in vials or other sample containers. It also may contain solid residue in vials with methanol from the VOC 5035 sample collection protocol.

Methylene chloride and water This solvent waste stream is comprised of mostly water with methylene chloride, and small percentages of other halogenated solvents (such as chloroform).

Lab Pack Materials Any sample that cannot be characterized into any of the other waste streams listed in this section become a lab pack material. The hazards and characteristics of materials in this category can vary.

PCB Liquids and Solids These waste streams contain polychlorinated biphenyls.

Pesticide Liquids and Solids These waste streams contain pesticides.

Solids Waste This solid waste stream contains unused portions of solid samples and other solids containing non hazardous levels of contaminants.

3.0 Applicable Documents/References

Department of Transportation. . 49 Code of Federal Regulations. 'Transportation.'

Environmental Protection Agency. . 40 Code of Federal Regulations. 'Protection of Environment.'

New Hampshire Department of Environmental Services. 1998. Env-Wm. 'Hazardous Waste Management.'

Occupational Health and Safety Administration. . 29 Code of Federal Regulations, Section 1910.1200. 'Hazard Communication.'

Occupational Health and Safety Administration. . 29 Code of Federal Regulations, Section 1910.134. 'Respiratory Protection.'

Absolute Resource Associates Standard Operating Procedure, QA-604. 'Chemical Hygiene Plan.'

4.0 Materials and Apparatus

Title: Laboratory Waste Disposal

4.1 Waste drums for each waste stream clearly marked with the waste name, start date and generator number.
4.2 Secondary containment system for all liquid waste samples.
4.3 Protective gear: goggles, gloves, lab coats
4.4 pH paper
4.5 Baking soda
4.6 Carboy or bucket (5 gallon for example)
4.7 Spill Kit

5.0 Method/Calibration/Interferences

5.1 Method Summary
This method describes the process used to determine how to dispose of laboratory waste and the procedure for disposal.

5.2 Calibration Procedure
Not Applicable

5.3 Interferences
Not all chemicals are compatible. When handling waste, it is imperative that the source and nature of the waste be understood prior to making any disposal decisions. A chart is attached which can be used as a reference regarding compatibility of wastes. Table 2.

6.0 Procedure

6.1 Sample Waste Segregation

6.1.1 ARA samples must be disposed compliant to DOT, EPA and New Hampshire State regulations.

6.1.2 The first step in the process of sample disposal is segregation of potentially hazardous waste from the non hazardous waste stream. In the event that a sample is determined to be hazardous as a result of the testing done by ARA, the samples are segregated by a flag (orange dot) put on the sample bottle by the analyst indicating the type of hazard. Following are ARA potentially hazardous designations:

PCB	>50ppm solid, >10 ppm liquid
TCLP	> TCLP limits
VOC	> 10ppm solid or liquid
TPH	>10000 ppm solid or liquid
Oil & Grease	>1000 ppm solid or liquid

Title: Laboratory Waste Disposal

Pesticides	>0.4 ppm solid or liquid
ABN	>4000ppm solid or liquid
Cyanide	>10ppm solid or liquid
pH	<2 or >12.5 (except for samples that have been preserved)
Ignitable	Pass
Flashpoint	<140 degrees F
Reactivity	Total Cyanide is >250ppm or Total Sulfide is >500ppm

Any Other Unusual Characteristic

6.1.3 The samples are flagged immediately upon receiving the information of potential hazard. The flagged samples remain in the Sample Receiving storage for 2 weeks after the report is mailed to the customer. The flagged samples are then removed to the waste storage area and are labeled with the date of removal to the waste area and are segregated for lab packing or are combined in an approved waste stream by the Hazardous Waste Coordinator/Manager.

6.1.4 Samples which are stored in the refrigerator for potential re-analysis or customer request are not considered waste until they are removed from the refrigerator.

6.2 Aqueous Sample Waste Disposal

6.2.1 On at least a monthly basis, the sample log in book is reviewed for samples that were collected more than 30 days ago. Unless otherwise requested by the customer, samples that are older than 30 days old, are disposed. Samples that are requested to be held by the customer are to be put in a separate box, labeled with the hold request and dated.

6.2.2 The lab # for the samples being disposed is recorded in the waste disposal logbook. The date of disposal, the matrix (solid or water) the initials of the person doing the disposal, and the means of disposal are recorded in the book.

6.2.3 The samples are removed from the refrigerator or metals area and placed into a labeled box, put in the waste disposal area.

6.2.4 Sample wastes are segregated by their separate waste streams. Samples that are flagged or otherwise noted as unusual are put in a box and labeled "Labpack" wastes.

6.2.5 Prior to disposing of waste, the appropriate personal protective equipment must be worn. Lab coat, safety glasses, and gloves must be worn. An apron, goggles or face shield are also highly recommended and available at the employee request.

Title: Laboratory Waste Disposal

6.2.6 Aqueous sample wastes, that have not been flagged, can be neutralized to a pH of 6-9 and disposed down the drain following the procedure below.

6.2.7 Collect the aqueous samples to be disposed. Assemble a 5 gallon carboy or bucket under a properly working hood. Empty the sample containers into the bucket, being careful to check each container for anything unusual (for example: floating product layer, heavy solids, any type of layering of liquids, unusual colors). Samples with solids should be decanted, and the remaining solids put in the solid waste drum.

6.2.8 It is also helpful to note the type of preservatives being added to the bucket as the basic preserved samples can aid in the neutralization of the acidic sample waste.

6.2.9 If any unusual odors or reactions are noticed during the combining of the waste under the hood, immediately contact the Lab Director or their designee for help.

6.2.10 Start with small batches. When the container is about ¼ full, check the pH. Slowly add baking soda to achieve the desired pH of 6-9. If excessive heat is noticed, add ice. Be sure to gently stir the mixture prior to measuring pH.

6.2.11 When the appropriate pH has been achieved, remove the container from the hood and dispose the neutralized waste in the sink. Run the water in the sink while disposing the neutralized waste and for a few minutes after the waste has been dumped.

6.3 Solid Waste Disposal

6.3.1 On at least a monthly basis, the sample log in book is reviewed for samples that were collected more than 30 days ago. Unless otherwise requested by the customer, samples that are older than 30 days old, are disposed.

6.3.2 The lab # for the samples being disposed is recorded in the waste disposal logbook. The date of disposal, the sample matrix (solid or water), the initials of the person doing the disposal, and the means of disposal are recorded in the book.

6.3.3 The samples are removed from the refrigerator or metals samples storage area and placed into a labeled box and put in the waste disposal area. The date the samples are removed from the refrigerator is marked on the box and they are disposed within 90 days.

6.3.4 Sample wastes are segregated by their separate waste streams. Samples that are flagged or otherwise noted as unusual are put in a box and labeled "Labpack" wastes.

Title: Laboratory Waste Disposal

6.3.5 All other non hazardous solid samples, with no free liquid, can be discarded in the 55 gallon drum labeled "Non Hazardous Waste" solids. The solids can be placed in the drum, in their original containers. Prior to putting any solid waste in any drum, insure that the drum is labeled as specified and that a start date is recorded on the drum.

6.3.6 The waste container (drum) must always be closed after use.

6.3.7 When the container is full, write the date on the drum and notify the Lab Director.

6.4 Laboratory Waste Stream Characterization

6.4.1 The following waste streams have been established at ARA. In the event that a waste is generated that is not on this list, the waste characterization flow chart will be used to determine the disposal/characterization method.

6.4.1.1 Dilute Metals: This stream consists of metals analysis digestates. This waste stream is neutralized and disposed in the sanitary sewer.

6.4.1.2 Volatile Aqueous Rinse Water Waste: This stream consists of rinse water produced in the loading and rinsing of samples and standards for VOC analyses. This waste stream is neutralized and disposed in the sanitary sewer

6.4.1.3 Aqueous Methylene Chloride Waste: This stream consists of mostly water with methylene chloride residue resulting from organic extractions. Small amounts of hexane may also be added to this stream (<5% of total volume in 55 Gallon drum). DOT Shipping Label: Hazardous Waste Liquid, N.O.S. (Methylene Chloride)

6.4.1.4 Waste Sulfuric Acid: This waste is generated from the analysis of samples for mercury. It contains up to 5% sulfuric acid, 1% stannous chloride, and 90-95 % water. May also contain some solids 1-2%. DOT Shipping Label: Waste Sulfuric Acid.

6.4.1.5 COD Waste: This is generated from the analysis for COD. Waste contains 10% sulfuric acid, 0.01% chromium, 0.5% mercuric sulfate and 80-90% water. Chromium is >5 ppm and mercury is >0.2 ppm DOT Shipping Label: Waste Corrosive Liquids, N.O.S.

6.4.1.6 Cyanide Liquids: Liquid waste, CN >10 ppm.

6.4.1.7 Cyanide Solids: Solids containing CN >10 ppm.

6.4.1.8 PCB Liquids and Solids: >30 ppm solid, >10 ppm liquid

6.4.1.9 Pesticides Liquids and Solids: > 20 ppm liquid or solid

6.4.1.10 Non Hazardous Solid Waste: Consists of disposed solid samples, sodium sulfate, sand, petroleum contaminated solids and

NO Free Liquids. DOT Shipping Label: Non Hazardous, Non Regulated

6.4.1.11 Vials with Methylene Chloride: 1-5 mL vials containing methylene chloride are added to this waste stream. These vials are the extracts remaining after the analysis for SVOCs. DOT Shipping Label: Hazardous Waste Liquids, N.O.S.

6.4.1.12 Solid sample waste with Methylenechloride: Waste from methylene chloride extraction including solid samples, sodium sulfate, sand and any other items exposed to DCM. DOT Shipping Label: Hazardous Waste Liquids, N.O.S.

6.4.1.13 Vials containing hexane or methanol are also permitted in this waste stream, however, a separate waste stream has been established for hexane and methanol vial waste. DOT Shipping Label: Hazardous Waste Liquids, N.O.S.

6.4.1.14 Vials with Hexane and Methanol: 1-5 mL vials containing hexane and methanol are in this waste stream. The methanol vials also contain up to 5 g of solid residue. DOT Shipping Label: Waste Flammable Liquids, N.O.S. (Hexane, methanol)

6.4.1.15 Cyanide Test Waste: This waste is generated from the analysis of Cyanide. It contains 3-5% pyridine, <1% HCl, <1% NaOH, <1% Barbituric Acid and 75-90% water. DOT Shipping Label: Non DOT Regulated Material.

6.4.1.16 TCLP Samples: Contains the leachate and solid residue from the TCLP analysis known to exceed the TCLP limits for metals. DOT Shipping Label: Waste, Toxic, Solids, Inorganic, N.O.S. (specify the metals that failed, eg. Pb, Cd)

6.4.1.17 Physiologically Available Cyanide Waste: Contains 0.40ppm Cd in a basic solution. This waste is lab packed.

6.4.1.18 Phenol Waste: Trace amounts of aminoantipyrine, potassium ferricyande in a basic solution. Neutralize and dispose in sanitary sewer.

6.4.1.19 Sulfide Waste: Waste is acidic. Contains trace amounts of ferric chloride, N,N dimethyl phenylenediamine in sulfuric acid solution. Neutralize and dispose in sanitary sewer.

6.4.1.20 TMAOH: Tetramethyl ammonium hydroxide solution. This waste is lab packed.

6.4.1.21 Hexane/Sulfuric Acid vials: 1-5mL vials containing pcb's in hexane and concentrated sulfuric acid. DOT Shipping Label: Hazardous Waste Liquids, N.O.S.

6.4.1.22 Total Kjeldahl Nitrogen: Non mercury liquid waste. See profile.

6.4.1.23 Spent Asbestos Sample Cartidges: cassettes, filters, slide and other related waste. See waste profile.

6.4.1.24 Lab Pack: Various flagged samples and expired chemicals are compiled for lab packing. An inventory of lab pack materials is made when the material is designated a waste and moved to the waste storage area. Materials are segregated, labeled and comply with the 90 day storage requirements for hazardous waste as appropriate.

6.5 Removal of Full Waste Containers

6.5.1 Correspondence with transport and disposal firms is maintained by the Hazardous Waste Coordinator/Manager or designee.

6.5.2 All applicable standards found in 49 CFR (DOT, Transportation) are complied with in the transport and disposal of ARA hazardous wastes.

6.5.3 Transports are scheduled by the Hazardous Waste Coordinator/Manager for disposal. All manifests, Land Ban forms, and other legal documentation is filled out by the Hazardous Waste Coordinator/Manager. All documentation is retained by the Hazardous Waste Coordinator/Manager.

7.0 Quality Control Requirements

7.1 Not Applicable

7.2 Initial Demonstration Requirements

7.2.1 Prior to performing this procedure the employee must be familiar with the emergency procedures in the laboratory.

7.2.2 Prior to performing this procedure, the employee must be familiar with the MSDS forms for all known chemicals in use.

8.0 Responsibilities

1) It is the Hazardous Waste Coordinator/Manager's responsibility to assure all portions of this document are complied with. It is also the Hazardous Waste Coordinator/Manager's responsibility to revise this document as needed.

2) It is the responsibility of all employees to notify the Hazardous Waste Coordinator/Manager of any samples suspected to be potentially hazardous and flag them appropriately for the safety of all employees. It is also every employee's responsibility to notify the Hazardous Waste Coordinator/Manager of

any problems they may have, or alterations they make, concerning their role in this document.

9.0 Health and Safety

9.1 Normal, accepted laboratory safety practices should be followed in accordance with the Chemical Hygiene Plan.

10.0 Pollution Prevention and Waste Management

10.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. During the disposal process, make note of excess sample volumes received. Make recommendations to use smaller sample containers to reduce waste.

10.2 The EPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The waste management procedures are described in this document.

Title: Laboratory Waste Disposal

TABLE 1:
SAMPLE WASTE STREAM CHARACTERIZATION GUIDANCE CHART

1) Is the material a solid? If YES, go to (2).
1) Is the material a liquid? If YES, go to (10)
2) Is the solid HIGHLY REACTIVE, or does it have some other UNUSUAL HAZARDOUS (such as contains asbestos) property? If YES, the material is a LAB PACK SOLID. If NO, go to (3).
3) Does the solid contain PCB's at > 30 ppm? If YES, the material is PCB SOLIDS. If NO, go to (4)
4) Does the solid contain PESTICIDES >0.4ppm? If YES, the material is PESTICIDE SOLIDS. If NO, go to (5).
5) Does the solid contain CYANIDE? If YES, the material is CYANIDE SOLIDS. If NO, go to (6).
6) Does the solid contain SOLVENTS at > 10 ppm? If YES, the material is characterized as FLAMMABLE WASTE WITH SOLIDS. If NO, go to (7).
7) Does the solid contain TCLP levels of metals or organics? If YES for metals, the material is TCLP METALS SOLIDS . If yes for organics, the materials is TCLP Organics Solids. If NO, go to (8).
8) NO to all of the above? If YES, the waste may be discarded as Non Hazardous Solid Waste.
9) Is the liquid HIGHLY REACTIVE, or does it have some other UNUSUAL HAZARDOUS property (such as it contains asbestos)? If YES, the material is LAB PACK LIQUID. If NO, go to (10).
10) Does the liquid contain PCB's at > 10 ppm? If YES, the material is PCB LIQUIDS. If NO, go to (11).

11) Does the liquid contain PESTICIDES? If YES, the material is PESTICIDE LIQUIDS. If

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NO, go to (12).

12) Does the liquid contain CYANIDE? If YES, the material is CYANIDE LIQUIDS. If NO, go to (13).
13) Is the liquid an OIL, or is it FLAMMABLE? If YES, the liquid is FLAMMABLE WASTE. If NO, go to (14).
14) Is the liquid FREON? If YES, the liquid is placed in the USED FREON waste to be recycled. If NO, go to (15).
15) Is the liquid a HALOGENATED SOLVENT? If YES, the liquid is METHYLENE CHLORIDE WASTE. If NO, go to (16).
16) Does the liquid contain SOLVENTS at > 500 ppb, OIL or GREASE at > 100 mg/L, PHENOL at > 100 ppb, or BOD's at > 200 mg/L? If YES, the material is characterized and disposed as Methylene Chloride Waste. If no, go to (17).
17) Does the liquid have a pH greater than 12.5? If YES, the liquid is BASIC WASTE. If NO, go to (18).
18) NO to all of the above? If YES, the liquid is DILUTE METALS AND ACIDS WASTE .

Title: Laboratory Waste Disposal

Table 2-Compatibility Guidance

Chemical Compatibility Chart

EPA-600/2-80-076 April 1980
A METHOD FOR DETERMINING THE COMPATIBILITY OF CHEMICAL MIXTURES

Municipal Environmental Laboratory
Office of Research and Development
U.S. Environmental Protection Agency
Cincinnati, Ohio 45268

***Caution*: This Chart is intended as an indication of some of the hazards that can be expected on mixing chemical wastes. Because of the differing activities of the thousands of compounds that may be encountered, it is not possible to make any chart definitive and all inclusive. It cannot be assumed to ensure compatibiltiy of wastes because wastes are not sclassified as hazardous on the chart, nor do any blanks necessarily mean that the mixture cannot result in a hazard occuring. Detailed instructions as to hazards involved in handling and disposing of any given waste should be obtained from the originator of the waste.**

No.	Reactivity Group Name	1	2	3	4	5	6	7	8	9	10	11
1	Acids, Mineral, Non-oxidizing	1										
2	Acids, Mineral, Oxidizing		2									
3	Acids, Organic		G H	3								
4	Alcohols and Glycols	H	H F	H P	4							
5	Aldehydes	H P	H F	H P		5						
6	Amides	H	H GT				6					
7	Amines, Aliphatic and Aromatic	H	H GT	H		H		7				
8	Azo Compounds, Diazo Compounds and Hydrazines	H G	H GT	H G	H G	H			8			
9	Carbamates	H G	H GT						G H	9		
10	Caustics	H	H	H		H				H G	10	
11	Cyanides	GT GF	GT GF	GT GF					G			11

Legend

Code	Consequences
H	Heat Generation
F	Fire
G	Innocuous and non-flammable gas genera
GT	Toxic Gas formation
GF	Flammable Gas formation
E	Explosion
P	Violent Polymerization
S	Solubilization of toxic substance
U	May be hazardous, but Unknown

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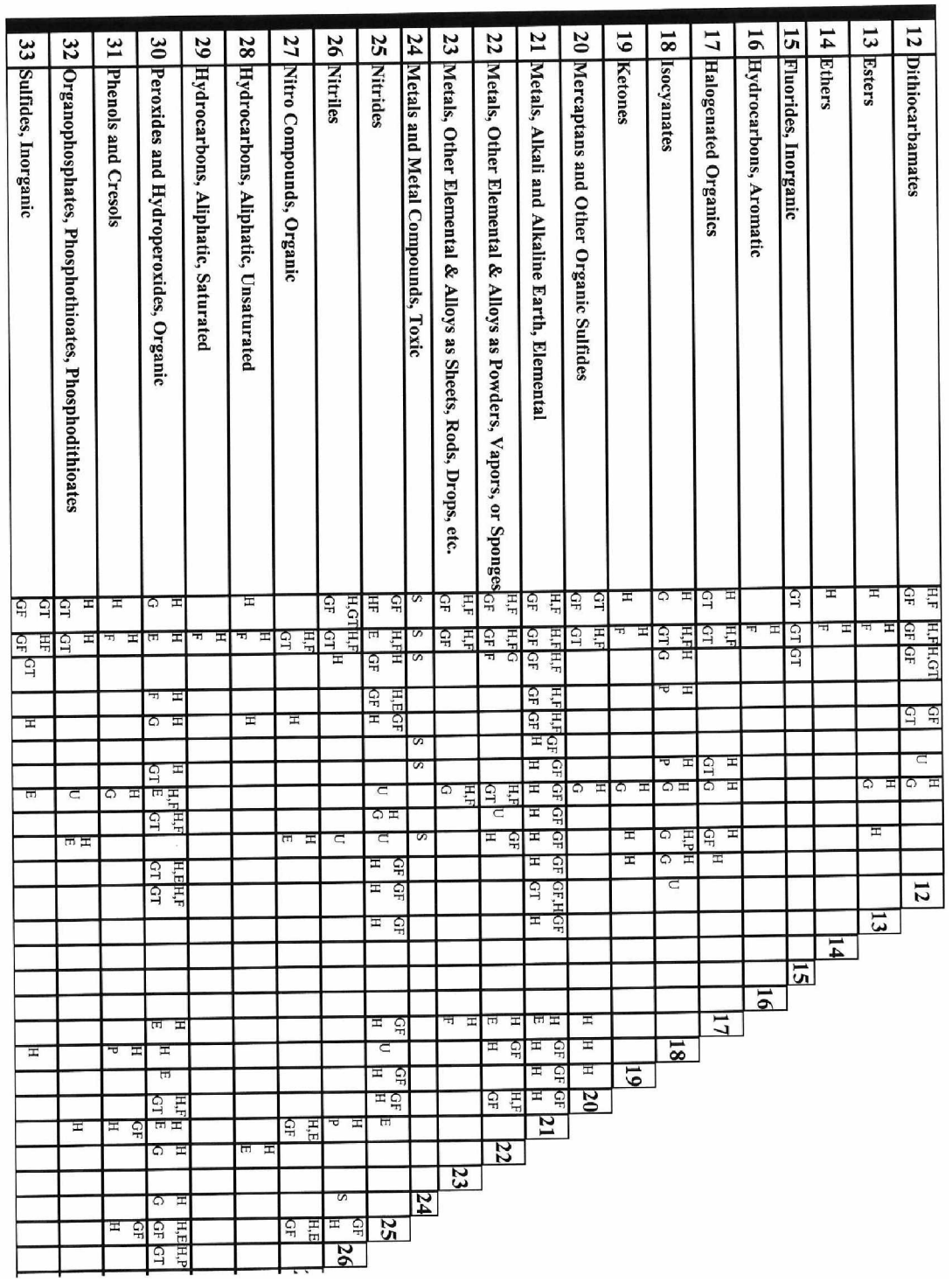
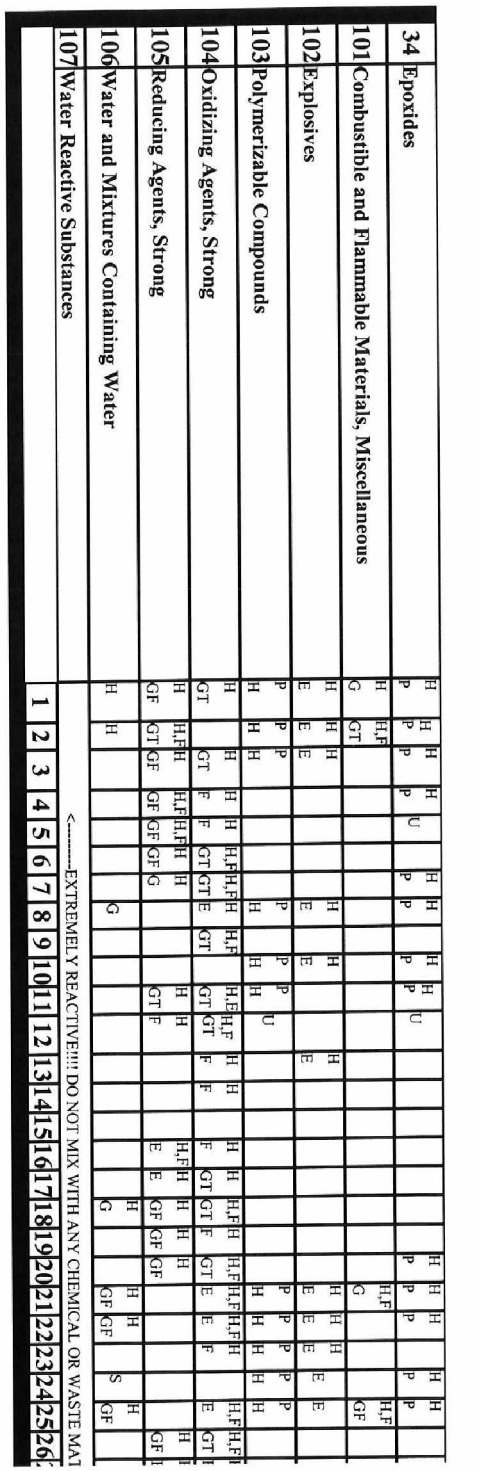


Title: Laboratory Waste Disposal



	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26
34 Epoxides	H P	H P	H P	H P	U		H P	H P		H P	H P	U								H P	H P	H P	H P	H P	H P	
101 Combustible and Flammable Materials, Miscellaneous	H G	H,F GT																			H,F G			H,F GF		
102 Explosives	H E	H E	H E				H E	H E	H E			H E								H E	H E	H E	H E	E E	E E	
103 Polymerizable Compounds	P H	P H	P H				P H		P H	P H	U									P H	P H	P H	P H	P H	P H	
104 Oxidizing Agents, Strong	H GT		H GT	H F	H F	H,F,H GT	H,F,H GT	H,F GT	H,E GT	H,F GT	H,F F	H F	H F			H F	H GT	H,F,H GT	H,F,H F	H,F,H GT	H,F,H E	H,F,H E	H,F,H F	H,F,H E	H,F,H GT	H
105 Reducing Agents, Strong	H GF	H,F,H GT	H,F,H GF	H,F,H GF	H,F,H GF	H G				H GT	H F					H,F,H E	H E	H GF	H GF	H GF					GF	
106 Water and Mixtures Containing Water	H H	H					G										H G			H GF	H GF		S	H GF		
107 Water Reactive Substances	<----------EXTREMELY REACTIVE!!!! DO NOT MIX WITH ANY CHEMICAL OR WASTE MAT																									

ABSOLUTE RESOURCE ASSOCIATES
Standard Operating Procedure QA-5120

SOP Number: QA-5120
Revision History
Cover Page
Page 1

Title: Analysis of VOCs in Water and Solid Samples by EPA Method 8260C

QA Officer: Jennifer Guerrette Date 8/1/17

Laboratory Director: [signature] Date 8/2/17

Author: [signature] Date 8-1-17

Analyst: _____ Date _____

By signing this SOP, the analyst has read and understood the latest version of the test method; has read and understood this SOP; acknowledges receipt of this SOP; and is responsible for the implementation of this SOP.

Revision History:

Revision	Changes	Date
1	Added MCP reference for MADEP work, minor edits	7/04
2	Added addendum to address low level SIM analysis instrument conditions	8/08
3	Deleted old Auto-sampler directions, updated standards, and minor updates and edits.	9/08
4	Corrections and additions to Appendix 1	3/09
5	Changes to Appendix 1 LCS limits to be consistent with 8260, CCV and LCS acceptance clarification, DoD language	12/10
6	Changes to addendum (RLs), updates to Co Name, Curve fit weighting, operating conditions, cal. Standards.	7/11
7	Changes to addendum (cond.& RLs)	6/12
8	ICV,CCV,LCS/D acceptance clarification, typographical fixes, Addendum to address Ethanol analysis instrument conditions, added abbreviations used.	4/13
9	Updated revision from 8260B to C, MDL location, removed CCC and SPCC, acceptance criteria clarification	7/13
10	Add language for references and LOD/Q & MDL file locations, Update addendum 1, 6.1 bfb analysis clarification	4/14
11	Added Storage Blank info & end CCV rqumnt for DoD, update stds. and addendum 1,	2/15
12	Update changes how IS/SS is made, Equipment update, And Addendum update (no VOA1), added method performance sec 7.1	3/16
13	Ethanol Addendum revised to include guidance for Method E1666, Update standards, operating conditions. SOP5000 added to refrcs.	2/17
14	Update to procedure for selecting mass spectrum for tune, sec 6.1.	7/17

Title: Analysis of VOCs in Water and Solid Samples by EPA Method 8260C

1.0 Purpose and Applicability

1.1 This purge and trap gas chromatograph/mass spectrometer method is applicable to the determination of volatile organic compounds including those listed below:

dichlorodifluoromethane	t-amyl-methyl ether (TAME)	bromoform
chloromethane	carbon tetrachloride	isopropylbenzene
vinyl chloride	1,2-dichloroethane	1,1,2,2-tetrachloroethane
bromomethane	benzene	1,2,3-trichloropropane
chloroethane	trichloroethene	n-propylbenzene
trichlorofluoromethane	1,2-dichloropropane	bromobenzene
diethyl ether	bromodichloromethane	1,3,5-trimethylbenzene
acetone	1,4-dioxane	2-chlorotoluene
1,1-dichloroethene	dibromomethane	4-chlorotoluene
methylene chloride	4-methyl-2-pentanone (MIBK)	tert-butylbenzene
carbon disulfide	cis-1,3-dichloropropene	1,2,4-trimethylbenzene
methyl t-butyl ether (MTBE)	toluene	sec-butylbenzene
trans-1,2-dichloroethene	trans-1,3-dichloropropene	1,3-dichlorobenzene
isopropyl ether (DIPE)	2-hexanone	4-isopropyltoluene
ethyl t-butyl ether (ETBE)	1,1,2-trichloroethane	1,4-dichlorobenzene
1,1-dichloroethane	1,3-dichloropropane	1,2-dichlorobenzene
t-butanol (TBA)	tetrachloroethene	n-butylbenzene
2-butanone (MEK)	dibromochloromethane	1,2-dibromo-3-chloropropane
2,2-dichloropropane	1,2-dibromoethane	1,2,4-trichlorobenzene
cis-1,2-dichloroethene	chlorobenzene	hexachlorobutadiene
chloroform	1,1,1,2-tetrachloroethane	naphthalene
bromochloromethane	ethylbenzene	1,2,3-trichlorobenzene
tetrahydrofuran (THF)	m&p-xylenes	1,3,5-trichlorobenzene
1,1,1-trichloroethane	o-xylene	
1,1-dichloropropene	styrene	

1.2 To view reporting limits for analytes listed above refer to Table 4.

1.3 This method is applicable to groundwater and other aqueous samples and solid samples as requested.

1.4 This method is for use only by or under the supervision of analysts experienced in the use of gas chromatography and mass spectrometry and in the interpretation of the resulting data. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 Definitions

2.1 Initial Calibration Verification (Instrument Performance Check) (ICV): A standard at a mid-point in the calibration curve to verify the integrity of the curve. The ICV

is made from a second source standard. All analytes must have a different lot number than the one that made the calibration curve.

2.2 Continuing Calibration Verification (CCV): A CCV is analyzed at the beginning of each analytical batch, when verifying the previous calibration. The CCV is analyzed at a mid-point of the initial calibration curve. The concentration of the CCV may be varied in order to reveal any concentration specific biases.

2.3 Calibration Standard (ICal): Standard solutions prepared from the stock standard at levels corresponding to the calibration curve. These are used to calibrate the instrument response with respect to analyte concentration.

2.4 Laboratory Control Sample (Laboratory Fortified Blank) (LCS): An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly as 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.

2.5 Method Blank (Laboratory Reagent Blank) (PB): An aliquot of reagent water or other blank matrix that is treated exactly as a sample, including exposure to all glassware, equipment, and reagents that are used with other samples. The PB is used to determine if the method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus at levels which may interfere with the analysis. The concentrations found in the PB must be below the reporting limit for each analyte.

2.6 Matrix Spike: An aliquot of a field sample spiked with a known amount of a standard.

2.7 BFB tune: 50ng of bromofluorobenzene is analyzed prior to sample analysis, to observe its mass spectrum. The ion abundances must meet the acceptance criteria as shown in Method 8260C. The BFB tune is valid for 12 hours.

2.8 Carry-over: when a sample is contaminated by analytes left in the purging device, trap, or syringe by a previous sample analysis with high levels of analytes.

2.9 Interferences: Occurrences affecting results on a sample analysis. Examples include: Impurities in the purge gas, solvent vapors in the lab, diffusion of volatile organic compounds through the septum seal into the sample, carry-over from samples with high levels of analytes.

2.10 Trip blank (field reagent blank): a VOC vial filled with reagent water is carried through sampling and handling protocol, and then analyzed along with the other project samples to check for cross contamination.

2.11 Storage blank: a sample of analyte-free water prepared by the laboratory and retained in the VOC storage area, used to detect contamination contributable to sample storage.

2.12 **Abbreviations**

2.12.1 MLCS/D Methanol Laboratory Control Sample/Duplicate

2.12.2 NATL Not at This Level
2.12.3 NDA No data available
2.12.4 NTC Non Target Compound: Notes are added to the results at the discretion of the analyst for occurrences such as presence of NTCs when peaks in the chromatogram are > approx. 2x internal standard peak height.
2.12.5 SIA Sub in Attached- Refers to a result that has to be substituted with the result from another analysis.
2.12.6 SIT Sub in This- Refers to a result that is to be substituted into a report of a previous analysis.
2.12.7 UP Unpreserved- Refers to samples that are run as unpreserved for special analysis. Example: Low level 1,4-Dioxane.

3.0 Applicable Documents/References

3.1 SW846 Revision 3, August 2006, Method 8260C.
3.2 SW846 Revision 3, May 2003, Method 5030C
3.3 Ethanol by SW846 Method 1666 RevA (Addendum 2)
3.4 ARA SOP QA-400 Sample Receiving and Identification
3.5 ARA QA Manual
3.6 ARA Manual Integration SOP QA-5000
3.7 ARA SOP QA-5125 Preparation of Solid Samples for Volatile Organic Analyses
3.8 MADEP MCP Guidance, CAM, July 2010
3.9 Current DoD Quality Systems Manual for Environmental Laboratories

4.0 Materials and Apparatus

4.1 Equipment:

4.1.1 Syringes: Gastight 10uL, 25uL, 50uL, 250uL, 500uL, & 1mL
4.1.2 Volumetric flasks: Grade A, 50mL & 100mL
4.1.3 VOA vials: 43mL glass with Teflon lined silicon septum
4.1.4 10mL Luer fitting syringe
4.1.5 pH paper
4.1.6 Centurion auto-sampler or equivalent
4.1.7 Tekmar-Dohrmann 3100 or 3000 Sample Concentrator: purging device, trap and desorber or equivalent.
4.1.8 Gas Chromatograph: Aligent 6890+ or 5890 series or equivalent.
4.1.9 Column: Rtx-502.2, 30 meter, 0.25mm ID, 1.4um. Source: Restek Chromatography Products # 10915 or equivalent.
4.1.10 Supelco Type K, VOCARB 3000 sorbent trap
4.1.11 Mass spectrometer: Aligent 5973N or 5971 or equivalent.
4.1.12 GC/MS interface: Capillary direct or equivalent.
4.1.13 Data system for quantitation: EnviroQuant

4.2 Reagents/Standards

4.2.1 Purified/deionized water (reagent grade). Source: Made In-House

4.2.2 IS/SS standard: diluted from stock standards to 50ug/mL in Methanol

4.2.3 Primary Standard (main std + gas std) diluted from stocks: 50ug/mL in methanol. Source A: Restek Chromatography Products

4.2.4 Second source standard: (main std + gas std) diluted from stocks: 50ug/mL in methanol. Source B: Absolute Standards and Supelco

4.2.5 Methanol: purge and trap grade. Source: Brand Nu Cat. # 232-1L or other reputable vendor.

5.0 Method/Calibration/Interferences

5.1 Method Summary: Helium is bubbled through a 5mL water sample contained in a purging chamber at ambient temperature. The purgeable analytes are transferred from the aqueous phase to the vapor phase and are swept through a solid sorbent where the analytes are trapped. After purging is complete, the trap is heated to transfer the analytes to the GC. The analytes are separated, and are transferred to the mass spectrometer for detection.

5.1.1 Samples are collected in duplicate in pre-preserved 40mL VOA vials.

5.1.2 Preserved samples must be analyzed within 14 days of sampling. Unpreserved samples must be analyzed within 7 days of sampling.

5.1.3 Samples are to be stored between 0 and 6^{o}C.

5.1.4 Shipment of samples must follow the requirements outlined in the sample login SOP.

5.2 Calibration Procedure

5.2.1 Stock standard solutions are purchased from Absolute Standards, Supelco, Restek, and Accustandard, and are NIST traceable. They are purchased at a concentration of 2000ug/mL in methanol when possible. Upon receipt, each standard is recorded in the Organics Standards Prep Log and assigned a Standard Identification number.

5.2.2 Calibration Standards: Two calibration standards are made at the same concentration, but each from a different source of stock standards. The Primary standard and Second Source standard are each made as two solutions. Dilutions of the stock standards are prepared in methanol with a final concentration of 50ug/mL. A 250uL gas tight syringe is used to transfer the stock standards into a class A 5mL volumetric flask filled with methanol.

5.2.3 Primary Standard: The 8260 “Main Standard” contains the majority of the analytes, which are more stable. It is made by adding 125uL of each of four stock standards to a final volume of 5mL methanol in a class A volumetric flask:

1) Restek mega mix #30431
2) Restek 2-CEVE #30265

3) Restek Oxygenates #30465
4) Restek Custom Mix 1 #561538
5) 250uL of Restek 1,4-Dioxane Standard #30287
6) 50uL of Restek VOA Calibration Mix 1 #30006.

The 8260 "Gas Standard" contains the components which degrade more quickly. It is made by adding 125uL each of stock standard to a final volume of 5mL methanol in a class A volumetric flask:

1) Restek 502.2 mix 1 #30042.
2) Restek Vinyl Acetate #30216

USE: Initial Calibration Curve & Continuing Calibration Verification

5.2.4 Second Source Standard: The 8260 "Main Standard" is made by adding 125uL of each of four stock standards to a final volume of 5mL methanol in a class A volumetric flask:

1) Supelco 524 VOC Calibration mix #5-02111
2) AccuStandard Custom Mix #M-8260-ADD-10x
3) Supelco California Oxygenates mix #4M6872-U
4) Absolute Standards Custom mix #98916

The 8260 "Gas Standard" is made by adding 125uL of stock standard to a final volume of 5mL methanol in a class A volumetric flask: Absolute Standard Mix 1 #30058.

USE: Initial Calibration Verification, Laboratory Control Samples & Matrix Spikes.

Note: Gas Standards are made once a week, Main Standards are made on a six month or earliest expiration indicated on stock vial. If signs of degradation are suspected a standard may be made before any expiration.

5.2.5 Internal Standard/Surrogate Solution (IS/SS)

5.2.6 The Internal and Surrogate solution contains Restek stock standards: IS # 30241,SS #30240, and 1,4-dioxane-d8 #30614, which have a concentration of 2500ug/mL and 2000ug/mL respectively in MeOH. The solution for use in the Centurion auto-sampler reservoir is made by adding 160uL of each of the Internal and the Surrogates plus 200uL of 1,4-dioxane-d8 to a MeOH final volume of 40mL. The final concentration is 10ug/mL.

5.2.7 Standards are stored in the VOA standards freezer. Main standards and IS/SS solutions are made as needed. Gas standards are replaced weekly.

5.3 Interferences

5.3.1 Presence of non-target compounds may interfere with the analysis of the samples. Presence of surfactants can cause the sample to foam during the purging step. Foaming over into the purge and trap apparatus necessitates significant instrument repairs and must be avoided. Samples suspected to

foam must be watched during the purging step. The purging process is aborted if the sample begins to foam over. The sample must be diluted prior to re-attempting analysis. In the event there is no indication of contamination in the sample, the sample is initially analyzed at a dilution. Refer to sample screening in the following section.

5.3.2 Samples can become contaminated by diffusion of volatile organics through the sample container septum during shipment and storage or by dissolution into the methanol used for preservation.

5.3.3 Carry-over can occur whenever a sample is analyzed immediately after a highly contaminated sample. When a highly concentrated sample is anticipated, it is recommended to analyze a blank afterward to check for carry-over.

6.0 Procedure

6.1 Tuning

6.1.1 Per the manufacturer's instructions, instrument parameters are set to achieve the method requirements for tuning as outlined in Table 1. These parameters are reviewed and optimized after instrument maintenance and prior to calibration.

6.1.2 Before analysis begins, 50ng of BFB is analyzed to establish a valid tune window. A dilution of the IS/SS standard mix is purged in order to introduce the BFB to the GCMS. The ion abundances are compared to the method requirements (see Table 1). These BFB criteria must be met before samples may be analyzed. The BFB should be evaluated using "autofind". If the resulting scans are not acceptable, a manual evaluation of an average peak spectrum, including the apex, is performed. The mass spectrum of the BFB should be acquired by selecting the apex of the peak, the scan before the apex and scan after the apex. A background subtraction, using a single scan within 20 scans of the BFB, is required to remove interfering ions. The BFB tune is valid for 12 hours. Note: it is possible to combine the BFB and calibration verification in a single standard as long as tuning and calibration verification acceptance criteria for the project can be met without interferences.

6.1.3 If the tune does not pass the criteria, it is re-analyzed. If it still unacceptable, the instrument is re-tuned and BFB is reanalyzed until all criteria are met.

6.2 Internal and Surrogate Standards: Internal standards and surrogates are added to all samples, blanks and standards. The Centurion auto-sampler adds 5uL of the IS/SS solution from reservoir vial.

6.3 Initial Calibration: Standards are analyzed at several concentrations in order to

describe the calibration range. Analytical results must be bracketed by calibration standards. A typical 8260C calibration curve includes points at 0.5,1, 2, 5, 10, 20, 50, 300, 500, and 800ug/L. The primary main and gas standards are diluted to the appropriate concentration using micro liter syringes and class A volumetric glassware.

6.3.1 The EnviroQuant software is used to construct a multi-point calibration curve. There are three curve fit types available: average response factor, linear regression or quadratic fit. The analyst's discretion is used in selecting the curve fit and weighting which best represents the data, paying particular attention to the low end of the calibration range. Usually, the inverse of concentration is most appropriate weighting. A curve that best fits each analyte is chosen based on the following criteria:

6.3.2 If average response factor is used, RPD is less than or equal to 15%.

6.3.3 If a polynomial fit is used, the correlation coefficient (r^2) is greater than or equal to 0.997 or less than or equal to 1.003.

6.3.4 Each level of the curve is then re-quantitated, using the newly prepared calibration curve, paying particular attention to the highest and lowest levels for each compound. The concentration of each analyte must agree within 30% of its expected value. If an analyte's concentration falls outside these limits, a different curve fit should be considered in order to meet this criterion.

6.4 Initial Calibration Verification

6.4.1 After an initial calibration curve is constructed, an initial calibration verification standard is analyzed to ensure the calibration is accurate. It is made using the second source main and gas standards at a concentration in the middle of the curve, typically at a concentration of 20ug/L. The calibration curve is valid, if all compounds reported by this method are within 20% of the true value. The ICV can be re-analyzed to verify analytes that do not meet the 20% criteria. Analysis may continue if an analyte shows a high recovery and is not detected in the associated samples of any subsequent batches. If an analyte shows low recovery (<80%) in an ICV, the calibration may be used if all of the following conditions are met:

6.4.1.1 The ICV recovery is at least 50%.

6.4.1.2 A point exists in the curve at ≤ ½ the reporting limit, demonstrating detectability at that concentration.

6.4.1.3 The analyte is not detected in any samples analyzed using this calibration. If a sample is found to contain such an analyte, the sample must be reanalyzed with an acceptable ICV or be flagged as an estimated value.

6.5 Continuing Calibration Verification (CCV)

6.5.1 A CCV is run at the beginning of each batch to ensure that the calibration is still valid. The CCV standard is prepared in the same manner as the initial calibration standards, typically at a concentration at or near 20ug/L. The concentration may be periodically varied in order to reveal any concentration specific biases. The CCV is quantitated. It must be within ± 20% of the expected concentration for all analytes that will be reported from the associated samples. Meeting this criterion indicates that the initial calibration is still valid, analysis of samples may proceed, utilizing the initial calibration curve for quantitation. If any of the compounds that are going to be reported are outside of this acceptance criteria, the CCV is still valid if the following criteria are met:

6.5.1.1 If the percent recovery for any compound is above 120% analysis can continue, but any concentrations above the reporting limit for this analyte would need to be re-analyzed or flagged accordingly if re-analysis is not possible.

6.5.1.2 If the recovery for any compound is less than 80%, analysis can continue if the following conditions are met:

6.5.1.2.1 Only 10% of the compounds are outside the acceptance limits.

6.5.1.2.2 The recovery of the analytes are greater than or equal to 50% and a point exists in the curve at ≤ ½ the reporting limit, which demonstrates detectability at that concentration.

6.5.1.2.3 If recovery of any analyte is below 50%, analysis can only continue if a standard is analyzed at the reporting limit and the compound is detectable. Any concentrations for an analyte detected at half the quantitation limit or greater would need to be re-analyzed or flagged accordingly if re-analysis is not possible.

6.5.2 For projects requiring DoD protocol, all compounds of interest in the CCV must be recovered between 80% and 120%. Project level approval would be needed for any analyte that does not meet this criterion. For projects requiring DoD protocol, a CCV is also analyzed at the end of the 12 hour window. All compounds of interest in the ending CCV must be recovered between 50% and 150%. Refer to QAM and DoD QSM for additional guidance.

6.6 Method Blanks: To insure that the GCMS system is free from carryover and there is no contamination in any reagents, a blank is analyzed. There must be no contamination at or above the reporting limit for all compounds reported. For

projects requiring DoD protocol, refer to the QAM for additional evaluation criterion for method blanks.

6.7 Duplicates: Customarily, precision data are obtained by comparison of results of LCS/LCSD. MS/MSD pairs and sample duplicates are also analyzed upon customer request, when adequate sample is supplied. An attempt is made to choose a sample known to have contamination, in order to obtain more meaningful results. Refer to the QA Manual for guidance on evaluating and reporting duplicate precision. MS/MSD,LCS/LCSD: Precision and accuracy are ensured in each analytical batch by analyzing one or both of the following: Matrix Spike/Matrix Spike Duplicate (which may be requested by a customer), Lab Control Sample/Lab Control Sample Duplicate pair (which is typically run near the end of a batch).

6.8 Laboratory Control Samples are performed by adding a known standard, the concentration should be near the middle of the calibration range. The full list of analytes are spiked into soil and water samples. The percent recovery should be between 70% and 130%, except for difficult compounds which may exhibit recoveries between 40% and 160%. Refer to Table 2 for a list of difficult analytes.

6.8.1 Up to 10% of the compounds may be outside the acceptance criteria.

6.8.2 Results may be reported if recoveries below the acceptance criteria, but >40%, a point exists in the curve at ≤ ½ the reporting limit, which demonstrates detectability at that concentration, and the analyte is not detected at ½ the reporting limit.

6.8.3 If recovery of any analyte is below 40%, the batch can only be considered valid if within the batch, a standard is analyzed at the reporting limit and the compound is detectable.

6.8.4 Compounds that are above the acceptance criteria (130%), do not need to be reanalyzed if affected compounds were not detected in associated samples.

6.8.5 The relative percent difference (RPD) between the LCS and LCSD should be less than or equal to 20%. Up to 10% of the full list of compounds can be outside this criterion without re-analysis. If concentrations of the effected compounds are detected in the samples and re-analysis is not possible and more than 10% of the compounds are outside the acceptance criteria, samples are noted of the non-conformance in either the report or the case narrative.

6.9 Matrix spikes are performed by adding a known standard to a sample. Recovery of the analytes follows the same acceptance criteria as the LCS/D. An LCS/D is evaluated in order to determine whether failure is caused by sample matrix interference or a problem with the analytical system. If failure is found to be a matrix interference, this information is included with the final report. If the failure is a result of a problem with the analytical system, the effected samples are re-

analyzed. The relative percent difference between the MS and MSD should be less than or equal to 20% for waters and 30% for solids.

6.10 Sample Preparation

6.10.1 Screening: Historical data can be reviewed to help determine if dilution of a sample is necessary. When high levels of contamination are suspected, it is recommended that an initial diluted run be performed. If necessary, a second analysis can then be done at an appropriate dilution.

6.10.2 Sample Collection: Solid samples are field preserved with methanol. Refer to the VOC Solid sample preparation SOP prior to analysis. Aqueous samples are collected without headspace in 40mL glass "VOA" vials with Teflon coated silicon septa, and preserved with 1:1 HCl. Customers are instructed to store and transport samples on ice. All samples are refrigerated upon receipt and are analyzed within 14 days of sampling. Unpreserved samples require analysis within 7 days.

6.10.3 Sample Analysis: If tune, calibration and blank are acceptable, analysis of samples may proceed.

6.11 Instrument Setup

6.11.1 Centurion Auto-sampler: The purge and trap mechanism is automatically cleaned after each analysis, so manual cleaning is not necessary. The IS/SS solution is added by the auto-sampler. All standards, blanks and samples must be run in 43ml VOA vials. Standards are prepared in 50 or 100mL volumetric flasks and transferred into VOA vials. Solid samples are prepared in 50mL volumetric flasks by adding 1mL of methanol extract to water, bringing to volume (for undiluted analysis), and transferring to VOA vial. Vials are loaded into the tray and the sequence for analysis is entered using the keyboard on the Centurion. Refer to the manufacturers instructions for detailed directions. 5mL of sample is used for undiluted analysis.

6.12 Setting up sequence: To proceed, there must be at least 100 MB space available on the drive where data are acquired. The sequence, which contains the sample information, is entered in the ChemStation software. The ChemStation is located on the computer used for data acquisition. To create a sequence, "Sequence Menu" is selected, then "Edit Sequence Table". Old information is deleted or changed, and new sample information is added, including correct filename, acquisition method, auto-sampler position, sample name and volume. The sequence is saved using the date the sequence starts. Begin analysis.

6.13 Preservation Check: After the sequence is complete, the remaining sample in the VOA vials is checked for adequate preservation using pH paper. The result is recorded in the instrument run log. Any sample with a pH above 2 is flagged in the final report.

6.14 Data Analysis: After a sample analysis is complete, the data are evaluated by

someone experienced with GCMS data evaluation and EnviroQuant software.

6.15 Data files are quantitated using the appropriate data analysis method prepared from the most recent initial calibration. Using the Qedit function, each analyte is reviewed for qualitative and quantitative accuracy using retention time, integration, mass spectrum and secondary ion ratios. Internal standard areas and surrogate recoveries are verified to be acceptable. In the case of failure the sample may be reanalyzed. A quant report is printed for each sample, reviewed and marked with initials and date.

6.16 Sample Calculations: As an internal standard method, response factors are determined by:

$$RF = \frac{(AC)(CIS)}{(AIS)(CC)}$$

where,
AC = Area of compound of interest
AIS = Area of Internal Standard
CC = Concentration of analyte of interest
CIS = Internal Standard Concentration
RF = Response Factor

When average response factor is used, the RF is determined for each calibration level. The response factors are averaged. This average is used to determine the sample concentration:

$$CC = \frac{(AC)(CIS)}{(AIS)(avgRF)}$$

where,
AC = Area of compound of interest
AIS = Area of Internal Standard
CC = Concentration of analyte of interest
CIS = Internal Standard Concentration
avgRF = average relative Response Factor

6.17 In cases where a curve fit is employed, a plot of concentration v. RF is performed using the calibration standard data. Using typical statistical calculations (method of least squares) a curve fit is determined. The equation of this curve is used to determine the sample concentrations.

6.18 In all cases, the Agilent EnviroQuant software is employed to perform the above calculations.

6.19 The generated quantitation report shows results in ug/L units, which for water samples (when multiplied by a dilution factor) can be reported directly.

6.20 For solid samples, sample preparation amounts need be included in the calculations:

Concentration (ug/g)=AF*(0.005*(EV+SW*(1-DF)))/(VA*SW*DF)

Where: AF=Amount found (ug/L) – Quant report Concentration
EV=extract volume (mL) - usually 10mL
SW=soil weight (g)
DF=Dry Fraction (unit-less)
VA=Volume Methanol Analyzed (mL) – usually 0.1mL

7.0 Quality Control Requirements

7.1 **Method Performance**

7.1.1 Initial Demonstration of Performance: This is used to characterize instrument performance. A minimum of four replicates of a standard of a separate source as the initial calibration are analyzed. The recovery for each analyte must be within ±20% of the expected concentrations.

7.1.2 Initial Calibration Verification: After a new initial calibration is performed, the ICV is analyzed from a second source. Its results must be within ±20% of the expected concentration in order to demonstrate accuracy of the initial calibration. See Sec 6.4 for the complete acceptance criteria.

7.1.3 Method Detection Limit: The MDLs are established for all analytes. Reagent water is fortified at a concentration of two to three times the estimated method detection limit. MDLs should be determined initially or whenever there is a significant change in the instrumental configuration.

7.1.3.1 To Determine an MDL, a minimum of seven replicates of the fortified reagent water are processed through entire analytical system. Calculate the MDL for each analyte as follows:

$$MDL = (t)*(s)$$

t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. For seven replicates t = 3.14.

s = standard deviation of the replicate analyses.

MDLs should be determined initially or whenever there is a significant change in the instrumental configuration. The MDL data are stored electronically, by method, in the QA/MDL file folder.

7.1.4 IDC/CDC: Proficiency of the analyst is ensured by documentation of an Initial or Continuing Demonstration of Capability (IDC or CDC). Typically, the analyst analyzes four LCS's with acceptable precision and accuracy or shows acceptable performance of a blind sample analysis (usually a PT). See QAM for additional information.

7.1.5 Control Charts: Control Charts are quality control tools which graphically display QC data over time. The data required for generation of accuracy and precision control charts is maintained by the laboratory. See QAM for additional information.

7.1.6 Inter-laboratory performance: Proficiency Test studies, conducted by approved providers, constitute the majority of inter-laboratory performance comparisons. Proficiency tests are performed at a frequency as required by method, regulation, or accrediting body. See QAM for additional information.

7.2 Laboratory Performance Quality Control and Corrective Actions

7.2.1 Continuing Calibration Verification: The CCV must meet the criteria as described in section 6.5. If the CCV performed at the beginning of a run is unacceptable, the CCV should be remade and re-analyzed , if acceptable the batch can be continued, otherwise corrective action should be taken which may include a new initial calibration. See section 6.5 for more guidance. For projects requiring DoD protocol, a CCV is also analyzed at the end of the 12 hour window. All compounds of interest in the ending CCV must be recovered between 50% and 150%.

7.2.2 Method Blank: The blank is analyzed after the CCV. Contamination in the blank must be lower than the Reporting Limit. If sample results show presence of the contaminant, the reported result must be flagged as possible lab contamination. Blank results are not subtracted from sample results.

7.2.3 Storage Blank: the sample storage blank is analyzed along with analytical samples at a minimum of once every 14 days. If storage blank results show presence of a contaminant above the reporting limit, samples analyzed since the last storage blank was run, as well as samples currently stored with the storage blank, must be checked for presence of that analyte. Samples showing similar contamination above the reporting limit must be flagged accordingly. Efforts must be made to identify and remove the source of the contamination. Once identified, corrective actions must be put in place to prevent a similar situation from recurring.

7.2.4 Laboratory Control Sample/Duplicate (LCS/D): an LCS/D is analyzed in every batch. The percent recovery is calculated and must be within the limits discussed in Section 6.8. LCS failures indicate method problems

and must be corrected. Samples associated with failed LCS/D are reanalyzed, or if no further sample is available, are flagged in the final report.

7.2.5 Matrix Spike: A matrix spike is analyzed when adequate sample exists or upon customer request. A known amount of analyte is added to an aliquot of a randomly chosen sample. The calculation of the percent recovery is as follows:

$$R = (Cs - C)/s * 100$$

R = percent recovery
Cs = Fortified sample concentration
C = Sample background concentration
s = Concentration equivalent of analyte added to the sample

7.2.5.1 If the sample matrix spike fails the criteria in Section 6.9 or other criteria as specified by the customer, the LCS results are reviewed to determine whether the failure is matrix specific or an analytical problem. If found to be matrix specific, the report for the spiked sample is noted with this failure.

7.2.6 Surrogate recoveries: Surrogates are added to all samples and standards. Recovery of surrogates must meet criteria of table 3 or other criteria as specified by the customer. Samples failing these criteria are reanalyzed. If the reanalysis shows acceptable recoveries, these results are reported. If the sample reanalysis shows a similar failure, the report includes a note discussing matrix interference.

7.2.7 Retention Times: The retention times in the analytical system should be stable such that peaks can be accurately identified. Variability in the system is reduced by using the relative retention times from the closest internal standard. The width of the retention time window used to make identifications can be based upon the measurements of actual retention times variations of standards over the course of several days. These retention times and windows are estimates. Three times the standard deviation of a retention time can be used to calculate a suggested window size for each analyte. As a default, 0.3 minutes is used. The experience of the analyst should weigh heavily in the qualitative interpretation of chromatograms.

7.2.8 LOD/LOQ: Refer to QAM *Analytical Procedures* section, for specific LOD/LOQ requirements. LOD/LOQ data are stored electronically in the QA/LOD&LOQ file folder.

8.0 Responsibilities

The analyst uses the Quality Control Criteria outlined in this SOP to evaluate and validate the data. It is the analyst's or technician's responsibility to follow this SOP and record all the data in the lab notebook. Any irregularities within the test or samples should be recorded in the lab notebook and reported to the lab manager who is responsible for reviewing the data.

9.0 Health and Safety

Normal, accepted laboratory safety practices should be followed during reagent preparation and instrument operation. Many of the analytes in this method are toxic or are known or suspected carcinogens. Every sample in the lab should be handled as if it is hazardous waste. All technicians shall be familiar with the Chemical Hygiene Plan (SOP QA604), and SDS location.

10.0 Pollution Prevention and Waste Management

10.0a Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Every effort is made to make only enough standard as will be used prior to expiration.

10.0b The EPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Waste management is regulated by the Hazardous Waste Coordinator. Expired standards in methanol are disposed of in the flammables waste stream. Sample waste is neutralized and is disposed into the municipal waste system.

Table 1—BFB Key m/z Abundance Criteria

Mass m/z	Abundance criteria
50	15-40% of Mass 95.
75	30-60% of Mass 95.
95	Base Peak, 100% Relative Abundance.
96	5-9% of Mass 95.
173	<2% of Mass 174.
174	>50% of Mass 95.

175 . . . . . 5-9% of Mass 174.
176 . . . . . >95% but <101% of Mass 174.
177 . . . . . 5-9% of Mass 176.

Table 2—Difficult Analytes

Compound	Compound
acetone	bromomethane
methyl ethyl ketone	chloromethane
4-methyl-2-pentanone	1,4-dioxane
2-hexanone	
dichlorodifluoromethane	

Table 3—Surrogate Compound Recovery Criteria

SURROGATE STANDARDS	Acceptance Limits
dibromofluoromethane	78-114*
toluene-D8	88-110*
4-bromofluorobenzene	86-115*

* For projects requiring DoD protocol, refer to the DoD Quality Systems Manual for Environmental Laboratories for required surrogate acceptance limits.

Title: Analysis of VOCs in Water and Solid Samples by EPA Method 8260C

Table 4—Typical Analyte Reporting Limits

Compound	ug/L	ug/g	Compound	ug/L	ug/g	Compound	ug/L	ug/g
dichlorodifluoromethane	2	0.1	1,1-dichloropropene	2	0.1	styrene	2	0.1
chloromethane	2	0.1	t-amyl-methyl ether (TAME)	2	0.1	bromoform	2	0.1
vinyl chloride	2	0.1	carbon tetrachloride	2	0.1	isopropylbenzene	2	0.1
bromomethane	2	0.2	1,2-dichloroethane	2	0.1	1,1,2,2-tetrachloroethane	2	0.1
chloroethane	2	0.1	benzene	2	0.1	1,2,3-trichloropropane	2	0.1
trichlorofluoromethane	2	0.1	trichloroethene	2	0.1	n-propylbenzene	2	0.1
diethyl ether	5	0.1	1,2-dichloropropane	2	0.1	bromobenzene	2	0.1
Acetone	50	2.0	Bromodichloromethane	0.6	0.1	1,3,5-trimethylbenzene	2	0.1
1,1-dichloroethene*	1	0.1	dibromomethane	2	0.1	2-chlorotoluene	2	0.1
methylene chloride	5	0.1	4-methyl-2-pentanone (MIBK)	10	0.4	4-chlorotoluene	2	0.1
carbon disulfide	2	0.1	cis-1,3-dichloropropene	2	0.1	tert-butylbenzene	2	0.1
MTBE	2	0.1	toluene	2	0.1	1,2,4-trimethylbenzene	2	0.1
trans-1,2-dichloroethene	2	0.1	trans-1,3-dichloropropene	2	0.1	sec-butylbenzene	2	0.1
isopropyl ether (DIPE)	2	0.1	2-hexanone	10	0.5	1,3-dichlorobenzene	2	0.1
ethyl t-butyl ether (ETBE)	2	0.1	1,1,2-trichloroethane	2	0.1	4-isopropyltoluene	2	0.1
1,1-dichloroethane	2	0.1	1,3-dichloropropane	2	0.1	1,4-dichlorobenzene	2	0.1
t-butanol (TBA)	30	2.0	tetrachloroethene	2	0.1	1,2-dichlorobenzene	2	0.1
2-butanone (MEK)	10	0.3	dibromochloromethane	2	0.1	n-butylbenzene	2	0.1
2,2-dichloropropane	2	0.1	1,2-dibromoethane	2	0.1	DBCP	2	0.1
cis-1,2-dichloroethene	2	0.1	chlorobenzene	2	0.1	1,2,4-trichlorobenzene	2	0.1
chloroform	2	0.1	1,1,1,2-tetrachloroethane	2	0.1	Hexachlorobutadiene	0.5	0.1
bromochloromethane	2	0.1	ethylbenzene	2	0.1	naphthalene	5	0.1
tetrahydrofuran (THF)	10	0.5	m&p-xylenes*	2	0.1	1,2,3-trichlorobenzene	2	0.1
1,1,1-trichloroethane	2	0.1	o-xylene	2	0.1	1,3,5-trichlorobenzene	2	0.1
1,4-dioxane	50	2.0						

*Reported based on lowest point in the calibration curve.
Note: Quantitation limits may vary based on instrument capabilities and customer requirements.

Analytical Conditions VOA5:

ECON Evolution Purge and Trap

Carrier Gas	Helium
Sorbent Trap	VOCARB 3000 (K) ECON
Line Temp	150° C
Valve Temp	150° C
Purge Time	11 minutes
Purge Flow	40mL/min
Desorb Preheat	255° C
Desorb Temp	260° C
Desorb Time	.5 minutes
Bake Time	8 minutes
Bake Temp	265° C

Agilent Technologies 7890A GC METHOD

OVEN

Initial temp: 35° C (On) Maximum temp: 270° C
Initial time: 4.00 min Equilibration time: 1.00 min

Ramps:

#	Rate	Final temp	Final time
1	15.00	220° C	2.0
2	0.0(Off)		

Post temp: 0 'C
Post time: 0.00 min
Run time: 18.33 min

FRONT INLET

Mode: Split
Initial temp: 180 'C (On)
Pressure: 7.179 psi (On)
Split ratio: 50:1
Split flow: 50mL/min
Total flow: 54 mL/min
Gas saver: Off
Gas type: Helium

COLUMN 1

Capillary Column Catalog Number: RESTEK 10915
Rtx-502.2
Max temperature: 270 ºC
Nominal length: 30.0 m
Nominal diameter: 250.00 um
Nominal film thickness: 1.40 um
Mode: constant pressure
Pressure: 7.179 psi
Nominal init pressure: 1 mL/min.
Average velocity: 360169 cm/sec
Inlet: Front Inlet
Outlet: MSD
Outlet pressure: vacuum

THERMAL AUX 2

Use: MSD Transfer Line Heater
Description:
Initial temp: 150 ºC (On)
Initial time: 0.00 min

#	Rate	Final temp	Final time
1	0.0(Off)		

GENERAL INFORMATION

Tune File :bfb.U
Acquisition Mode : Scan

ADDENDUM 1

EDB, DBCP & 1,4-Dioxane Analysis P&T-GC/MS Conditions to meet NHDES Limits

Samples are collected in 40mL VOA vials in duplicate. Samples are to be stored at 4±2°C until time of analysis. Samples do not require a chemical preservative. Sample holding time is 14 days from collection. These analytes are determined at the reporting limits shown below, using a Selected Ion Monitoring (SIM) method. Since the analysis doesn't use scan mode, BFB tune verification is not required. SIM parameters are chosen to collect unique ions for the 3 analytes, an internal standard and a surrogate.

The conditions are the same as outlined in the above SOP, with the following changes:

- Data acquisition method: V3SIMDX2.m
  - SIM method 88 and 96 m/z only
  - Tune file optimized for low mass sensitivity
    VOA3
    BFB524.U
- Heated sparge to 40° C
- Centurion Auto-sampler: Use the 8260 parameters (5mL purge)
- Internal standard = 1,4-dioxane-D8 at 10 ug/L added by the Centurion auto-sampler.
- IS requirements: 0.5 to 2.0 times initial cal. Average

Dilutions of stock standards are prepared in methanol with a final concentration of 50ug/mL.

The primary standard is made by adding 50uL of Restek 1,4-Dioxane and 500uL of Absolute Standards EPA Method 504 EDB & DBCP to a final volume of 2mL in Methanol.

Calibration standard concentrations (ug/L):

EDB & DBCP	p-Dioxane
0.05	0.05
0.2	0.2
0.4	0.4
1	1
4	4
10	10
20	20
50	50

The secondary standard is made from a separate source by adding 50uL of Absolute Standards Custom mix and 500uL of Ultra EPA Method 504.1 mixture to a final volume of 2mL in Methanol.

ICV(ug/L):

4	8

QC Acceptance limits:
ICV: 80-120% recovery
CCV: 70-130% recovery
LCS/LCSD: 70-130% recovery
MS/MSD: 70-130% recovery
LCS/LCSD RPD: 20%
MS/MSD & DUP RPD: 30%

Reporting Limits (ug/L): EDB = 0.05ug/L, DBCP = 0.2ug/L, p-Dioxane = 0.25ug/L.

ADDENDUM 2

Ethanol Analysis P&T-GC/MS Conditions

Ethanol by SW846 Method 1666 RevA

Samples are collected in preserved 40mL VOA vials in duplicate. Samples are to be stored at 0-6°C until time of analysis. Sample holding time is 14 days from collection. This analyte is determined at the reporting limits shown below. A BFB is run as described in section 6.1.

The conditions are the same as outlined in the above SOP, with the following changes:

Run on VOA5

Centurion autosampler:

- No standard added by Centurion

Evolution ECON P&T:

Use Method ETOH
Heated sparge to 40° C

Data acquisition method: V58260.m

Internal Standard: Restek Custom Ethanol-d6 Standard (added manually)

Calibration standard concentrations (ug/L): 100, 200, 500, 1000, and 5000.

Primary Standard: Absolute Standards or equivalent. Uses: Initial calibration and continuing calibration.

Secondary Standard: Restek or equivalent. Uses: Initial calibration verification, Laboratory control sample, and Matrix spike.

QC Acceptance limits:
ICV: 80-120%
CCV: 70-130%
LCS/LCSD: 70-130%
MS/MSD: 70-130%
LCS/LCSD RPD: 20%
MS/MSD & DUP RPD: 30%

Reporting Limit: 100ug/L

Title: Preparation of Solid Samples for Volatile Organic Analyses by 5035A

QA Officer: Jennifer Guenette Date: 2/20/17

Laboratory Director: _____ Date: 2/20/17

Author: _____ Date: 2-20-17

Analyst: _____ Date: _____

By signing this SOP, the analyst has read and understood the latest version of the test method; has read and understood this SOP; acknowledges receipt of this SOP; and is responsible for the implementation of this SOP.

Revision History:

Revision	Changes	Date
1	Change to 10mL of methanol for prep	11/04
2	Update method procedure and correct record keeping, Co name updated	07/11
3	Added "A" to method, to reflect revision being used.	1/13
4	Added Sand cleaning procedure	7/13
5	Update language for references, Added VPH/GRO LCS standard	4/14
6	Update Standards used	4/15
7	Correct amount of VPH standard added to LCSs and MSs. Method performance section added to 7.0	3\16
8	4.2.4 second source stnds updated.	2/17

Title: Preparation of Solid Samples for Volatile Organic Analyses by 5035A

Purpose and Applicability

1.1 This SOP specifies the procedures followed for preparation of field preserved methanolic soil extracts for VOC analysis.

1.2 This method applies to all solid samples methanol preserved in the field for analysis by 8260, 8021, MA VPH, ME GRO and 8015 GRO.

1.3 This method is used only by or under the supervision of analysts experienced in the use of general laboratory equipment.

2.0 Definitions

NA

3.0 Applicable Documents/References

3.1 ARA SOP 402: Bottle Order Preparation
3.2 ARA Volatile Vials for Field Preservation Log
3.3 ARA Sample Readiness SOP QA-801
3.4 SW846 Method 5035A
3.5 ARA soil prep logbook

4.0 Materials and Apparatus

4.1 Equipment:

4.1.1 Laboratory Balance
4.1.2 Centrifuge
4.1.3 VOA vials: 40mL with Teflon lined silicon septum top.
4.1.4 Gas tight syringe, 10uL & 25uL
4.1.5 VOA vial rack
4.1.6 Scupula
4.1.7 Drying oven

4.2 Reagents/Standards:

4.2.1 Sand for blanks: Used as an analyte-free solid matrix for QC. Sand is purchased from a hardware/home improvement store and must be cleaned before use. Place several cups of sand in a fine mesh bag. Rinse with lab grade water to remove fine particles. Bake in a foil loaf pan in the muffle furnace at 500 degrees Celsius for two hours or until the sand is dry. Allow to cool completely. Assign an Organic Standards Prep number.

Title: Preparation of Solid Samples for Volatile Organic Analyses by 5035A

4.2.2 Methanol: Purge and Trap grade. Used for preparation of standards and extraction of solid samples.

4.2.3 α, α, α -trifluorotoluene surrogate solution (TFT): Restek, 2500ug/mL in methanol.

4.2.4 8260 Second Source Standards: The 8260 Second Source "Main Standard"

1) Supelco 524 VOC Calibration mix
2) AccuStandard Method 8260 Additions
3) Supelco California Oxygenates mix
4) Absolute Standards Custom mix

The 8260 Second Source "Gas Standard"

1) Absolute Standard 502/524 High Concentration VOC Mix 1.

Note: The above stands may be purchased from a reputable source such as Supelco, Absolute Standards, etc. as long as they are from a different source than the calibration standard and all analytes of interest are present.

4.2.5 VPH Second Source Standard: MA VPH Standard from Restek or equivalent, but different than vendor for calibration standard.

5.0 Method Summary

5.1 In order to minimize losses as a result of evaporation and microbial action, soils for VOC analysis are preserved with methanol in the field. An equal proportion of soil is dispersed into methanol (usually 10g soil/ 10mL MeOH) with a minimum of disruption at the time of sampling. Samples preserved in this way have holding times of 14 days. The exception is MA VPH, which allows 28 days prior to analysis. A 40mL VOA vial, of known tare weight, pre-prepared with 10mL methanol is sent to the field for sampling. Upon return to the lab, the gross weight is measured to determine the net weight. Surrogate is added, the vial is shaken and allowed to settle prior to analysis. Methanol extracts are stored with aqueous samples for VOC analysis at 6°C to just above freezing.

5.2 Shipment of samples must follow the requirements of the method.

5.3 Calibration Procedure

5.3.1 NA.

5.4 **Interferences:** It is important that Purge and Trap grade methanol is used, as lower grades of methanol may contain significant concentrations of acetone and other target or non-target compounds.

Title: Preparation of Solid Samples for Volatile Organic Analyses by 5035A

6.0 Procedure

6.1 Bottle Preparation: Refer to section 6.9 of SOP 402: Bottle Order Preparation

6.2 Sample Preparation:

6.2.1 Upon receipt at ARALLC, all soil samples are weighed on the laboratory balance to determine their final weight. The prep weight is documented by vial number in the MeOH log. This, along with the final weight, are recorded in the Solid Sample Prep Log to determine the net weight of the soil sample.

6.2.2 Soil samples are then assigned to a sample prep group. The sample prep group is identified by the QC sample prep group number. Dates, Analyst, QC and Sample ID numbers and standards are recorded in the Solid Sample Prep Log. Each sample prep group includes a QC set and up to 20 field samples received within the same week.

6.2.3 40mL vials are prepared for use as a blank, LCS and LCSD by adding 10mL MeOH and 10g blank sand to each. They are identified by MB, LCS and LCSD followed by the sample prep group number.

6.2.4 Any additional sample containers provided for duplicate analysis or matrix spiking are identified in the Solid Sample Prep Log.

6.2.5 4uL of α,α,α-trifluorotoluene surrogate solution is added to all field and QC samples with a gastight syringe.

6.2.6 For analysis by 8260 use Second Source Standards for Method 8260 spike MS (if any) and LCSs by adding 5uL of each of five stock standards:

1) Absolute Standard 502/524 High Concentration VOC Mix 1
2) Supelco 524 VOC Calibration mix
3) AccuStandard Method 8260 Additions
4) Supelco California Oxygenates mix
5) Supelco Custom mix
6) 10uL Absolute Standard 1,3,5-Trichlorobenzene

6.2.7 For VPH/GRO use the Second Source MA VPH Standard. 25uL is added to spike MS (if any) and LCSs.

6.2.8 Samples and QC are shaken for 2 minutes.

6.2.9 The samples are returned to the VOA refrigerator and are allowed to settle prior to analysis. Samples that do not settle, can be centrifuged at ~2000rpm for ~2 minutes.

6.3 Instrument Setup: NA

Title: Preparation of Solid Samples for Volatile Organic Analyses by 5035A

6.4 Analysis: NA

6.5 Sample Calculations: NA

7.0 Quality Control Requirements

7.1 Method Performance

7.1.1 See determinative method.

7.2 Laboratory Performance Quality Control and Corrective Actions

7.2.1 Trip blanks are included with all bottle orders for VOC soils. In addition to these, soil prep blanks are analyzed to monitor for contamination. Contamination above the reporting limits in a blank must be noted with the results of associated samples.

7.2.2 If the recovery of analytes in a matrix spike or LCS is outside the limits as stated in the analytical method, corrective action is taken. Corrective actions may include re-analysis and/or a description of the QC failure in the final report.

8.0 Responsibilities

8.1 The analyst uses the Quality Control Criteria outlined in this SOP to evaluate and validate the data. It is the analyst's or technician's responsibility to follow this SOP and record all data in the lab notebook. Any irregularities within the test or samples should be recorded in the lab notebook and reported to the lab manager who is responsible for reviewing the data.

9.0 Health and Safety

9.1 Normal, accepted laboratory safety practices should be followed during reagent preparation and instrument operation. However, every sample in the lab should be handled as if it is hazardous waste. All technicians shall be familiar with the Chemical Hygiene Plan (SOP QA604), and SDS location.

10.0 Pollution Prevention and Waste Management

10.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Every effort is made to

make only enough standard as will be used prior to expiration.

10.2 The EPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Waste management in this laboratory is regulated by the Safety director. As indicated in section 6.4.1.13 of the Laboratory and Sample Waste Characterization and Disposal SOP# QA-5001: Vials containing 1-10mL hexane or methanol, with methanol vials also containing up to 10g solid waste are disposed of in the waste container labeled: DOT shipping label: Waste Flammable Liquids, N.O.S.(Hexane, Methanol). For any sample not falling within this category, see Table1: Sample Waste Stream Characterization Guidance Chart, also located in SOP QA-5001.

Title: Volatile Petroleum Hydrocarbons by MADEP-VPH-04-1.1

QA Officer: _____ Date 4/29/16

Laboratory Director: _____ Date 5/2/16

Author: _____ Date 5-2-16

Analyst: _____ Date _____

By signing this SOP the analyst has read and understood the latest version of the test method, has read and understood this SOP, acknowledges receipt of this SOP, and is responsible for the implementation of this SOP.

Revision History:

Revision	Changes	Date
1	Added MADEP MCP Guidance and Method Updates for new revision of method.	7/04
2	Update MADEP MCP guidance and language corrections, Co name changed, Clarified Curve fit weighting.	7/11
3	Adjust language for equipment, add range recoveries to table	1/13
4	Update language for referencing electronic files	4/14
5	Modified SOP for running FID/PID	9/14
6	Clarifications to sections: 4.2, 5.2, 6.1.6, 6.6.2, 5.3.1, 5.3.1.1, 6.4, 7.1.2.	10/14
7	Range Calculations, Method Performance clarified	4/16

Title: **Volatile Petroleum Hydrocarbons by MADEP-VPH-04-1.1**

1. Purpose and Applicability

1.1 This purge and trap GC-PID/FID method is applicable to the determination of volatile petroleum hydrocarbons and ranges including those listed:

methyl t-butyl ether (MTBE)	ethylbenzene	C5-C8 Aliphatics
benzene	m&p-xylenes	C9-C12 Aliphatics
toluene	o-xylene	C9-C10 Aromatics
naphthalene		

1.2 This method is applicable to groundwater and other aqueous and solid samples as requested.

1.3 This method is for use only by or under the supervision of analysts experienced in the use of GC-PID/FID and in the interpretation of the resulting data. Each analyst must demonstrate the ability to generate acceptable results with this method.

2. Definitions

2.1 Calibration Standard (ICal): Standard solutions prepared from the stock standard at levels corresponding to the calibration. These are used to calibrate the instrument response with respect to analyte concentration.

2.2 Carry-over: when a sample is contaminated by analytes left in the purging device, trap or syringe by a previous sample analysis with high levels of analytes.

2.3 Continuing Calibration Verification (CCV): A standard analyzed at the beginning and end of each analytical batch, used to verify the previous calibration. The CCV is analyzed near the mid-point of the initial calibration. The concentration of the CCV may be varied in order to reveal any concentration specific biases.

2.4 Initial Calibration Verification (Instrument Performance Check) (ICV): A second source standard analyzed at a concentration near the mid-point in the calibration to verify the integrity of the calibration.

2.5 Interferences: Occurrences affecting the results of a sample analysis. Examples include: Impurities in the purge gas, solvent vapors in the lab, diffusion of volatile organic compounds through the septum seal into the sample, carry-over from samples with high levels of analytes.

2.6 Laboratory Control Samples (Laboratory Fortified Blank) (LCS & LCSD): An aliquot of reagent water or other blank matrices to which known quantities of second source standard is added. The LCS and LCSD are analyzed exactly as a sample. Their purpose is to determine whether the methodology is in control, and to monitor accuracy and precision.

2.7 Matrix Spike: An aliquot of a field sample spiked with a known amount of second source standard.

Title: **Volatile Petroleum Hydrocarbons by MADEP-VPH-04-1.1**

2.8 Prep Blank (Laboratory Reagent Blank) (Method Blank) (PB): An aliquot of reagent water or other blank matrix that is treated exactly as a sample, including exposure to all glassware, equipment, and reagents that are used with other samples. The PB is used to determine if the method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus at levels which may interfere with the analysis. The concentrations found in the PB must be below the reporting limit for each analyte.

2.9 Second Source Standard (Spiking Solution): a solution prepared from a separate source other than used for the calibration standards, containing known concentrations of method analytes.

2.10 Trip blank (field reagent blank): a VOC vial filled with reagent water (or a methanol blank for solid/soil samples) is carried through sampling and handling protocol, then analyzed to check for cross contamination.

3. Applicable Documents/References

3.1 MADEP-VPH-04-1.1
3.2 MADEP QA/QC Requirements and Performance Standards for Method VPH
3.3 ARA SOP QA-5000 Manual Integration
3.4 ARA SOP QA-5125 Preparation of Solid Samples for Volatile Organic Analyses
3.5 ARA SOP QA-400 Sample Receiving and Identification
3.6 ARA SOP QA-402 Bottle Order Preparation
3.7 ARA SOP QA-5834 Total Solids (% Dry Matter)
3.8 QA-003 ARA QA Manual

4. Materials and Apparatus

4.1 Equipment:

4.1.1 Syringes: Gastight 10uL, 25uL, 50uL, 250uL, 500uL, and 1,000uL
4.1.2 Class “A” Volumetric flasks: 5mL, 50mL & 100mL
4.1.3 VOA vials: 40mL glass with Teflon lined silicon septum
4.1.4 pH paper
4.1.5 Auto-sampler: Varian Archon
4.1.6 Purge and Trap Device: Tekmar 3000, or equivalent.
4.1.7 Trap: Supelco Purge Trap M, or equivalent
4.1.8 Gas Chromatograph: Agilent Technologies 7890A, or equivalent
4.1.9 Detectors: PID/FID OI Analytical Model 4450, or equivalent
4.1.10 Column: Restek RTX-502.2, 105 meter, 0.53mm ID, 3um df, or equivalent
4.1.11 Data system: Agilent EnviroQuant

4.2 Reagents/Standards:

4.2.1 Purified/Deionized Distilled water (reagent grade).

Title: **Volatile Petroleum Hydrocarbons by MADEP-VPH-04-1.1**

4.2.2 Surrogate Standard: 2,5-dibromotoluene purchased from Restek or other reputable supplier. The concentration is typically 10,000ug/mL, which is diluted to a final concentration of 200ug/mL and used in the Archon reservoir.

4.2.3 Surrogate Standard: α, α, α-Trifluorotoluene purchased from Restek or other reputable supplier. The concentration is typically 2500ug/mL. This Standard is added to methanol preserved solid VPH samples and associated QC.

4.2.4 Primary Standard: VPH + Surrogate mix diluted from stock in methanol. Ordered from Restek Chromatography Products or other reputable source at approximately 10,000ug/mL. The diluted standard is prepared monthly or as needed if signs of degradation are suspected. USE: Initial Calibration and Continuing Calibration Verification

4.2.5 Second source standard: VPH mix diluted from stock standards in methanol. Ordered from Absolute Standards or other reputable source at approximately 2000ug/mL (concentration of individual analytes may vary). The diluted standard is prepared monthly or as needed if signs of degradation are suspected. USE: Laboratory Control Samples, and Matrix Spikes.

4.2.6 Methanol: purge and trap grade. Source: Brand NU Labs (Cat # 232-1L) or equivalent.

4.2.7 Standards prepared in MeOH and are stored in screw cap, Teflon lined, amber vials with minimal headspace at ≤ -10 degrees C.

5. Method/Calibration/Interferences

5.1 Method Summary

5.1.1 Helium is bubbled through a 5mL water sample contained in a sparge tube at ambient temperature. The purgeable analytes are transferred from the aqueous phase to the vapor phase and are swept through a solid sorbent where the analytes are trapped. After purging is complete, the trap is heated to transfer the analytes to the GC, where the analytes are separated and measured by PID then FID.

5.2 Sample Handling and Preservation

5.2.1 See SOP QA400 for additional information regarding sample handling. Water samples are collected in duplicate in 40mL VOA vials with no headspace. The vials have Teflon coated septa and are pre-preserved with HCl. Solid samples are collected in 40 mL VOA vials that are pre-filled with 10mL of purge & trap grade (or equivalent) methanol. Approximately 10 g of solid sample is added to the methanol, to achieve a ratio of 1:1 ±25%. For proper preservation, the soil/sediment in the vial must be completely covered by the methanol. Additional sample is collected for dry

weight determination. All samples must be placed on ice/cooled during storage and transportation and are refrigerated at above freezing to 6 degrees C upon receipt.

5.2.2 Preserved water samples must be analyzed within 14 days of sampling. Solid samples must be analyzed within 28 days of sampling.

5.2.3 Shipment of samples must follow the requirements outlined in the sample receipt SOP QA400. See the Bottle Order Preparation SOP 400 (6.0) for information on Trip Blank preparation.

5.3 Calibration Procedure

5.3.1 Initial Calibration: Standards are analyzed at several concentrations in order to describe the calibration range. Analytical results must be bracketed by calibration standards. A typical VPH calibration includes points at 1, 5, 25, 100 & 250 ug/L of each component. The range concentrations are multiples of these, based on the number of components eluting in each range. e.g. the C5-C8 range concentrations are 3, 15, 75, 300 & 750, since pentane, 2-methylpentane and 2,2,4-trimethylpentane elute in this range. Note that the reporting limit is Method defined as "100x the concentration of the lowest calibration standard for the associated analyte", i.e. 100 ug/L. The VPH primary standard is diluted to the appropriate concentration using micro liter syringes and class A volumetric glassware. Chromatograms are evaluated for proper integration of target and range analytes. Aliphatic and aromatic ranges are integrated in the same manner as samples, where the collective area of all peaks in the range are integrated from the beginning and end points as defined in the method, e.g. C5-C8 begins just before the elution of pentane and ends just before the elution of nonane.

Hydrocarbon Range	Beginning Marker	Ending Marker
C5-C8 Aliphatic Hydrocarbons (FID)	before n-Pentane	before n-Nonane
C9-C12 Aliphatic Hydrocarbons (FID)	before n-Nonane	before Naphthalene
C9-C10 Aromatic Hydrocarbons (PID)	after o-Xylene	before Naphthalene

The EnviroQuant software is used to construct a multi-point calibration for target analytes. Two fit types are available: average response factor, linear regression. Quadratic fit is used only in rare exceptions and if used will be noted in the final report. The analyst's discretion is used in selecting the fit and weighting which best represents the data at the time of calibration. Particular attention is paid to the low end of the calibration range. An appropriate fit is chosen when the calibration is constructed. It is not changed during the time the calibration is in use.

Title: **Volatile Petroleum Hydrocarbons by MADEP-VPH-04-1.1**

5.3.1.1 . For range analytes, linear regression is used. The fit that best matches each target analyte is chosen based on the following criteria:

5.3.1.1.1. If average response factor is used, RSD is less than or equal to 25%.

5.3.1.1.2. If a linear fit is used, the correlation coefficient (r^2) is greater than or equal to 0.99.

5.3.1.2 The calibration of hydrocarbon ranges is determined in a locked Excel spreadsheet. An Excel spreadsheet is used for determination of ranges, because EnviroQuant does not have the ability to calculate the ranges (as defined in the method). Peak areas for analytes that are not included in the range calibration are subtracted from the total response. The range analytes are quantitated using linear regression. The upper end of the calibration range for the VPH ranges is limited by the abundance (peak height) of the largest peak used in the calibration of the specific aliphatic and aromatic range in the highest calibration standard, not the concentration of the highest concentration in the calibration standard. During Curve evaluation the Excel spreadsheet is to be referenced, to verify the correlation coefficient meets acceptance criteria.

5.3.2 To verify the reporting limits, a point in the calibration that falls at or below the reporting limit is then re-quantitated for each reported analyte, using the newly prepared calibration. The concentration of each reported analyte must agree within 40% of its expected value to be acceptable.

5.3.3 Initial Calibration Verification (ICV): After the calibration is constructed, an initial calibration verification is run, to ensure that the calibration is valid. The ICV is prepared in the same manner as the LCS (LCS may be used), at a concentration near the middle of the calibration range. The concentration may be varied periodically in order to reveal any concentration specific biases. See Section 7.2.1 for acceptance criteria.

5.4 Interferences

5.4.1 Samples can become contaminated by diffusion of volatile organics through the sample container septum during shipment and storage or by dissolution into the methanol used for preservation.

5.4.2 Carry-over can occur whenever a low-concentration sample is analyzed immediately after a high-concentration sample. Whenever a highly concentrated sample is anticipated, following it with a blank is advised to minimize carry-over.

5.4.3 Presence of non-target compounds may interfere with the analysis of the samples, and may cause dilution to be necessary.

5.4.4 Presence of surfactants can cause the sample to foam during the purging step. Foaming into the purge and trap apparatus necessitates significant instrument repairs and must be avoided. Samples suspected to foam must be watched during the purging step. The purging process is aborted if the

sample begins to foam over. Foaming samples may require analysis at a dilution. Refer to sample screening in the following section (6.2.1).

6. Procedure

6.1 Procedural Qualifiers

6.1.1 Surrogate Standards: The Archon auto-sampler adds 1uL of the SS solution (dibromotoluene) to all samples, blanks and standards.

6.1.2 Continuing Calibration Verification: A CCV is analyzed to validate that the current calibration is valid. When the criteria are met, as discussed in 7.2.2, the analysis of samples may proceed. After the analysis of a batch of 20 samples or less, another bracketing CCV is run, with the same acceptance criteria as the opening CCV. Refer to the spreadsheet "QCCHECKS VPH.XLS" for evaluation of the CCV. A sample of the worksheet is included as Table 4.

6.1.3 Method Blanks: To insure that the system is free from carryover and there is no contamination in any reagents, a blank sample is analyzed with every batch. There must be no contamination at or above the reporting limit for all compounds reported by this method. Refer to Table 2 for analytical reporting limits.

6.1.4 Lab Control Sample: An LCS is analyzed in each batch of 20 samples or less. The LCS is prepared by fortifying a reagent water blank (or a methanol blank for solid/soil samples) with the matrix spiking solution containing all components in the standard. The spike recovery for targets and ranges must acceptable. See QC section 7.2.4 for criteria. If the acceptance criteria are not met, the situation must be investigated, corrected, and affected samples must be reanalyzed if necessary. In the event that samples are reported associated with out of control QC, this information is discussed in the final report. The LCS may be evaluated as an ending CCV, according to the CCV acceptance criteria. Nonane may exceed this limit as described in the VPH method 10.4.2.3.

6.1.5 LCS Duplicates: An LCS duplicate is prepared in the same manner as the LCS and run with each batch. Precision data are obtained by comparison of results of individual concentrations (not percent recoveries) of the LCS & LCSD. If the RPD of the replicates is outside the acceptance criteria, this situation is investigated and corrected. The impact to the batch must be assessed and if necessary reanalysis of affected samples performed. Any exceedances are discussed in the final report.

6.1.6 Reporting limit check standard: As part of each initial calibration and at least monthly, a standard at or below the reporting must be analyzed. The percent recovery of the reported analytes must be ± 40% or recalibration is necessary.

Title: **Volatile Petroleum Hydrocarbons by MADEP-VPH-04-1.1**

6.1.7 Matrix spikes/Matrix Spike duplicates, if requested by the customer, are performed by adding a known concentration of standard to a sample. The final concentration (including sample and spike) should be near the middle of the calibration range. The native concentrations in the sample are subtracted and the full target list is evaluated for acceptance. See QC section 7.2.5 for acceptance criteria.

6.1.8 Preservation Check: After the analysis is complete, the remaining sample in the VOA vials is checked for adequate preservation using pH paper. The result is recorded in the instrument run log. Any sample with a pH above 2 is flagged in the final report.

6.2 Sample Preparation

6.2.1 Refer to the VOC Solid sample preparation SOP QA5125.

6.2.2 Screening

6.2.2.1 Historical data can be reviewed to help determine if dilution of a sample is necessary. When high levels of contamination are suspected, it is recommended that an initial diluted run be performed. A second analysis can then be done at an appropriate dilution.

6.3 Instrument Setup

6.3.1 Loading Samples and Standards

6.3.1.1 The purge and trap device is automatically cleaned after each analysis, so manual cleaning is not necessary. The SS solution is added by the auto-sampler. All standards, blanks and samples must be run in 40mL VOA vials. Standards are prepared in 50 or 100mL volumetric flasks and transferred into VOA vials. Solid samples are prepared in 50mL volumetric flasks by adding 1mL of methanol extract to water, bringing to volume (for undiluted analysis), and transferring to VOA vial. Vials are loaded into the tray and the sequence for analysis is entered using the keypad on the Archon auto-sampler. Refer to the manufacturers instructions for detailed directions. 5mL of sample is used for undiluted analysis.

6.3.2 Setting up sequence

6.3.2.1 To proceed, there must be at least 100 MB space available on the "C" drive where data are acquired. The sequence, which contains the sample information, is entered in the EnviroQuant software. EnviroQuant is located on the computer used for data acquisition. To create a sequence, "Sequence Menu" is selected, then "Edit Sequence Table". Sequence information includes filename, acquisition method, auto-sampler position, sample name and volume/dilution. The sequence is saved using the date the sequence starts. Begin analysis.

6.4 Sample Analysis

6.4.1 When CCV (or initial calibration) and blank are acceptable, analysis of samples may proceed. See Table 5 for typical sequence of QC and samples.

6.4.2 If concentrations found in field samples exceed the calibration range, reanalysis at a dilution is necessary. The top of the calibration range is defined for *target analytes* as the highest concentration used in the initial calibration i.e. 250ug/L for most compounds. The upper limit for *range analytes* is defined as the maximum response (peak height) observed for a range analyte component e.g. decane from the initial calibration from the highest concentration calibration standard. This peak height limit is determined separately for the PID and FID detectors.

6.4.3 Using an uncompromised vial for water samples or remaining methanol from a soil sample extract, an appropriate dilution is prepared using a gas-tight microliter syringe and 50mL class A volumetric flask. This diluted sample is transferred to a pre-cleaned 40mL volumetric flask with no headspace. Alternatively, the Archon auto-sampler can be programmed to perform dilutions in a range from x5 to x200. All dilutions are prepared using VOA-free DI water. Dilutions are performed such that the response for major constituents is in the upper half of the calibration range.

6.4.4 When the analysis of a highly contaminated sample is anticipated, it should be followed by a blank to ensure there is no carryover to the following sample analysis.

6.5 Data Analysis

6.5.1 After a sample analysis is complete, the data are evaluated by a qualified analyst experienced with GC-PID/FID data evaluation and EnviroQuant software.

6.5.2 Data files are quantitated using the appropriate data analysis method prepared from the most recent initial calibration. Using the Qedit function, each analyte is reviewed for qualitative and quantitative accuracy using retention time, integration, and comparison of PID and FID signals. Sample range analytes are integrated in the same manner as standards (see sec 5.3.1.2). Analyte concentrations are determined in Enviroquant and range concentrations are determined in a locked Excel spreadsheet. Surrogate recoveries are verified to be acceptable. In the case of surrogate failure, the sample must be reanalyzed, unless there are assignable reasons for failure such as interference from coeluting peaks or high moisture content in the sample. The sample may be reported if the surrogate fails high and the associated targets and ranges are non-detect. A quant report is printed for each sample, reviewed and marked with initials and date.

6.6 Sample Calculations

6.6.1 Average Response Factor calculations

As an external standard method, response factors are determined by:

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$$RF = \frac{(AC)}{(CC)}$$

where,
AC = Area of compound of interest
CC = Concentration of analyte of interest
RF = Response Factor

When average response factor is used, the RF is determined for each calibration level. The response factors are averaged. This average is used to determine the sample concentration:

$$CC = \frac{(AC)}{(avgRF)}$$

where,
AC = Area of compound of interest
CC = Concentration of analyte of interest
avgRF = average relative Response Factor

6.6.2 **Linear Regression calculations**
More commonly, linear regression is used. Using Enviroquant and Excel, linear regression by the method of least squares, a slope (m) and intercept (b) are determined which fit the data. The regression algorithm does not force the origin.

$AC = m * CC + b$

Once the slope and intercept are established, and solving for CC, the following equation is used to calculate the concentration (CC):

$CC = (AC – b) / m$

6.6.3 Sample results

The quantitation report shows results in ug/L units, which for water samples (when multiplied by a dilution factor) can be reported directly.

For solid samples, sample preparation amounts are included in the calculations. Unless otherwise instructed by the customer and noted in the report, soil samples are reported on a dry weight basis. See SOP QA5834 sec 6.2 for the procedure to determine percent moisture in a solid sample.

Concentration (ug/g) = AF*(0.005*(EV+SW*(1-DF)))/(VA*SW*DF)

Where: AF=Amount found (ug/L) – Quant report Concentration

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EV=extract volume (mL) – usually 10mL
SW=soil weight (g)
DF=Dry Fraction (unit-less)
VA=Volume Methanol Analyzed (mL) – usually 0.1mL

Note: For the C5-C8 range, the FID peak area of α,α,α-trifluorotoluene is subtracted from the total C5-C8 area used in the calculation.

7. Quality Control Requirements

7.1 Method Performance

7.1.1 Initial Demonstration of Performance: Precision and accuracy for the use of this method by the laboratory and analyst are demonstrated by the analysis of 7 LCS samples at concentrations near 50% of the highest standard in the calibration. Accuracy limits are ±30% of the true value and the precision limit is 25% RSD. An initial demonstration is performed for each analyst and after any significant change in instrumentation.

7.1.2 Method Detection Limit: The MDLs may be established for all analytes if required by regulatory standards. They are not required by the MA VPH method.

7.1.2.1 To Determine an MDL, reagent water is fortified at a concentration of two to three times the estimated method detection limit. A minimum of seven replicates of the fortified reagent water are processed through entire analytical system. Calculate the MDL for each analyte as follows:

MDL = (t)*(s)

t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. For seven replicates t = 3.14.
s = standard deviation of the replicate analyses.

7.1.2.2 MDLs should be determined initially or whenever there is a significant change in the instrumental configuration (if required). The MDL data are stored electronically, by method, in the QA/MDL file folder. See QAM section 11.5 for additional information regarding MDLs and RLs.

7.1.3 LOD/LOQ: Refer to QAM Analytical Procedures section, for specific LOD/LOQ requirements. LOD/LOQ data is stored electronically in the QA/LOD&LOQ file folder.

7.1.4 IDC/CDC: Proficiency of the analyst is ensured by documentation of an Initial or Continuing Demonstration of Capability (IDC or CDC). See

section 7.1.1 for procedure.

7.1.5 Control Charts: Control Charts are quality control tools which graphically display QC data over time. The data required for generation of accuracy and precision control charts is maintained by the laboratory. See QAM for additional information.

7.1.6 Inter-laboratory performance: Proficiency Test studies, conducted by approved providers, constitute the majority of inter-laboratory performance comparisons. Proficiency tests are performed at a frequency as required by method, regulation, or accrediting body. See QAM for additional information.

7.2 Laboratory Performance Quality Control and Corrective Actions

7.2.1 **Initial Calibration Verification**: The ICV must analyzed after an initial calibration, be from a second source and be within ±30% of the expected concentration.

7.2.2 **Continuing Calibration Verification:** The CCV must be within ± 25% of the expected concentration for all individual analytes and reported ranges. Nonane may exceed this limit as described in the VPH method 10.4.2.1. If the CCV performed at the beginning of a run is unacceptable, the CCV should be remade and reanalyzed, if acceptable, the batch can be continued, otherwise a new initial calibration is required. The LCS may be used as the end CCV if it meets the CCV acceptance criteria.

7.2.3 **Method Blank:** The PB is analyzed after the CCV. Contamination in the PB must be lower than the Reporting Limit. If sample results show presence of a contaminant, the source of the contamination must be found and eliminated. The impact to any results must be evaluated. If necessary, samples are reanalyzed. If the problem cannot be resolved, an analyte found in the sample where the associated PB is contaminated with the same analyte must be flagged in the report. Blank results are not subtracted from sample results.

7.2.4 **Laboratory Control Sample/LCS Duplicate (LCS/LCSD):** An LCS/LCSD pair are analyzed in every batch of 20 samples or less. The percent recovery is calculated for the reported individual analytes and the aliphatic and aromatic hydrocarbon ranges these must be within the limits 70-130%. The LCSD RPD limits are 25%. LCS failures indicate method problems and all samples associated with a failing LCS must be considered suspect. Samples associated with failed LCSs are reanalyzed, or if no further sample is available are flagged in the final report. If the LCS is evaluated as an end CCV, the recovery must be 75-125%.

7.2.5 **Matrix Spike (&MSD):** A matrix spike is analyzed upon customer request. A known amount of analyte is added to an aliquot of a randomly chosen sample. The calculation of the percent recovery is as follows:

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$$R = (Cs - C)/s * 100$$

R = percent recovery
Cs = Fortified sample concentration
C = Sample background concentration
s = Concentration equivalent of analyte added to the sample

If the sample matrix spike fails the criteria 70-130%, the LCS results are reviewed to determine whether the failure is matrix specific or an analytical problem. If found to be matrix specific, the report for the spiked sample is noted with this failure. If it is an analytical problem, the samples would be rerun, given there is ample volume that remains, otherwise the report would be noted. If the RPD between replicates is greater than 50%, this is noted in the final report.

7.2.6 **Surrogate recoveries:** Surrogates are added to all samples and standards at the time of analysis. Recovery of surrogates must meet criteria of 70-130% (see Table 1). Samples failing these criteria are reanalyzed, unless the failure can be attributed to the obvious interference such as hydrocarbons, % moisture of the solid/sediment sample is >25% and surrogate recovery is >10%. If the reanalysis shows acceptable recoveries, these results are reported. Any failures are noted in the report.

7.2.7 **Corrective Actions:** The QA Manual is to be referenced for further guidance on corrective actions.

8. Retention Times

8.1 The retention times in the analytical system should be stable such that peaks can be accurately identified. The width of the retention time window used to make identifications can be based upon the measurements of actual retention times variations of standards over the course of several days. These retention times and windows are estimates. Three times the standard deviation of a retention time can be used to calculate a suggested window size for each analyte. As a default, 0.3 minutes is used. The retention time windows for the VPH ranges are established as per Section 9.3 of the MADEP VPH method. The experience of the analyst should weigh heavily in the qualitative interpretation of chromatograms.

9. Responsibilities

9.1 The analyst uses the Quality Control Criteria outlined in this SOP to evaluate and validate the data. It is the analyst's or technician's responsibility to follow this SOP and record all the data in the lab notebook. Any irregularities within the test

or samples should be recorded in the lab notebook and reported to the lab manager who is responsible for reviewing the data.

10. Health and Safety

10.1 Normal accepted laboratory safety practices should be followed during reagent preparation and instrument operation. Many of the analytes in this method are toxic or are known or suspected carcinogens. Every sample in the lab should be handled as if it is hazardous waste. All technicians shall be familiar with the Chemical Hygiene Plan (SOP QA604), and SDS location.

11. Pollution Prevention and Waste Management

11.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Every effort is made to make only enough standard as will be used prior to expiration.

11.2 The EPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Waste management is regulated by the Safety director. Expired standards in methanol are dispose in the flammables waste stream. Sample waste is neutralized and is disposed into the municipal waste system.

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Table 1 - Surrogate Compound Recovery Criteria

SURROGATE STANDARDS	Acceptance Limits
2,5-dibromotoluene as aliphatic (FID)	70-130
2,5-dibromotoluene as aromatic (PID)	70-130
ααα-trifluorotoluene (PID- Solids only)	70-130

Table 2—Analyte Reporting Limits

Compound	ug/L	ug/g
MTBE	2	0.1
benzene	1	0.1
toluene	2	0.1
ethylbenzene	2	0.1
m&p-xylenes	2	0.1
o-xylene	2	0.1
naphthalene	5	0.2
C5-C8 Aliphatics	100	5
C9-C12 Aliphatics	100	5
C9-C10 Aromatics	100	5

Table 3 -Analytical Conditions

Purge and Trap

Purge Gas	Helium
Sorbent Trap	BTEXTRAP M
Line Temp.	150° C
Valve Temp.	150° C
Purge Time	11 minutes
Purge Flow	40ml/min
Desorb Preheat	245° C
Desorb Temp.	250°C
Desorb Time	2 minutes
Bake Time	6 minutes
Bake Temp.	270° C

Title: **Volatile Petroleum Hydrocarbons by MADEP-VPH-04-1.1**

Instrument Control Parameters

Sample Inlet:	GC
Injection Source:	Manual
Injection Location:	Front SS

HP7890A Temperature Parameters

Zone Temperatures:	**State**	**Set point**
Inlet A:	On	150° C
Inlet B:	Off	Off
Detector A:	On	225° C
Detector B:	On	225° C

Inlet Parameters:

Mode:	Split (2:1)
He Flow:	constant flow (10mL/min)

Oven Parameters:

Oven Equib Time:	0.10 minutes
Oven Max:	270° C
Oven State:	On

Oven Program:

Initial Temperature:	35° C
Initial Time:	2.00 min.

Ramps:

Level	Rate	Final Temperature	Final Time (minutes)
1	20	135° C	0.00
2	5	180° C	0.00
3	45	220° C	8.00

Run Time: 24.89 min.

Column:

Restek Rtx-502.2
Length: 105 m
Diameter: 0.53 mm ID
Film thickness: 3 um df
Max. Temp. 270° C
Cat. #: 10910

Detector Parameters

PID/PID Conditions

H_2 flow:	30mL/min
Air flow:	170mL/min
Makeup flow:	20mL/min (He)
PID signal:	front
FID signal:	rear

Title: **Volatile Petroleum Hydrocarbons by MADEP-VPH-04-1.1**

Table 4
VPH CCV Summary Report

VPH Continuing Calibration Verification

File	X:\VOA06\2014\Sep14\093014\V6093005.D
Sample	**ccv 100ppb 5mL**
Operator	lmm
Aq Date/Time	09/30/14
Misc	5
Aq Method	V6VPH7.M
Quant Method	MA VPH

Standard Concentration		100
VPH Recovery Limits	75%	125%

	Compound	Instrument DLs	Amount Found	% Recovery
19	SS 2,5-dibromotoluene		144.25	103.0%
39	SS 2,5-dibromotoluene #2		143.14	102.2%
3	methyl-t-butyl ether (MTBE)	1	95.11	95.1%
5	benzene	1	104.46	104.5%
6	toluene	1	104.24	104.2%
10	ethylbenzene	1	104.27	104.3%
11	m&p-xylenes	1	204.15	102.1%
12	o-xylene	1	104.33	104.3%
18	naphthalene	1	103.43	103.4%
7	C5-C8 Aliphatics	100	316.03	105.3%
16	C9-C12 Aliphatics	100	273.84	91.3%
17	C9-C10 Aromatics	100	106.30	106.3%
21	pentane #2	*1*	*105.42*	105.4%
22	2-methylpentane #2	*1*	*106.10*	106.1%
24	2,2,4-trimethylpentane #2	*1*	*103.12*	103.1%
29	nonane #2	*1*	*89.16*	89.2%
33	decane #2	*1*	*91.39*	91.4%
14	1,2,4-trimethylbenzene	*1*	*104.47*	104.5%
35	n-butylcyclohexane #2	*1*	*89.93*	89.9%

Title: **Volatile Petroleum Hydrocarbons by MADEP-VPH-04-1.1**

Table 5-Typical Analytical Sequence with Acceptance Criteria

Sample Number	Instrument Run Log	Acceptance Limit
1	CCV (or ICAL)	±25%
2	ICB	BRL
3	Sample 1	
4	Sample 2	
5	Sample 3	
6	Sample 4	
7	Sample 5	
8	Sample 6	
9	Sample 7	
10	Sample 8	
11	Sample 9	
12	Sample 10	
13	LCS*	± 30%
14	Sample 11	
15	Sample 12	
16	Sample 13	
17	Sample 14	
18	Sample 15	
19	Sample 16	
20	Sample 17	
21	Sample 18	
22	Sample 19	
23	Sample 20	
24	Sample 20 MS (upon request)	± 30%
25	Sample 20 MSD (upon request)	± 30%, RPD ± 50%
26	LCSD	± 30%, RPD ± 25%
27	CCV	± 25%
28	CCB	BRL
29	Sample 1	

*LCS may be run after an initial calibration as a verification or at the end of the batch.

ABSOLUTE RESOURCE ASSOCIATES
Standard Operating Procedure QA-5303

SOP Number: QA-5303
Revision History
Cover Page

Title: Analysis of Polychlorinated Biphenyls in Soil and Water Extracts by EPA 8082A

QA Officer: _____ Date: 8/2/17

Laboratory Director: _____ Date: 8/2/17

Author: _____ Date: 8/3/17

Analyst: _____ Date: _____

By signing this SOP, the analyst has read and understood the latest version of the test method; has read and understood this SOP; acknowledges receipt of this SOP; and is responsible for the implementation of this SOP.

Revision History:

Revision	Changes	Date
4	Add in MADEP Guidance on QA/QCUpdate to current practice w/ 8000 series methods	7/03
5	Update with final MADEP QA/QC and add congener analysis	8/04
6	Change title of table to Reporting Limits	8/05
7	Added method 3546	1/07
8	Fixed typos and made clarifications	11/07
9	Added aqueous prep instead of referencing another SOP	2/08
10	Removed low-level analysis, added microwave operating parameters	6/08
11	Made changes to acid clean up procedure. Added info about conformational column	1/09
12	Added prep. proc. for wipes and product. Updated instrument acquisition method. Copper clean-up. Analyze midpoint standards for remaining aroclors. DoD Language	12/10
13	Added requirement of reporting results from dual columns if a disparity between the two columns (RPD >40%) occurs, changed name, clarified Curve fit weighting, and holding times	8/11
14	Update QC recoveries for MCP projects, update calibration point concentrations, general edits	1/13

Title: Analysis of PCB in Soil and Water Extracts by EPA 8082A

15	Clarified waste dilution prep, maintenance procedures, Updated method from 8082 to 8082A, MDL location,	7/13
16	Add blank sand prep for QC; changed BDL to BRL; MDL, LOQ location	4/14
17	Clarified calibration concentrations, referenced necessary clean up procedures, revised microwave program settings, added method performance to 7.1	3/16
18	Water reporting limit change. Clarified primary extraction method for solid PCBs in section 6.3, revised acid clean up information in section 6.6.1; Added table with typical GC instrument conditions; added Aroclors 1262 and 1268 to Applicability section; updated current Acquisition method	2/17
19	Clarify DOD requirement: report positive confirmed results from primary column; updated CCV section to refer to QAM for additional guidance	7/17

Title: Analysis of PCB in Soil and Water Extracts by EPA 8082A

Polychlorinated Biphenyls- EPA 8082A

1.0 Purpose and Applicability

1.1 This procedure details the steps to be followed for the analysis of soil, water, and product extracts for PCBs. It is applicable to the following mixtures: Aroclors 1242, 1254, 1221, 1232, 1248, 1260, 1016, and 1262 and 1268 when requested. It is also applicable to congeners IUPAC numbers 1,5,18,31,44,52,66,87,101,110,138,141,151,153,170,180,183,187,206 (congeners not listed may also be appropriate for this method). Extracts are analyzed by Gas Chromatography-Electron Capture Detector (GC-ECD) or Gas Chromatography-Electrolytic Conductivity Detector (GC-ELCD).

1.2 This method is recommended for use only by or under the supervision of experienced analysts in the use of a gas chromatograph and the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method as described in the procedure in section 7.0.

2.0 Definitions

2.1 Continuing Calibration Blank (CCB): A volume of extraction solvent fortified with the surrogate standard. A CCB is run at the beginning of a run when the calibration curve in place is being verified, at the start of a batch, after every ten samples, and as a closing bracket. No CCB is run at the start of a batch if a new calibration curve is built on that day, in this case, an ICB would be analyzed.

2.2 Initial Calibration Verification (Instrument Performance Check) (ICV): A standard at a mid-point in the calibration curve to verify the integrity of the curve. The ICV is made from a second source standard. All analytes must have a different lot number than the one that made the calibration curve. The percent recovery for the ICV is ± 15%.

2.3 Initial Calibration Blank (ICB): A volume of extraction solvent fortified with the surrogate standard. The ICB is analyzed immediately following the instrument calibration.

2.4 Continuing Calibration Verification (CCV): A CCV is analyzed every ten samples, at the start of a batch (if verifying a previous curve), and at the end of every batch. A CCV is a mid-point on the calibration curve. The concentration of the CCV should be varied during a run. The percent recovery for the CCV is ± 15%.

2.5 Calibration Standard (ICal): Standard solutions prepared from the stock standard at levels corresponding to the calibration curve. These are used to calibrate the instrument response with respect to analyte concentration.

2.6 Field Duplicates: A duplicate sample taken in the field. These are only done at the customer's request.

2.7 Laboratory Control Sample (Laboratory Fortified Blank) (LCS): An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The spiking mixture source is one different from the calibration standards. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The percent recoveries for analytes in the LCS are 40-140%. For projects requiring DoD protocol, refer to the DoD Quality Systems Manual for Environmental Laboratories for required LCS/D acceptance limits.

2.8 Laboratory Control Sample Duplicate (LCSD): See definition above.

2.9 Prep Blank (Laboratory Reagent Blank) (PB): An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, and reagents that are used with other samples. The PB is used to determine if the method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. The PB must be below the reporting limit for each analyte. For projects requiring DoD protocol, refer to the QAM for additional evaluation criterion for method blanks.

2.10 Linear Calibration Range (LCR): The concentration range over which the instrument response is linear and calibrated for.

2.11 Stock Standard Solution (SSS): A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source (e.g. Fisher Scientific or Ultra Scientific). Documentation of the purity of the SSS must be retained for traceability purposes in the organic standards binder.

2.12 Matrix Spike: An aliquot of a sample spiked with a known amount of spiking solution. Matrix spikes are only done when requested by the customer.

2.13 Matrix Spike Duplicate: See definition above. Matrix spike duplicates are only done when requested by the customer.

2.14 Surrogates: Organic compounds which are similar to analytes of interest in chemical composition, extraction, and chromatography, but are not normally found in environmental samples. These compounds are spiked into all blanks, standards, and samples prior to analysis. Percent recoveries are calculated for each surrogate.

3.0 Applicable Documents/References

3.1 SW846 Revision 1, Feb 2007, Method 8082A

3.2 40 CFR Part 136.1

3.3 Organic Standards Preparation Logbook

3.4 GC Instrumentation Run Logbook

3.5 Test Methods for Evaluating Solid Waste Physical/Chemical, US EPA SW 846, Final Update: Method 8082A

3.6 ARA SOP QA-400 Sample Receiving and Identification
3.7 ARA SOP 5000 Manual Integration
3.8 MADEP MCP QA/QC Requirements Method 8082A, July 2010
3.9 ARA SOP 5305 Soxhlet Extraction
3.10 ARA SOP QA-5524 Liquid Liquid Extraction
3.11 ARA SOP QA-5522 Microwave Assisted Extraction
3.12 Methods, USEPA SW 846, Method 3546
3.13 Test Methods for Evaluating Solid Waste Physical/Chemical, US EPA SW 846, Final Update: Method 3580A Waste Dilution
3.14 Test Methods for Evaluating Solid Waste Physical/Chemical, US EPA SW 846, Final Update: Method 3665A Sulfuric Acid Cleanup
3.15 Instrument Manufacturer's Manuals
3.16 ARA QA Manual
3.17 Current DoD Quality Systems Manual for Environmental Laboratories

4.0 Materials and Apparatus

4.1 Equipment:

4.1.1 Class A volumetric flasks as required.
4.1.2 Syringes as required for dilutions.
4.1.3 HP vials for standards and samples.
4.1.4 HP GC 5890 with column, ECD and HP data system, or equivalent.
4.1.5 Analytical balance for weighing standards, solids, and products.
4.1.6 Refrigerator for storage of standards and extracts.
4.1.7 Suggested column for ECD: CLPII 30M x 0.53mm id wide-bore connected to a guard column with a y-connector
4.1.8 Suggested confirmation column for ECD: CLPI 30M x 0.53mm id wide-bore connected to a guard column with a y-connector
4.1.9 Labconco Rapid-Vap
4.1.10 MarsXpress Microwave vessels
4.1.11 Microwave Accelerated Reaction System, CEM MARS model.

4.2 Reagents/Standards:

4.2.1 Hexane - Pesticide grade or equivalent
4.2.2 Acetone - Pesticide grade or equivalent
4.2.3 1:1 Hexane/Acetone mixture prepared daily
4.2.4 Dichloromethane - Pesticide grade or equivalent
4.2.5 Individual standards of Aroclors 1016,1221,1242,1232, 1248,1254, and 1260 (and 1262 and 1268 if requested) at concentrations of 100 to 1000 ug/mL supplied by ChemServ or other commercial retailers.
4.2.6 Surrogate standard containing tetrachlorometaxylene (TCMX) and decachlorobiphenyl (DCBP). (A surrogate containing only TCMX is used for

congener analysis).

4.2.7 PCB congeners (ACOE list)

4.2.8 Sand for blanks: Used as an analyte-free, solid matrix for QC. Sand is purchased from a hardware/home improvement store and must be cleaned before use. Place several cups of sand in a fine mesh bag. Rinse with lab grade water to remove fine particles. Bake in a foil loaf pan in the muffle furnace at 500 degrees Celsius for two hours or until the sand is dry. Allow to cool completely. Assign an Organic Standards Prep number

5.0 Method/Calibration/Interferences

5.1 Method Summary: Solid samples are collected in 4oz glass jars and water samples are collected in 1L Glass containers. Both must be refrigerated or iced at 0-6ºC from collection to extraction times. Samples must be extracted within one (1) year from the collection date. Sample analysis must occur within 40 days of the extraction date. Shipment of samples must follow the requirement of method. Refer to SOP QA-400.

5.2 See Table 2 for typical Reporting Limits.

5.3 The extracts are set up on an auto sampler for the GC. The method S3ZEBA (or the most recent acquisition method) is loaded into the software. This has all of the temperature setting requirements programmed in for analysis. The information is logged into the computer software sample table and into the GC-ECD run log. Once the GC run is complete the information is integrated and the data are analyzed and calculated.

5.4 Calibration Procedure: PCB working standards are prepared by diluting stock standard Aroclors into hexane at 5ug/mL each. Unless the sample history is known, generally the Aroclors 1016 and 1260 are prepared at a minimum of five points in suggested calibration concentrations of 4, 2, 1, 0.5, 0.1, and 0.05. This mix provides the analyst with nearly the entire range of individual congeners present in all of the Aroclor mixes. The analyst's discretion is used in selecting the curve fit and weighting which best represents the data, paying particular attention to the low end of the calibration range. Usually, the inverse of concentration is most appropriate weighting. If linear fit is to be used to create the curve, the plot of area versus concentration must achieve an R^2 value of 0.995 or greater. If this approach fails to be adequate, a quadratic fit with a minimum of 6 points may be used if the curve is not too visually skewed and the R^2 value is 0.995 or greater. If quadratic line fit is to be used, all integrations in all six levels of the curve for that particular aroclor should be checked for errors. Whenever considerable instrument maintenance is performed, all remaining aroclors that do not have a five-point curve should be analyzed at a midpoint concentration for chromatographic comparison purposes. In the event that an Aroclor other than 1016 or 1260 is found in the sample, the analyst performs a

single point calibration for the Aroclor found for pattern recognition purposes. This single point is analyzed generally within 12 hours of the sample analysis. If quadratic curve fit is employed for the 1016 and 1260 calibration curve, any other Aroclors detected in the samples would need to be evaluated from a six point calibration for that Aroclor. If a six point curve already exists for this Aroclor, the single point standard will act as a CCV to verify the curve. A calibration blank containing the surrogates TCMX and DCBP is prepared at a concentration equal to the expected concentration of the samples. This standard will be run immediately following the calibration curve (ICB).

5.5 Calibration Verification Requirements: The calibration curve must have an R^2 value (calibration coefficient) of 0.995 or greater. An ICV and an ICB must be run immediately following the calibration curve. The ICV must be recovered at ± 15% the expected value. The ICB must be below the reporting limit for each analyte.

5.6 Interferences

5.6.1 Contaminants in glassware, reagents, or solvents can cause elevated baselines or peaks not associated with analytes of interest in gas chromatograms. Glassware must be rinsed with the last solvent used, detergent washed in hot water, followed by tap water and distilled water rinses. After drain drying, the glassware is rinsed with pesticide grade hexane or acetone. Reagent contamination can be avoided by flushing the syringe prior to use at least three times using pesticide grade solvent.

5.6.2 Phthalate peaks appearing in chromatograms can cause a quantitative error or misidentification of a target analyte which has the same retention time as the phthalate. Avoid handling plastics during the extraction process to minimize phthalate cross contamination of glassware.

5.6.3 Elemental Sulfur peaks, especially at high concentrations, pose similar problems as phthalates in chromatograms. Elemental copper is added to extracts to remove the sulfur. Refer to the Method 3660B for the sulfur cleanup procedure.

5.6.4 Sulfuric acid clean-up is routinely performed on extracts. For details on the acid clean up procedure refer to section 6.6. Florisil clean up can be performed if extracts react poorly during acid cleaning. For details on the Florisil clean-up procedure refer to QA-5304 section 6.4.

5.7 Instrument Maintenance

5.7.1 Preventative maintenance shall be provided for all instruments and equipment as specified by the manufacturer, instrument manual or as established by the Technical Director or QA Officer. Preventive maintenance shall be conducted in order to assure timely, accurate and reproducible analytical processes in a safe laboratory.

5.7.1.1 Preventative or routine maintenance includes:
- 5.7.1.1.1 Changing injection port septa
- 5.7.1.1.2 Changing injection liners
- 5.7.1.1.3 Column clipping
- 5.7.1.1.4 Autosampler syringe replacement

5.7.2 Instrument repair may be necessary when troubleshooting identifies that a component of the instrument has failed or is functioning improperly. The following resources may be utilized for identifying failed components and making repairs:
- 5.7.2.1 Instrument Manufacturer's Manuals
- 5.7.2.2 Technical/Laboratory Director
- 5.7.2.3 PTS/ETS Technical Support
- 5.7.2.4 Instrument Specific Technical Support

5.7.3 Instrument maintenance, other than daily routine maintenance, must be recorded in the assigned instrument maintenance log. Once maintenance is complete, the operation of the system must be confirmed by the analysis of instrument specific QC, such as blanks, CCVs, or calibration.

6.0 Procedure

6.1 Sample Preparation: Aqueous Sample
- 6.1.1 See ARA SOP QA-5524.

6.2 Solid Samples by Microwave Assisted Extraction
- 6.2.1 See ARA SOP QA-5522.

6.3 Solid Samples by Soxhlet extraction (applicable for all building materials which would not be appropriate to microwave or for soil samples if necessary for the customer)
- 6.3.1 See ARA SOP QA-5305.

6.4 Sample Preparation: Wipe samples
- 6.4.1 Allow to come to room temperature
- 6.4.2 Add 0.1mL of surrogate to each wipe
- 6.4.3 Add 20mL of hexane and shake vigorously for 1 minute. Allow to settle and repeat, shaking 2 more times.
- 6.4.4 Draw up 1mL of extract and clean up with sulfuric acid.

6.5 Sample Preparation: Waste Dilution
- 6.5.1 To a certified clean pre-labeled 40mL VOC vial, add 500mg of product/sample
- 6.5.2 Add 0.1mL of surrogate solution to vial/sample.
- 6.5.3 Using a graduated cylinder, add 10mL of hexane

6.5.4 Shake the vial to combine product and solvent
6.5.5 Confirm the sample completely dissolves into the solvent
6.5.6 Pull off 1mL of extract and clean up with sulfuric acid. See section 6.6.
6.5.7 Consult the technical director or lab director and refer to SW-846 3580A for additional guidance regarding samples that do not completely dissolve in hexane.

6.6 Sulfuric Acid Clean-Up
6.6.1 In an autosampler vial, or other appropriate container, carefully add 0.8mL concentrated sulfuric acid to 1mL of sample extract. (Sample solvent must be hexane)
6.6.2 Cap the vial and shake vigorously for 15-30 seconds.
6.6.3 Allow the vial to settle for a minimum of one minute. Alternatively, the vial may be placed in a centrifuge to assist in the separation of the phases.
6.6.4 Once a defined separation has been established, use a glass pipette to pull off the top layer and put it in a separate labeled autosampler vial.
6.6.5 The extract may be subjected to additional acid clean up steps if considerable color is still present.
6.6.6 Dispose of the remaining sulfuric acid in the appropriate waste stream.

6.7 Instrument Setup: See the operating manual "HP 5890 Series II and HP 5890 Series II Plus Gas Chromatographs" for complete details on instrument setup.
See Table 4 for typical GC instrument conditions; summarized below:
Usable ECD conditions:
Column: 30m x .32mm ID with .53mm ID with guard as well as a confirmational column of dissimilar chemistry
Inlet Pressure: 10psi @120C
Split Flow: 55ml/min
Injector Temp: 200C
Detector Temp: 310C
Oven Temp: Init. Temp=120C, 9C/min to 300C and hold for 5min
Run time: 26.00 min (may vary depending on most recent acquisition method)
Injection size: 1uL

6.8 Analysis
6.8.1 PCBs: Identification of Aroclors is based on pattern recognition in the chromatograms. An Aroclor is considered a potential hit if at least three peaks fall within the retention time window of the Aroclor and the concentration is greater than half the detection limit.

6.8.2 A five point calibration for Aroclors 1016 and 1260 is prepared, bracketing the reporting range (single mixtures, e.g. 1242, can be used for calibration of project specific samples). Single point standards are analyzed for each of the other Aroclors of interest. For each Aroclor, 3-5 characteristic peaks are used for quantitation. Peaks are chosen that are at least 25% of the height of the largest Aroclor peak. For each Aroclor, the set of 3 to 5 peaks should include at least one peak that is unique to that Aroclor. The concentrations of each of these peaks are averaged to determine the final result. Care must be taken if more than one Aroclor is present in a sample, as peaks common to both Aroclors should not be used for quantitation. See Method 8082A for more specific guidance.

6.8.3 After following the calibration procedure, check the integration of blanks, QC samples and calibration standards, and samples. Blanks must not contain analytes of interest above the reporting limit. PCB standards and samples are checked for proper integration and analyzed with HP ChemStation software. Laboratory quality control samples are analyzed and quantitated the same as field samples.

6.8.4 A total of ten sample analyses are performed before checking calibration again or every twelve hours, whichever occurs first. A mid-level PCB standard (CCV) is used to verify the original calibration curve and must be within +/-15% of the expected concentration. If the CCV passes this criterion then the original curve is used to quantitate proceeding samples. Successive CCVs should vary in concentration throughout the entire run. If an Aroclor hit is above the upper level of the calibration curve, the sample must be diluted and rerun so that the response is within the calibration range or the original value must be qualified as estimated. Surrogate recoveries for waters and solids should be within 30-150%. For projects requiring DoD protocol, refer to the DoD Quality Systems Manual for Environmental Laboratories for required surrogate acceptance limits. If surrogate recoveries are not within this range, or if interferences are present which prevent quantitation, qualify the sample data and provide the sample chromatogram in the report. The sample will need to be re-extracted if there are no obvious chromatographic interferences. Otherwise the initial data will need to be qualified accordingly.

6.8.5 Second column confirmation is performed for all positive hits.

6.8.6 The RPD between the dual columns should be calculated.

6.8.7 For DOD compliant projects, results shall be reported from the primary column or detector, unless project-specific requirements state otherwise.

6.8.8 In the absence of project-specific reporting requirements, if the results exhibit disparity (ie. >40% RPD) both results should be reported and the disparity

should be noted. If the RPD is <40%, the concentration has been confirmed and higher result of the two columns can be reported.

6.8.9 If project-specific reporting requirements necessitate, both results can be reported.

6.8.10 For MCP PCB analysis, all QC must also be evaluated and pass on the secondary column by the same acceptance criteria as the primary column. For positive PCB concentrations in samples, the higher result of the two columns must be reported, unless obvious interferences are observed. If the RPD between the dual column results exceeds 40%, both sets of results are reported.

7.0 Quality Control Requirements

7.1 Method Performance

7.1.1 Initial Demonstration of Performance: This is used to characterize instrument performance (linear calibration ranges and analysis of the ICV) and laboratory performance (MDL's) prior to analysis by this method.

7.1.1.1 Linear Calibration Range: The concentration range over which the instrument response is linear and calibrated for. Must be a minimum of five standards and a blank and verified at ±15% by a second source. The calibration coefficient must be 0.995 or better.

7.1.1.2 Initial Calibration Verification: The ICV must be from a second source and be within ±15% of the true value.

7.1.1.3 Method Detection Limit: The MDLs must be established for Aroclor 1016 and 1260. Reagent water is fortified at a concentration of two to three times the estimated instrument detection limit. To determine the MDL values, take seven replicates of the fortified reagent water and process through entire analytical system (the same preparation process as the LCS). Calculate the values of the MDL for each analyte. Calculate the MDL as follows:

$$MDL = (t)*(S)$$

t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. For seven replicates t = 3.14.

S = Standard deviation of the replicate analysis.

MDLs should be determined initially or whenever there is a significant change to the method or instrumentation. The MDL data are stored electronically, by method, in the QA/MDL file folder.

7.1.2 LOD/LOQ: Refer to QAM *Analytical Procedures* section, for specific LOD/LOQ requirements. LOD/LOQ data are stored electronically in the QA/LOD&LOQ file folder.

7.1.3 IDC/CDC: Proficiency of the analyst is ensured by documentation of an Initial or Continuing Demonstration of Capability (IDC or CDC). Typically, the analyst analyzes four LCS's with acceptable precision and accuracy or shows acceptable performance of a blind sample analysis (usually a PT). See QAM for additional information.

7.1.4 Control Charts: Control Charts are quality control tools which graphically display QC data over time. The data required for generation of accuracy and precision control charts is maintained by the laboratory. See QAM for additional information.

7.1.5 Inter-laboratory performance: Proficiency Test studies, conducted by approved providers, constitute the majority of inter-laboratory performance comparisons. Proficiency tests are performed at a frequency as required by method, regulation, or accrediting body. See QAM for additional information.

7.2 Laboratory Performance Quality Control and Corrective Actions

7.2.1 Continuing Calibration Verification: The CCV must be within ±15% of the expected values. If the CCV performed at the beginning of a run (in order to verify a curve) is out of control, remake and re-analyze the CCV, if it passes the batch can continue. If the re-prepped standard is still out of control, a new calibration curve is prepared. If the CCV performed after 10 samples is out of control stop running. As regulations allow, if the CCV is high, samples that do not contain Aroclors above the quantitation limit are valid. For samples with detectable Aroclors, locate the source of the problem and re-analyze the CCV. If the CCV is within ±15% of the expected value, re-analyze these samples back to the last acceptable CCV. See ARA QAM for additional guidance regarding CCVs.

7.2.2 Continuing Calibration Blank: The CCB is analyzed at the same frequency as the CCV. The CCB must not have any Aroclor concentrations above the reporting limit. If the CCB is not below the reporting limit, the source of the error or contamination must be found and eliminated. The CCB level must be evaluated to determine the impact, if any to the samples. If sample data are impacted, the samples should be reanalyzed if possible.

7.2.3 Prep Blank: The PB is analyzed once every batch or 24 hours, whichever is

reached first. The PB undergoes the sample preparation process. The PB must be below the reporting limit of the analyte. For projects requiring DoD protocol, refer to the QAM for additional evaluation criterion for method blanks. If the batch has been impacted, the samples must be re-prepared and reanalyzed or the data flagged in the report. See ARA QA Manual for more specific guidance.

7.2.4 Laboratory Control Sample (LCS): The LCS is analyzed once every batch or 24 hours, whichever is reached first. The percent recovery is calculated and must be within the acceptable range of 40-140%. For projects requiring DoD protocol, refer to the DoD Quality Systems Manual for Environmental Laboratories for required LCS/D acceptance limits. If the LCS is out of control, all sample data associated with that LCS is suspect and must be reprocessed and reanalyzed after the problem is corrected. If this is not possible due to limited sample volume or holding times, the analytical data in the report must be flagged.

7.2.5 Laboratory Control Sample Duplicate (LCSD): See definition above. The LCSD is prepared at the same frequency as the LCS. The relative percent difference between the LCS and the LCSD should be $\leq$20% for waters and $\leq$30% for solids.

7.2.6 Matrix Spike and Matrix spike Duplicate: Performed at discretion of the lab or as requested by data user. A known amount of analyte is added to an aliquot of a randomly chosen sample. Percent recovery limits are 40-140%. For projects requiring DoD protocol, refer to the DoD Quality Systems Manual for Environmental Laboratories for required LCS/D acceptance limits. The calculation of the percent recovery is as follows:

$$R = (Cs - C)/s * 100$$

R = percent recovery
Cs = Fortified sample concentration
C=Sample background concentration
S= Concentration equivalent of analyte added to the sample

The RPD limits are +/- 50 % for Aroclors and +/- 30% for Congeners. The formula used to calculate the relative percent difference is:

$$RPD = (D1-D2)/ ((D1+D2)/2) *100$$

D1 = The initial result of the analyte
D2 = The duplicate result of the analyte

7.2.7 Surrogates: All samples, standards, QC, blanks, are spiked with surrogates. The surrogate recoveries must be 30-150%. For projects requiring DoD protocol, refer to the DoD Quality Systems Manual for Environmental Laboratories for required surrogate acceptance limits. If the surrogate limits are not met and sample matrix interference is obvious, the samples do not need to be re-extracted. For these samples, the chromatogram is provided to the customer as well as a footnote discussing the interference. However, if sample is available and there is no apparent reason for the failure, the samples are to be re-extracted. If re-extractions confirm the initial result, both sets of results are to be reported. Exceedances are noted in the report.

8.0 Responsibilities

8.1 The analyst uses the Quality Control Criteria outlined in this SOP to evaluate and validate the data. It is the analyst's or technician's responsibility/job to follow this SOP and record all the data in the lab notebook. Any irregularities within the test or samples should be recorded in the lab notebook and reported to the lab manager who is responsible for reviewing the data.

9.0 Health and Safety

9.1 Normal, accepted laboratory safety practices should be followed during reagent preparation and instrument operation. However, every sample in the lab should be handled as if it is hazardous waste or a potential health hazard. The following parameters covered by this method have been tentatively classified as known or suspected human or mammalian carcinogens: dichloromethane and PCBs. All technicians shall be familiar with the Chemical Hygiene Plan. Refer to Chemical Hygiene Plan (SOP QA604), and SDS sheets.

10.0 Pollution Prevention and Waste Management

10.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Samples and standards following a run are stored at 4ºC for 30 days then discarded after this time period into appropriately labeled containers. The containers are then removed by a waste management specialist.

10.2 The EPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Waste management in this laboratory is regulated by the Safety director. Local, state and federal regulations are followed in the disposal of waste generated from this analysis.

Title: Analysis of PCB in Soil and Water Extracts by EPA 8082A

Table 1
Analytical Sequence with Acceptance Criteria

PCB Sequence

File or Vial Number	Instrument Run Log	Acceptance Limit
1	ICV*	± 15%
2	ICB**	BRL
3	PB	BRL
4	LCS	40-140%
5	LCSD	40-140%
6	Sample 1	
7	Sample 2	
8	Sample 3	
9	Sample 4	
10	Sample 5	
11	Sample 6	
12	Sample 7	
13	Sample 8	
14	Sample 9	
15	Sample 10	
16	CCB	BRL
17	CCV	+/- 15%
18	Sample 11	
19	Sample 12	
20	Sample 13	
21	Sample 14	
22	Sample 15	
23	Sample 16	
24	Sample 17	
25	Sample 18	
26	Sample 19	
27	Sample 20	
28	CCB	BRL
29	CCV	+/- 15%

* Sample 1 above will be an ICV if run right after a curve otherwise it would be a CCV.
* Sample 2 above will be an ICB if run right after a curve otherwise it would be a CCB.

Title: Analysis of PCB in Soil and Water Extracts by EPA 8082A

Table 2

Reporting Limits

Compound	Water (ug/L)	Solid (ug/g)
PCB 1016	0.2	0.2
PCB 1221	0.2	0.2
PCB 1232	0.2	0.2
PCB 1242	0.2	0.2
PCB 1248	0.2	0.2
PCB 1254	0.2	0.2
PCB 1260	0.2	0.2
Congeners	0.01	

Table 3

CEM Microwave Operating Parameters

Method Name: 3546
Vessels: MarsXpress Vessels
Operating Power: 100% equals 1600W
Parameters: Heat to 100°C during a 10 minute period
Hold temperature for 10 minutes
Cool down for 10 minutes

Title: Analysis of PCB in Soil and Water Extracts by EPA 8082A

Table 4: Typical GC instrument conditions for PCB analysis (subject to change):

INSTRUMENT CONTROL PARAMETERS

Sample Inlet: GC
Injection Source: GC ALS

7673 Injector

Front Injector:

Sample Washes	1
Sample Pumps	4
Injection Volume	1.0 microliters
Syringe Size	10.0 microliters
On Column	Off
Nanoliter Adapter	Off
PostInj Solvent A Washes	4
PostInj Solvent B Washes	0
Viscosity Delay	0 seconds
Plunger Speed	Fast

Back Injector:
No parameters specified

HP5890 Temperature Parameters

Zone Temperatures:	State	Setpoint
Inlet A:	On	175 C
Inlet B:	Off	175 C
Detector A:	On	310 C
Detector B:	On	310 C
Auxiliary:	Off	50 C

Oven Parameters:

Oven Equib Time:	0.00 minutes
Oven Max:	340 C
Oven State:	On
Cryo State:	Off
Cryo Blast:	Off
Ambient:	25 C

Title: Analysis of PCB in Soil and Water Extracts by EPA 8082A

Oven Program:

Initial Temperature: 120 C
Initial Time: 0.50 minutes

Level	Rate (C/minute)	Final Temperature (C)	Final Time (minutes)
1	45.0	200	0.00
2(A)	15.0	230	0.00
3(B)	15.0	330	1.00

Next Run Time: 11.94 minutes

HP5890 Inlet Pressure Programs

GC Pressure Units: psi

Inlet A:

Constant Flow: On
Constant Flow Pressure: 12.0 psi
Constant Flow Temperature: 120 C
Initial Pressure: 0.0 psi
Initial Time: 480.00 minutes

Level	Rate (psi/minute)	Final Pressure (psi)	Final Time (minutes)
1	0.00	0.0	0.00
2(A)	0.00	0.0	0.00
3(B)	0.00	0.0	0.00

Total Program Time: 480.00 minutes

Column Length: 30.00 m
Column Diameter: 0.320 mm
Gas: He
Vacuum Compensation: Off

Inlet B:

Constant Flow: Off
Constant Flow Pressure: 12.0 psi

Title: Analysis of PCB in Soil and Water Extracts by EPA 8082A

Constant Flow Temperature: 120 C
Initial Pressure: 0.0 psi
Initial Time: 480.00 minutes

Level	Rate (psi/minute)	Final Pressure (psi)	Final Time (minutes)
1	0.00	0.0	0.00
2(A)	0.00	0.0	0.00
3(B)	0.00	0.0	0.00

Total Program Time: 480.00 minutes

Column Length: 34.94 m
Column Diameter: 0.520 mm
Gas: He
Vacuum Compensation: Off

Title: Preparation & Analysis of Organo-Chlorine Pesticides in Soil and Water Samples by Method 8081B

QA Officer: _____ Date: 8/2/17

Laboratory Director: _____ Date: 8/2/17

Author: _____ Date: 8/3/17

Analyst: _____ Date: _____

By signing this SOP, the analyst has read and understood the latest version of the test method; has read and understood this SOP; acknowledges receipt of this SOP; and is responsible for the implementation of this SOP.

Revision History:

Revision	Changes	Date
1	Method 3546 added	5/07
2	Add QC Prep information	8/07
3	Solvent for 3546, reformatting, typos. QC #s from LIMs. Removed info on two solutions, DoD Language	12/10
4	Changed Co name. Clarified Curve fit weighting & quadratic acceptance; breakdown timeframe, Extraction procedures moved to separate SOPs.	8/11
5	Added 4 additional RIM pesticides, list of co-eluting pesticides	1/13
6	Add practice of verification of a detection limit check standard, MDL location; updated calibration concentrations	7/13
7	Updated calibration concentrations; changed BDL to BRL; MDL & LOQ locations	4/14
8	Update Florisil procedure for solid.	2/15
9	Added the toxaphene and chlordane RL standard. Method performance added. Section 8 restructured.	3/16
10	Endrin criteria missing in section 8. Added. Specified primary method of solid extraction in section 6.2; Added table with typical GC instrument conditions; updated reporting limits table to account for column clean-up step; clarified tox/chlor quant method requirement; corrected Florisil clean-up reference	2/17
11	Clarify DOD requirement: report positive confirmed results from primary column; updated CCV section to refer to QAM for additional guidance.	7/17

Title: Preparation & Analysis of Organo-Chlorine Pesticides in Soil and Water Samples by Method 8081B

1.0 Purpose and Applicability

1.1 This procedure details the steps to be followed for the preparation, clean-up and analysis of water and soil samples for organo-chlorine pesticides. It is applicable to the following compounds: Aldrin, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, chlordane, 4,4'-DDT, 4,4-DDE, 4,4'-DDD, dieldrin, endosulfan I, endosulfan II, endosulfan sulfate, endrin, endrin aldehyde, heptachlor, heptachlor epoxide and toxaphene. For special projects (i.e., ACOE RIM projects), this method is also applicable to the additional four compounds: hexachlorobenzene, oxychlordane, trans-Nonachlor, and cis-Nonachlor. Analysis is performed by Gas Chromatography-Electron Capture Detector (GC-ECD) using a dual column configuration.

1.2 This method is recommended for use only by or under the supervision of experienced analysts in the use of a gas chromatograph and the interpretation of pesticides gas chromatography data. Each analyst must demonstrate the ability to generate acceptable results with this method as described in the procedure in section 7.0.

2.0 Definitions

2.1 Initial Calibration Verification (ICV): A standard at a mid-point concentration in the calibration curve to verify the integrity of the curve. The ICV is made using standards from a separate manufacturer as the standards used for initial calibration. The acceptance criteria is provided in section 8.

2.2 Continuing Calibration Verification (CCV): A CCV is analyzed before and after every ten samples. The CCV is analyzed at a mid-point concentration of the calibration curve. The concentration of the CCV is varied periodically. The acceptance criteria is provided in section 8.

2.3 Calibration Standard (ICal): Standards used to calibrate the instrument's response with respect to analyte concentration.

2.4 Field Duplicates: A replicate sample taken in the field, used to monitor the precision of the whole analytical process, including field sampling procedures. These are done only at a customer's request.

2.5 Laboratory Control Sample (LCS): An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The LCS is prepared and analyzed exactly as a sample. Its purpose is to demonstrate that procedures are in control, and the laboratory is capable of making accurate and precise measurements. The acceptance criteria are listed in Table 2.

2.6 Prep Blank (PB): An aliquot of reagent water or other blank matrix which is treated exactly as a sample, including exposure to all glassware, equipment, and reagents that are used with other samples. The PB is used to determine if the

method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. Criteria is provided in section 8. For projects requiring DoD protocol, refer to the QAM for additional evaluation criterion for preparation blanks.

2.7 Stock Standard: A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source (e.g. Absolute Standards, Ultra Scientific). Documentation of the purity must be retained for traceability in the organic standards binder.

2.8 Matrix Spike: An aliquot of a field sample spiked with a known amount of a standard.

3.0 Applicable Documents/References

3.1 Standards Methods 509A
3.2 SW-846 Method 3510C Separatory Funnel Extraction
3.3 SW-846 Method 3550B Sonication Extraction
3.4 SW-846 Method 3540C Soxhlet Extraction
3.5 SW-846 Method 3546 Microwave Accelerated Extraction
3.6 SW-846 Method 3620B Florisil Column Cleanup
3.7 SW-846 Method 3660B Sulfur Cleanup
3.8 SW-846 Method 8081B GC-ECD Analysis
3.9 ARA Organic Standards Preparation Logbook
3.10 ARA GC Instrumentation Run Logbook
3.11 ARA Glassware Preparation SOP
3.12 ARA QA Manual
3.13 Current DoD Quality Systems Manual for Environmental Laboratories
3.14 Manual Integration SOP QA5000
3.15 MADEP BWSC-CAM July 2010
3.16 ARA SOP QA-400 Sample Receiving and Identification

4.0 Materials and Apparatus

4.1 Equipment:

4.1.1 Class A volumetric flasks as required.
4.1.2 Syringes as required for dilutions.
4.1.3 Auto-sampler vials for standards and samples.
4.1.4 HP/Agilent 5890 Gas Chromatograph configured with dual columns and ECDs and HP3365 & EnviroQuant data system.
4.1.5 Analytical balance for weighing standards and solids.
4.1.6 Refrigerator and freezer for storage of samples, standards and extracts.
4.1.7 Analytical columns: Primary – Restek CLP-II 30M x 0.32mm ID,

Confirmation - Restek CLP-I 30M x 0.32mm ID.

4.1.8 Labconco Rapid-Vap

4.2 Reagents/Standards:

4.2.1 Hexane - pesticide quality or equivalent.

4.2.2 Pesticide standards Mix A and Mix B at concentrations of 5 to 50 ug/mL purchased from Restek, Absolute Standards, or equivalent.

4.2.3 Standards of Chlordane and Toxaphene purchased from ChemService or equivalent.

4.2.4 Evaluation mix containing DDT and Endrin for checking breakdown.

5.0 Method Summary

5.1 Aqueous samples are collected in 1 liter glass containers with Teflon seals. Aqueous samples are extracted within 7 days of collection. If the extraction is not started within 72 hours of collection, the pH is adjusted to 5.0-9.0 with either NaOH or H_2SO_4. Soil samples are collected in 4 ounce glass containers with Teflon seals and are extracted within 14 days of collection. Shipment of samples must follow the requirements of the method. Refer to SOP QA-400.

5.2 See Table 3 for typical Reporting Limits.

5.3 Sample analysis must occur within 40 days of sample extraction. Extracts are injected into the GC for analysis. After the GC analysis is completed, the data are evaluated using HP/Agilent software.

5.4 Interferences

5.4.1 Contaminants in reagents and solvents can cause elevated baselines and peaks associated or not associated with analytes of interest. Additionally, contamination on glassware can be problematic. Refer to Glassware cleaning SOP.

5.4.2 Co-extracted polar organic compounds can cause interference in chromatograms. Florisil column cleanup is very effective in removing these compounds. See section 6.4 for cleanup procedure.

5.4.3 Phthalate contamination can be misidentified as a target analyte having the same retention time as the contaminant. Second column confirmation will disqualify these peaks, however this contamination can complicate the data evaluation. Avoid handling plastics during the extraction process to minimize phthalate contamination.

5.4.4 Elemental Sulfur, especially at high concentrations, poses similar problems in chromatograms. Elemental copper is added to extracts to remove the sulfur. Refer to the Method 3660B for the sulfur cleanup procedure.

5.4.5 Co-eluting target compounds: Alpha-Chlordane and trans-Nonachlor co-elute on column #1. Therefore, care must be taken when using a curve that incorporates the four additional compounds. Any reportable alpha-Chlordane or trans-Nonachlor concentrations must be thoroughly examined and reported from column #2.

6.0 Procedure

6.1 Sample Preparation: Aqueous samples
See ARA SOP# 5524 for extraction procedure.

6.2 Sample Preparation: Soil/solid samples by Microwave extraction
See ARA SOP# 5522 for extraction procedure. This is the primary method of extraction for Pesticides in soil

6.3 Sample Preparation: Soil/solid samples Sonication extraction
See ARA SOP# 5520 for extraction procedure.

6.4 Florisil Clean up:

6.4.1 Plug a disposable chromatography column with a small amount of glass wool. Place 1g of florisil that is kept in the oven to remove any impurities.

6.4.2 Rinse florisil in column with enough hexane just to make it wet.

6.4.3 In a syringe pull 0.1mL of fresh methanol then pull 0.5mL of the sample extract for water samples. For solid samples pull 0.2mL of fresh methanol and 1mL of the extract.

6.4.4 Inject into column and rinse with a mixture of 5mL of MeCl2 and 15mL of hexane. Collect in a pre labeled 40mL vial.

6.4.5 Concentrate sample in evaporator to 1mL. Remove from RV tube into a properly labeled 1.5mL vial

7.0 Analysis

7.1 Refer to the manual “HP 5890 Series II Plus Gas Chromatographs” for instrument setup instructions.

7.2 See Table 3 for typical GC instrument conditions.

7.3 GC performance must be demonstrated prior to sample and calibration standard analysis. An evaluation mixture containing DDT and Endrin is used for calculation of breakdown of these analytes. Acceptability criteria is provided in section 8. Degradation breakdown must be evaluated every 12 hours. If this criterion is not achieved, instrument maintenance must be performed. Refer to “Pest Endrin&DDT Breakdn Calc.xls” to facilitate the breakdown calculation.

7.4 Pesticides Working Standards: Prepared by diluting commercial stock mixes to

give the following concentrations for the analytes of interest: 2, 1, 0.5, 0.1, 0.02, and 0.01ug/mL. Toxaphene is diluted from a commercial stock to concentrations of 0.5 to 5.0ug/mL. Standards should be refrigerated and must be replaced after twelve months, or sooner if comparison with a check standard indicates a problem. A single point surrogate standard is run at the level expected in extracts. This standard contains two surrogates: Tetrachloro-m-xylene (TCMX) and Decachlorobiphenyl (DCBP).

7.5 Initial Calibration:

7.5.1 Pesticides: An evaluation (DDT/Endrin) mix is run; see Section 8.2.2 for acceptance limits. Once the evaluation mix passes, a multi-point (5 point minimum) calibration is performed using the pesticide mixes referred to above. The calibration range should cover the expected range of concentrations found in samples and the working range of the detector, with the lowest point at or below the reporting limit.

7.5.2 Calibration Verification Requirements: The analyst's discretion is used in selecting the curve fit and weighting which best represents the data, paying particular attention to the low end of the calibration range. Usually, the inverse of concentration is most appropriate weighting. For linear regression, the calibration curve must have an r^2 of 0.99 or greater. A quadratic curve fit may also be used. To use a quadratic curve fit, a minimum of 6 points must be included in the calibration and the curve fit must have an r^2 of 0.99 or greater. An ICV must be run following the calibration curve.

7.5.3 A detection limit check standard at the reporting limit is analyzed to verify detectability of the system throughout a sequence. The detection limit check standard is most often run after initial instrument QC and a set of samples, to verify detection with changes in system performance. Similarly, a standard mix of toxaphene and chlordane should be run at the corresponding reporting limits (0.2 ug/mL for toxaphene and 0.5 ug/mL for technical chlordane). Toxaphene and chlordane may also be analyzed separately at the above reporting limits. This reporting limit standard is run to allow for pattern recognition of these analytes. A separate quantitation method for toxaphene and chlordane should be used on the reporting limit check standards and on all QC and samples for detectability. If these compounds are detected or recognized in a sample, then the sample must be run again after calibration or calibration verification of a valid quantitation method.

1.1.1 Data Evaluation: A chromatographic peak is considered a potential pesticide hit if the peak falls within the retention time window for the analyte on the primary column and the concentration is greater than half

the reporting limit. The results of a potential hit are compared with the data from the confirmation column. If the pesticide is present on the secondary column and the quantitation agrees within ±40%, then the result from the primary column is reported. In some cases, regulatory programs dictate the higher of the two results is reported. For DOD compliant projects, results shall be reported from the primary column or detector, unless project-specific requirements state otherwise.

7.5.4 A total of up to ten samples are performed before verifying the calibration. A mid-level Pesticide standard (CCV) is used to verify the calibration curve. See section 8 for acceptance criteria. If the CCV is acceptable, the calibration curve is considered to be valid. If a pesticide is determined to be above the highest point of the calibration curve, the sample is diluted and rerun so that the response is bracketed by points in the initial calibration. Any reported data not bracketed by standards must be flagged as an estimated concentration in the final report. Surrogate recoveries must be acceptable. If surrogate recoveries are outside this range, or if interferences are present preventing quantitation, the effected samples are re-extracted if sufficient sample exists. If no additional sample exists, data are reported and flagged accordingly.

7.6 Sample Calculations

7.5.1 The results obtained using the chromatographic data reduction software are in units of ug/mL of the extract. These results must be converted to ug/L or ug/g accordingly, adjusting for measured weights and volumes measured in the extraction process.

7.5.2 To facilitate calculation and reporting these results, the following spreadsheets are used.

Water samples: Pest AQ 8081 Template.XLS
Soil/Solid samples: Pest Solid 8081 Template.XLS.

7.5.3 <u>Aqueous Samples</u>:

Concentration (ug/L) = (Extract Concentration (ug/mL) * Intermediate volume(mL) * Final volume(mL) * Instrument Dilution factor)/(Volume extracted (L) * Aliquot volume(mL))

7.5.4 <u>Soil/Solid Samples</u>:

Concentration (ug/g) = (Extract Concentration (ug/mL) * Intermediate volume(mL) * Final volume(mL) * Instrument Dilution factor)/(sample weight (g) *dry fraction* Aliquot volume(mL))

7.5.5 Calculation of Quality Control Samples

7.5.5.1 Blanks – Calculate concentration of any analytes present

in the blank. Blanks are considered contaminated if any analytes are present at greater than the reporting limit. For projects requiring DoD protocol, refer to the QAM for additional evaluation criterion for method blanks.

7.5.5.2 Spikes - Determine the amount of analyte in the sample that was chosen for spiking (note that for laboratory control samples this number is zero). Calculate the concentration of each analyte found in the spiked sample using the formulas above. Refer to extraction preparation log book for spike concentration. The spreadsheet: Pest LCSChk.XLS can be used to facilitate calculation.

Recovery (%) = 100*(Amount found in spiked sample - amount found in original sample)/(Spiked concentration)

Most spikes are reported with spike duplicates. Precision of analysis is expressed as the Relative Percent Difference (RPD) calculated according to the following equation:

RPD = 100 * (Spike1 - Spike2)□ / ((Spike1 + Spike2)/2)

8.0 Quality Control

8.1 Method Performance

8.1.1 Initial Demonstration of Capability (IDC): Prior to use of the method, a demonstration of capability must be performed as described in the QA Manual. This is used to characterize method and analyst performance prior to analysis by this method. Acceptable recovery ranges for the QC samples analyzed in the IDC are the same as acceptability limits for Laboratory Control Samples.

8.1.1.a Calibration Range: This is the working range of the calibration curve.

8.1.1.b Initial Calibration Verification: The ICV must be prepared from a second source from the initial calibration.

8.1.1.c Method Detection Limit: The MDLs must be established for all analytes. A blank sample matrix is fortified at a concentration of two to three times the anticipated method detection limit. To determine the MDL, seven replicates are processed through the entire analytical system (the same preparation process as the LCS). Calculate the concentration for each analyte. Calculate the MDL

as follows:

$$MDL = (t)*(S)$$

t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. For seven replicates t = 3.14.

S = Standard deviation of the replicate analysis.

MDLs should be determined initially and whenever there is a significant modification to the analytical procedure. The MDL data are stored electronically, by method, in the QA/MDL file folder.

8.1.2 LOD/LOQ: Refer to QAM *Analytical Procedures* section, for specific LOD/LOQ requirements. LOD/LOQ data are stored electronically in the QA/LOD&LOQ file folder**.**

8.1.3 IDC/CDC: Proficiency of the analyst is ensured by documentation of an Initial or Continuing Demonstration of Capability (IDC or CDC). Typically, the analyst analyzes four LCS's with acceptable precision and accuracy or shows acceptable performance of a blind sample analysis (usually a PT). See QAM for additional information.

8.1.4 Control Charts: Control Charts are quality control tools which graphically display QC data over time. The data required for generation of accuracy and precision control charts is maintained by the laboratory. See QAM for additional information.

8.1.5 Inter-laboratory performance: Proficiency Test studies, conducted by approved providers, constitute the majority of inter-laboratory performance comparisons. Proficiency tests are performed at a frequency as required by method, regulation, or accrediting body. See QAM for additional information.

8.2 Laboratory Performance Quality Control and Corrective Actions

8.2.1 Consult the QA Manual for additional corrective action guidance.

8.2.2 Endrin/DDT Breakdown Standard: Breakdown of DDT and Endrin to DDE, DDD, Endrin Aldehyde, and Endrin Ketone must not exceed 15%.

8.2.3 Initial/Continuing Calibration Verification: The recovery of the ICV and CCV must be within ±15% of the expected concentrations. If the ICV or CCV performed at the beginning of the run is out of control, either re-

prepare or re-analyze the CCV, or recalibrate the instrument. As regulations allow, if the CCV is high, samples that do not contain target analytes above the quantitation limit are valid. Refer to the ARA QA Manual for additional guidance concerning CCV evaluation.

8.2.4 Detection Limit Check Standard (DET): The peaks must fully resolve in the detection limit chromatogram for accurate identification. The individual compounds must be able to be qualitatively identified by the current quantitation method.

8.2.5 Chlordane/Toxaphene Reporting Limit Check Standard (C/T RL): The peaks must fully resolve in the reporting limit standard to allow for accurate identification.

8.2.6 Continuing Calibration Blank: The CCB is analyzed following a CCV. The concentrations found in the CCB must be below the reporting limit for the analyte. If the CCB is not below the reporting limit the source of the contamination must be located and corrected. Any effect of the CCB on the samples in the batch must be evaluated and noted in the report if the sample results are impacted. If the concentration of the sample is greater than ten times the amount in the blank, there may be no impact to the data.

8.2.7 Prep Blank: The PB is prepared with every batch of 20 samples or 24 hours, whichever occurs first. The PB follows the same process as samples. The PB results must be below the reporting limit of each analyte. For projects requiring DoD protocol, refer to the QAM for additional evaluation criterion for method blanks.

8.2.8 Laboratory Control Sample (LCS): The LCS is analyzed with each batch as described in the Prep blank section. The percent recovery is calculated for each analyte and must be 40% to 140% of the true value. If the LCS is out of control, the source of the problem must be identified. Refer to the spreadsheet Pest LCSChk.XLS to assist in calculations. For projects requiring DoD protocol, refer to the DoD Quality Systems Manual for Environmental Laboratories for required LCS/D acceptance limits.

8.2.9 Surrogate Standard Solution: Tetrachloro-m-xylene (TCMX) and Decachlorobiphenyl (DCBP) are used as surrogate standards for extracted batch QC and samples. These compounds are added to samples and subsequently analyzed to determine extraction efficiency. The acceptable recovery for these compounds is 30% to 150%.

8.2.10 Matrix Spike and Matrix spike Duplicate: Matrix spike and matrix spike duplicates are analyzed per customer request. A known amount of analytes are added to an aliquot of sample. The calculation of the percent recovery is as follows:

$$R = (Cs - C)/S * 100$$

R = percent recovery
Cs = Fortified sample concentration
C=Sample background concentration
S= Concentration of analyte added to the sample

The formula used to calculate the relative percent difference is:

$$RPD = (D1-D2)/((D1+D2)/2) *100$$

D1 = The initial result of the analyte
D2 = The duplicate result of the analyte

The RPD acceptance criteria is 30% for single component compounds and 50% for multi-component compounds. MS and MSD must have recoveries between 30% and 150% to meet acceptability criteria. For projects requiring DoD protocol, refer to the DoD Quality Systems Manual for Environmental Laboratories for required MS/D acceptance limits.

9.0 Responsibilities

The analyst uses the Quality Control Criteria outlined in this SOP to evaluate and validate the data. It is the analyst's or technician's responsibility/job to follow the Quality Control Criteria outlined in this SOP to evaluate and validate the data. It is the analyst or technicians job to note any irregularities within the test or samples should be recorded in the lab notebook and reported to the lab manager who is responsible for reviewing the data.

10.0 Health and Safety

Normal, accepted laboratory safety practices should be followed during reagent preparation and instrument operation. However, every sample in the lab should be handled as if it is hazardous waste or a potential health hazard. The following parameters covered by this method have been tentatively classified as known or suspected human or mammalian carcinogens: 4-4'DDT, 4-4'DDD and BHC. All technicians shall be familiar with the Chemical Hygiene Plan. Refer to Chemical Hygiene Plan (SOP QA604), and SDS sheets.

11.0 Pollution Prevention and Waste Management

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. When possible, reduced sample volumes

are requested for solid samples. Samples and standards following a run are stored at 4C for 30 days then discarded after this time period into appropriately labeled containers. The waste generated from the extraction is collected in designated waste stream carboys and removed per the Laboratory Waste Disposal SOP by a waste management specialist.

The EPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Waste management in this laboratory is managed by the Hazardous Waste Coordinator.

Title: Preparation & Analysis of Organo-Chlorine Pesticides in Soil and Water Samples by Method 8081B

Table 1
Sample Analytical Sequence with Acceptance Criteria

File Name/Auto-sampler Position	Sample Identification		Acceptance Limit
1	*CCV		± 15%
2	CCB		BRL
3	PB		BRL
4	LCS		40-140%
5	Sample 1		
6	Sample 2		
7	Sample 3		
8	Sample 4		
9	Sample 5		
10	Sample 6		
11	Sample 7		
12	Sample 8		
13	Sample 9		
14	Sample 10		
15	CCV		+/-15%
16	CCB		BRL
17	DET	Identifiable	
18	C/T RL	Identifiable	
19	Sample 11		
21	Sample 12		
22	Sample 13		
23	Sample 14		
24	Sample 15		
25	Sample 16		
26	Sample 17		
27	Sample 18		
28	Sample 19		
29	Sample 20		
30	Sample 20 Matrix Spike		30-150%
31	Sample 20 Matrix Spike Duplicate		30-150%
32	CCV		+/- 15%
33	CCB		BRL
34	DET	Identifiable	
35	C/T RL	Identifiable	

* Sample 1 above will be an ICV if run right after a curve otherwise it would be a CCV.

Title: Preparation & Analysis of Organo-Chlorine Pesticides in Soil and Water Samples by Method 8081B

Table 2
Typical Reporting Limits

Analyte	Reporting Limit (ug/L) (Note: No column clean-up)	Reporting Limit (ug/L) (Note: With column clean-up)	Reporting Limit (ug/g) (Note: RL not impacted by column clean-up step)
alpha-BHC	0.05	0.1	0.04
beta-BHC	0.05	0.1	0.04
delta-BHC	0.05	0.1	0.04
gamma-BHC (Lindane)	0.05	0.1	0.04
Heptachlor	0.05	0.1	0.04
Aldrin	0.05	0.1	0.04
Heptachlor Epoxide	0.05	0.1	0.04
Endosulfan I	0.05	0.1	0.04
Dieldrin	0.05	0.1	0.04
4,4'-DDE	0.05	0.1	0.04
Endrin	0.05	0.1	0.04
Endosulfan II	0.05	0.1	0.04
4,4'-DDD	0.05	0.1	0.04
Endosulfan Sulfate	0.05	0.1	0.04
4,4'-DDT	0.05	0.1	0.04
Methoxychlor	0.05	0.1	0.04
Endrin Ketone	0.05	0.1	0.04
Endrin Aldehyde	0.05	0.1	0.04
alpha-Chlordane	0.05	0.1	0.04
gamma-Chlordane	0.05	0.1	0.04
Toxaphene	0.4		0.2

Note: Solid reporting limits are based on 100% dry weight.

Title: Preparation & Analysis of Organo-Chlorine Pesticides in Soil and Water Samples by Method 8081B

Table 3: Typical GC instrument conditions for Pesticide analysis (subject to change):

INSTRUMENT CONTROL PARAMETERS

Sample Inlet: GC
Injection Source: GC ALS

7673 Injector

Front Injector:

Sample Washes	1
Sample Pumps	4
Injection Volume	1.0 microliters
Syringe Size	10.0 microliters
On Column	Off
Nanoliter Adapter	Off
PostInj Solvent A Washes	4
PostInj Solvent B Washes	0
Viscosity Delay	0 seconds
Plunger Speed	Fast

Back Injector:
No parameters specified

HP5890 Temperature Parameters

Zone Temperatures:	State	Setpoint
Inlet A:	On	175 C
Inlet B:	Off	175 C
Detector A:	On	310 C
Detector B:	On	310 C
Auxiliary:	Off	50 C

Oven Parameters:

Oven Equib Time:	0.00 minutes
Oven Max:	340 C
Oven State:	On
Cryo State:	Off
Cryo Blast:	Off
Ambient:	25 C

Title: Preparation & Analysis of Organo-Chlorine Pesticides in Soil and Water Samples by Method 8081B

Oven Program:

Initial Temperature: 80 C
Initial Time: 1.00 minutes

Level	Rate (C/minute)	Final Temperature (C)	Final Time (minutes)
1	25.0	165	1.00
2(A)	8.0	300	3.00
3(B)	0.0	50	1.00

Next Run Time: 25.27 minutes

HP5890 Inlet Pressure Programs
GC Pressure Units: psi

Inlet A:

Constant Flow: On
Constant Flow Pressure: 12.0 psi
Constant Flow Temperature: 120 C
Initial Pressure: 0.0 psi
Initial Time: 480.00 minutes

Level	Rate (psi/minute)	Final Pressure (psi)	Final Time (minutes)
1	0.00	0.0	0.00
2(A)	0.00	0.0	0.00
3(B)	0.00	0.0	0.00

Total Program Time: 480.00 minutes

Column Length: 34.94 m
Column Diameter: 0.520 mm
Gas: He
Vacuum Compensation: Off

Inlet B:

Constant Flow: Off
Constant Flow Pressure: 12.0 psi
Constant Flow Temperature: 120 C

Title: Preparation & Analysis of Organo-Chlorine Pesticides in Soil and Water Samples by Method 8081B

Initial Pressure: 0.0 psi
Initial Time: 480.00 minutes

Level	Rate (psi/minute)	Final Pressure (psi)	Final Time (minutes)
1	0.00	0.0	0.00
2(A)	0.00	0.0	0.00
3(B)	0.00	0.0	0.00

Total Program Time: 480.00 minutes

Column Length: 34.94 m
Column Diameter: 0.520 mm
Gas: He
Vacuum Compensation: Off

Absolute Resource Associates
Standard Operating Procedure QA-5305

Title: Soxhlet Extraction by EPA Method 3540C

QA Officer: _____ Date: 2/20/17

Laboratory Director: _____ Date: 2/20/17

Author: _____ Date: 2/20/17

Analyst: _____ Date: _____

By signing this SOP, the analyst has read and understood the latest version of the test method; has read and understood this SOP; acknowledges receipt of this SOP; and is responsible for the implementation of this SOP.

Revision History:

Revision	Changes	Date
1	Clarification of some procedural steps	6/06
2	Clarify extraction solvent	6/08
3	Glassware cleaning steps added, Solvent system table, Co name changed.	8/11
4	Added Sand cleaning procedure	7/13
5	RV Program Table	3/14
6	Changes to Chiller information	2/15
7	Revised method capabilities and sample preparation information, revised RV settings, added method performance reference	3/16
8	Revised extraction time in sec 5.1.1, revised section 6.1.13 to include accommodations for large extract volume and extracts concentrated to 5mL, revised evaporator speed for SVOC-10.	2/17

Title: Soxhlet Extraction by EPA Method 3540C

1.0 Purpose and Applicability

1.1 This method is applicable to extraction of non-volatile and semi-volatiles organic compounds as well as isolation and concentration of water-insoluble and slightly water soluble organic compounds. This extraction procedure is applicable to the following determinative methods:

Method	SOP#
EPA8270	5200
EPA8082	5303
EPA8081	5304
EPA8015	5501

1.2 This method is capable of extracting these compounds from soils, sludges, wastes and other materials.

1.3 This method is recommended for use only by or under the supervision of analysts experienced in the use of soxhlet glassware and solvents. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 Definitions

2.1 Field Duplicates: A duplicate sample taken in the field. These are only done at the customer's request

2.2 Laboratory Control Sample (Laboratory Fortified Blank) (LCS): An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

2.3 Laboratory Control Sample Duplicate (Laboratory Fortified Blank Duplicate) (LCSD): See definition above.

2.4 Prep Blank (Laboratory Reagent Blank) (PB): An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, and reagents that are used with other samples. The PB is used to determine if the method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. The PB must be below the reporting limit for the analyte.

2.5 Matrix Spike / Matrix Spike Duplicate: An aliquot of a field sample spiked with a known amount of standard. Matrix spike / matrix spike duplicates are only done

when requested by the customer.

3.0 Applicable Documents/References

3.1 EPA Method 3540C.
3.2 ARA SOP QA-800, Use, Calibration, and Maintenance of Laboratory Equipment Inorganics Laboratories
3.3 ARA SOP QA-400 Sampling Receipt and Storage
3.4 ARA Sample Readiness SOP QA801
3.5 ARA QA Manual QAM-003
3.6 Labconco RapidVap Evaporation System User's Manual

4.0 Materials and Apparatus

4.1 Equipment

4.1.1 Soxhlet extractor/hotplate apparatus
4.1.2 500mL Boston round bottom flask
4.1.3 disposable glass fiber extraction thimble
4.1.4 soxhlet extraction tube
4.1.5 condenser
4.1.6 disposable Scoopula
4.1.7 50 mL beaker
4.1.8 re-circulating chiller
4.1.9 disposable pipettes
4.1.10 various sizes of gas tight micro-liter syringes
4.1.11 laboratory scale
4.1.12 RapidVap extract concentrator
4.1.13 RapidVap concentration tube
4.1.14 40mL VOC vials w/ Teflon lined caps
4.1.15 8mL VOC vials w/ Teflon lined caps
4.1.16 1.5mL auto sampler vials w/ Teflon lined caps
4.1.17 forceps/tongs
4.1.18 Drying oven capable of maintaining 105 C
4.1.19 Disposable aluminum weighing dishes

4.2 Reagents/Standards:

4.2.1 Sodium Sulfate: Purchased from a reputable supplier such as UCT, Inc. The sodium sulfate should be purified by heating at 400^{o}C for four hours or extraction with methylene chloride. The purification step may be skipped as long as routine analysis of preparation blanks demonstrates the absence of contamination.
4.2.2 Sand for blanks: Used as an analyte-free, solid matrix for QC. Sand is purchased from a hardware/home improvement store and must be cleaned

before use. Place several cups of sand in a fine mesh bag. Rinse with lab grade water to remove fine particles. Bake in a foil loaf pan in the muffle furnace at 500 degrees Celsius for two hours or until the sand is dry. Allow to cool completely. Assign an Organic Standards Prep number.

4.2.3 50/50 Mix Acetone/Methylene Chloride: Pesticide residue grade or equivalent.

4.2.4 50/50 Mix Hexane/Acetone: Pesticide residue grade or equivalent. Prepared fresh daily.

4.2.5 Methylene Chloride: Pesticide residue grade or equivalent.

4.2.6 Hexane: Pesticide residue grade or equivalent.

4.2.7 Surrogate Standard: Refer to analysis method for specific analytes, concentrations, and solvent used to prepare the standard.

4.2.8 Spiking Solution: Refer to analysis method for specific analytes, concentrations, and solvent used to prepare the standard.

5.0 Method/Calibration/Interferences

5.1 Method Summary

5.1.1 An aliquot of sample is dried with sodium sulfate and transferred to an extraction thimble which is placed in a soxhlet extraction tube. The sample is then extracted in a soxhlet extractor for 16 to 24 hours. The resulting extract is concentrated for the appropriate analysis.

5.1.2 Sample Collection, Preservation and Storage: Samples are collected in jars appropriate to the method for analysis. Sample preservation and holding times are specific to the analysis method. Shipment of samples must follow the preservation requirements of the method.

5.1.3 Appropriate Solvent Systems

5.1.3.1 Soil/sediment and aqueous sludge samples shall be extracted using either of the following solvent systems:

5.1.3.1.1 Acetone/Hexane (1:1) (v/v)

5.1.3.1.2 Methylene chloride/Acetone (1:1) (v/v)

5.1.3.2 Other samples shall be extracted using the following:

5.1.3.2.1 Methylene chloride

5.1.3.2.2 Toluene/Methanol (1:1) (v/v)

5.2 Calibration Procedure

5.2.1 See analysis method/SOP for specific calibration procedures.

5.3 Interferences

5.3.1 Interferents: See analysis method/SOP for specific interferences.

6.0 Procedure

6.1 Sample Preparation

6.1.1 Homogenize the sample by mixing it with a scoopula. Measure an

adequate amount of sample to meet the required analytical detection limits into a beaker labeled with the lab ID number. A similar amount of sodium sulfate is mixed with the sample until a free-flowing homogenous sample is created.

6.1.2 An aliquot of sample should also be prepared for percent dry weight determination. Refer to QA-5834 6.2a.

6.1.3 A preparation blank, LCS and LCSD are prepared using laboratory sand and sodium sulfate. The amount of sand used for this QC is the same as used for the associated samples.

6.1.4 The soil and sodium sulfate mixture is transferred to a disposable cellulose extraction thimble which is then set in the same small beaker until the rest of the soxhlet extractor apparatus is ready to be assembled.

6.1.5 The appropriate method specific surrogate is added directly to the soil/sodium sulfate mixture inside the extraction thimble using a gas tight micro-liter syringe. Add spike to the LCS/LCSD and any MS/MSD. See applicable determinative method SOP for standard solutions to be used. The appropriate volumes of standards to add are listed in Table 1.

6.1.6 A previously cleaned and solvent rinsed Boston round bottom flask is filled with an adequate volume of the appropriate solvent to achieve the cycling as outlined in section 6.1.9 (typically 120-150 mL) and three to five Teflon boiling stones are dropped in. The appropriate lab ID is written on the flask.

6.1.7 The thimble is lowered to the bottom of a previously cleaned and solvent rinsed soxhlet extraction tube which is attached to the labeled flask. This set-up is then placed on the soxhlet extractor hot plate with a condenser tube attached to the top which is allowed to rest freely on top of the extraction tube. The condenser tubes are rinsed with solvent previously to attaching the extraction tube.

6.1.8 The attached chiller/re-circulator is turned on and allowed to cool so that the difference between the water or coolant is 20 to 25 degrees C from the boiling point of the solvent (typically 12 ^{o} C). (ex. Chiller should maintain 15 ^{o} C for methylene chloride with a boiling point of 40).

6.1.9 The hot plate is turned on and the time at which the solvent in the flask begins to boil is recorded in the notebook. Make sure that the heating controls are set at a level to produce 4-6 complete cycles per hour. Check that the ground glass connections are tight, the water or coolant level in the chiller is adequate, and there is enough solvent in the flask to cycle through the apparatus without going to dryness.

6.1.10 Sixteen to 24 hours later the hot plate is turned off and once the entire system has cooled, the chiller/re-circulator can be turned off (this time is also recorded in the notebook). A stream of methylene chloride is rinsed

through the condenser back into the collection flask.

6.1.11 The condenser is removed and the extraction tube and flask are tipped to one side to siphon the remaining solvent into the flask.

6.1.12 A plug of glass wool is added to a glass funnel and approximately ten grams of sodium sulfate is added to the funnel. This set-up is rinsed with methylene chloride and set on top of a RV tube that is labeled with the lab number ID.

6.1.13 The extract is poured through the funnel. The flask and funnel are rinsed with methylene chloride into the RV tube and the sample is concentrated to meet a volume that will achieve the desired analytical detection limits (this volume is typically 1mL or 5mL depending on the concentration of the surrogate and spike solutions used). If the initial volume of the extract is such that concentration on the standard program settings could cause the sample to splash or spill in the evaporator, the speed and nitrogen flow can be adjusted accordingly until the volume is decreased sufficiently to return to standard settings.

6.1.14 Depending on the method of analysis the extract is either ready for analysis, exchanged into a different solvent, or cleaned up by an appropriate method.

6.1.15 Before the next sample set-up, all condensers are rinsed with methylene chloride and the extraction tube and flask are washed and solvent rinsed. The extraction thimble is disposed of in the solid waste stream after drying in the hood.

6.1.16 All relevant information is recorded in the appropriate prep notebook including initial and final volumes, date, analyst initials, standard IDs etc.

7.0 Quality Control Requirements

7.1 Method Performance

7.1.1 **Initial Demonstration Requirements:** See the QA Manual or specific analysis SOP for these requirements.

7.2 **Laboratory Performance Quality Control and Corrective Actions**

7.2.1 Prep Blank: The PB is analyzed once every 24 hours. The 24 hours begin at the time the extractor begins cycling. The PB undergoes the sample preparation process. The PB must be below the reporting limit of the analyte. See analysis method for procedure taken to deal with out of control prep blanks.

7.2.2 Laboratory Control Sample (LCS): The LCS is analyzed every 24 hours. The 24 hours begin at the time the extractor begins cycling. See analysis method for particular acceptance criteria and procedure taken to deal with an out of control LCS.

7.2.3 Matrix Spike: See analysis method for particular frequency and procedure

taken to deal with a failing matrix spike. A known amount of analyte is added to an aliquot of a randomly chosen sample or a customer specified sample. The calculation of the percent recovery is as follows:

$$R = (Cs - C)/s * 100$$

R = percent recovery
Cs = Fortified sample concentration
C = Sample background concentration
s = Concentration equivalent of analyte added to the sample

7.2.4 Matrix Duplicate: See analysis method for particular frequency, relative percent difference acceptance criteria and procedure taken to deal with an out of control duplicate result. The RPD is calculated as follows:

$$RPD = (D1-D2)/((D1+D2)/2) *100$$

D1 = The initial result of the analyte
D2 = The duplicate result of the analyte

8.0 Responsibilities

8.1 The analyst uses the Quality Control Criteria outlined in this SOP to evaluate and validate the data. It is the analyst's or technician's responsibility to follow this SOP and record all the data in the lab notebook. Any irregularities within the test or samples should be recorded in the lab notebook and reported to the lab manager who is responsible for reviewing the data.

9.0 Health and Safety

9.1 Normal, accepted laboratory safety practices should be followed during reagent preparation and instrument operation. However, every sample in the lab should be handled as if it is hazardous. Refer to Chemical Hygiene Plan (SOP QA604), and SDS sheets

10.0 Pollution Prevention and Waste Management

10.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation.

10.2 The EPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Waste management in this laboratory is regulated by the Safety director. The waste generated from this

extraction is handled in accordance with local, state and federal regulations as described in the Waste Management SOP.

Title: Soxhlet Extraction by EPA Method 3540C

Table 1: Surrogate and Spike amounts for each method

Method	Surrogate	Spike
EPA8270	0.1mL	0.4mL
EPA8082	0.1mL	0.1mL
EPA8081	0.1mL	0.1mL
EPA8015	0.1mL	0.1mL

Table 2
Labconco Rapid-Vap Settings for "Program 1"

PROGRAM 1

Speed= 60%
Heat= 42° C
NO_2= 15 psi
Time= 28 minutes to start
Sample No.= Number of Rapid-Vap glassware to be concentrated at one time

PROGRAM 2

Speed=60%
Heat=62 ° C
NO2=15psi
Time= 4 minutes to start
Sample No.= Number of Rapid-Vap glassware to be concentrated at one time

ABSOLUTE RESOURCE ASSOCIATES
Standard Operating Procedure QA-5313

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Title: Extractable Petroleum Hydrocarbons –MADEP 2004-1.1

QA Officer: Jennifer Guerrette Date: 2/20/17

Laboratory Director: [signature] Date: 2/20/17

Author: Jennifer L. Lowe Date: 2/20/17

Analyst: _____ Date: _____

By signing this SOP, the analyst has read and understood the latest version of the test method; has read and understood this SOP; acknowledges receipt of this SOP; and is responsible for the implementation of this SOP.

Revision History:

Revision	Changes	Date
2	Update per current practice. Incorporate MADEP MCP QA/QC Guidance	7/03
3	Update to May 2004 Revision 1.1	7/04
4	ICV/CCV guidance criteria updated	6/06
5	Add method 3546	3/08
6	Changes to Aqueous Procedure, Silica gel Activation and Torque for tubes	3/10
7	Added Program 2 for Rapid Vap. ICV Modification. CCV acceptance criteria. Added C_{28} to C_{20} ratio and peak resolution. CAM RLs. DoD Language	12/10
8	Co Name updated, clarified curve fit and weighting selection.	8/11
9	Re-formated, Added Sand cleaning procedure, Reference to Maint Log, MDL location	7/13
10	MDL& LOQ location	4/14
11	Change CCV limits to ±25%	11/25/14
12	Added new Maine target requirements and more LCS/LCSD guidance	06/15
13	CCV acceptance criteria clarification, corrected layer separation time-AQ samples, revised RV program settings. Moved all qc requ to the qc sec & added Method Performance	3/16
14	Added tables with typical GC-FID and GCMS instrument conditions; updated suggested GC column; updated recommended calibration levels for ranges; added analysis for resolution of naphthalene and dodecane	2/17

Title: Extractable Petroleum Hydrocarbons –MADEP 2004-1.1

1. Purpose and Applicability

1.1. This procedure details the analysis of extractable petroleum hydrocarbon (EPH) concentrations in solids and waters as aliphatics and aromatics. EPH encompasses ranges C9-C18 aliphatic hydrocarbons, C19-C36 aliphatic hydrocarbons, C11-C22 aromatic hydrocarbons, and the 17 target PAH analytes. This method is suitable for analysis of waters, soils, sediments, wastes, sludges, and non-aqueous phase liquids (NAPL). The suitable products evaluated by this method include kerosene, fuel oils #2, #4, #6, diesel fuel, jet fuel, and lubricating oils. Extracts are analyzed by GC FID and GC MS. The reporting limits achievable by this method are listed in Table 1.

1.2. This method is recommended for use only by or under the supervision of analysts experienced in the use of gas chromatography and in the interpretation of the resulting chromatograms associated with EPH. Each analyst must demonstrate the ability to generate acceptable results with this method.

2. Definitions

2.1. Initial Calibration Verification (Instrument Performance Check) (ICV): A standard at a mid-point in the calibration curve to verify the integrity of the curve. The ICV is made from a second source standard. All analytes must have a different lot number than the one that made the calibration curve.

2.2. Continuing Calibration Verification (CCV): A CCV is analyzed every ten samples, at the start of a batch (if verifying a previous curve), and at the end of every batch or after 24 hours, which ever comes first. A CCV is usually a mid-point of the calibration curve. The concentration of the CCV should be varied during a run.

2.3. Calibration Standard (ICal): Standard solutions prepared from the stock standard at levels corresponding to the calibration curve. The solutions are used to calibrate the instrument response with respect to analyte concentration.

2.4. Field Duplicates: A duplicate sample taken in the field. These are only done at the customer's request.

2.5. Laboratory Control Sample (Laboratory Fortified Blank) (LCS): An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The LCS is a different source than the calibration standards, and is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

2.6. Laboratory Control Sample Duplicate (Laboratory Fortified Blank Duplicate) (LCSD): See definition above.

2.7. Prep Blank (Laboratory Reagent Blank) (PB): An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, and reagents that are used with other samples. The PB is used to determine if the method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

2.8. Linear Calibration Range (LCR): The concentration range over which the instrument response is linear and calibrated for.

2.9. Quality Control Sample (QCS): See definition for ICV.

2.10. Stock Standard Solution (SSS): A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source (e.g. Fisher Scientific or Absolute Standards). Documentation of the purity of the SSS must be retained for traceability purposes in the organic standards binder.

2.11. Matrix Spike: An aliquot of a sample spiked with a known amount of calibration standard. This is a measure of the performance of the method in a sample matrix. A matrix spike is only extracted when requested by a customer.

2.12. Matrix Spike Duplicate: See definition above.

2.13. Aliphatic Hydrocarbon Standard: A 14 component n-alkane standard from C9-C36 inclusive used to establish range retention time windows and to quantitate aliphatic hydrocarbons within that range.

2.14. Aromatic Hydrocarbon Standard: A 17 component polynuclear aromatic hydrocarbon (PAH) mixture from C11-C22 used to establish retention time windows and to quantitate aromatic hydrocarbons within that range.

2.15. Diesel PAH analytes: A subset of target PAH analytes consisting of naphthalene, 2 methyl naphthalene, phenanthrene, and acenapthene.

2.16. Fractionation Surrogate (FS): A standard consisting of 2-fluorobiphenyl and 2-bromonaphthalene in hexane that is spiked into the sample extract immediately prior to fractionation.

2.17. Surrogate Standard (SS): A standard consisting of 1-chlorooctadecane and ortho-terphenyl in acetone used for spiking all samples. Surrogate standards are 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.

2.18. Unadjusted C11-C22 Aromatic Hydrocarbons: All aromatic compounds eluting from naphthalene through benzo(g,h,i)perylene inclusive.

2.19. Adjusted C11-C22 Aromatic Hydrocarbons: All aromatic compounds eluting from naphthalene through benzo(g,h,i)perylene not including the area contributed to by the PAH target compounds.

3. Applicable Documents/References

3.1. SOP QA-400 Sample Receiving and Identification
3.2. SOP QA-402 Bottle Order Preparation
3.3. Method for the Determination of Extractable Petroleum Hydrocarbons (EPH), Massachusetts Department of Environmental Protection May 2004 Revision 1.1
3.4. EPA Method 8270C
3.5. SOP QA-5515 Preparation and analysis of PAHs, Base/Neutrals and Acids by EPA Method 8270C
3.6. SOP QA-5000 Manual Chromatographic Peak Integration Procedures
3.7. MADEP MCP QA/QC Requirements and Performance Standards
3.8. ARA Sample Readiness SOP QA801
3.9. SOP QA-5305 Soxhlet Extraction
3.10. ARA QA Manual
3.11. Current DoD Quality Systems Manual for Environmental Laboratories

4. Materials and Apparatus

4.1. Equipment:

4.1.1. Labconco Rapid-Vap and glassware
4.1.2. Microwave vessels with vent plugs and caps
4.1.3. Soxhlet extraction apparatus
4.1.4. Recirculating chiller

4.1.5. Disposable cellulose extraction thimbles
4.1.6. Glass wool
4.1.7. 200mL chromatography columns with Teflon stopcocks
4.1.8. 10-1000uL syringes
4.1.9. HP 5890 with analytical column, FID, and HP data system or equivalent
4.1.10. Suggested column: Phenomenex Zebron ZB-5, 30m, 0.5um film thickness, 0.25mmId
4.1.11. Refrigerator for storage of samples and standards
4.1.12. 1L amber glass bottles
4.1.13. 4 oz amber glass wide mouth jars
4.1.14. 1.5mL glass vials with Teflon-lined caps
4.1.15. 250mL glass jar with Teflon-lined covers
4.1.16. 1L separatory funnels with Teflon stopcock
4.1.17. 25 mL graduated cylinders
4.1.18. Glass funnels
4.1.19. Class A disposable glass pipette tips
4.1.20. 1L graduated cylinder
4.1.21. Analytical balance capable of accurately weighing 0.0001g for standards
4.1.22. Top-loading balance capable of weighing soil samples to the nearest 0.1g
4.1.23. Microwave Accelerated Reaction System, CEM MARS model

4.2. Reagents/Standards:

4.2.1. Reagent One: Pesticide grade dichloromethane or equivalent

4.2.2. Reagent Two: Pesticide grade n-hexane or equivalent

4.2.3. Reagent Three: Pesticide grade acetone or equivalent

4.2.4. Reagent Four: Sodium Sulfate: Purchased from a reputable supplier such as UCT, Inc. The sodium sulfate should be purified by heating at 400^{o}C for four hours or extraction with methylene chloride. The purification step may be skipped as long as routine analysis of preparation blanks demonstrates the absence of contamination.

4.2.5. Reagent Five: 28-200 mesh Silica Gel: Purchased from a reputable supplier such as Fisher Scientific. The silica gel is stored in a glass beaker in a drying oven to prevent absorption of moisture. Before use, the silica gel is allowed to cool in a desiccator. The silica gel should be activated @130C for at least 16 hours, and heated to 150-160C for several hours before use. The activation step may be skipped as long as routine analysis of the fractionation check standards demonstrates acceptable performance. Prepared cartridges may also be used.

4.2.6. Standard One: MADEP EPH aliphatics hydrocarbon calibration standard at 200ppm in hexane. This standard may also contain the fractionation surrogate and COD.

4.2.7. Standard Two: MADEP EPH aromatics hydrocarbon calibration standard at 200ppm in methylene chloride. This standard may also contain the fractionation surrogate and OTP.

4.2.8. Standard Three: Extraction surrogate standard containing ortho-terphenyl and chlorooctadecane at 1000ppm each in acetone.

4.2.9. Standard Four: Fractionation surrogate containing 2-fluorobiphenyl and 2-bromonaphthalene at 40 ug/mL each in hexane.

4.2.10. Sand for blanks: Used as an analyte-free solid matrix for QC. Sand is purchased from a hardware/home improvement store and must be cleaned before use. Place several cups of sand in a fine mesh bag. Rinse with lab grade water to remove fine particles. Bake in a foil loaf pan in the muffle furnace at 500 degrees Celsius for two hours or until the sand is dry. Allow to cool completely. Assign an Organic Standards Prep number.

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5. Method/Calibration/Interferences

5.1. Method Summary

5.1.1. Samples are extracted either by a separatory funnel method, soxhlet extraction method or microwave accelerated reaction method. The resulting extract is separated into two fractions by means of a silica gel fractionation. These extracts are quantitatively concentrated in a Labconco Rapid-Vap. The extracts are analyzed by GC FID to determine range concentrations and by GCMS to determine PAH target concentrations. After analysis, the analytes in the sample extract are compared to a calibration that is generated by analysis of standards.

5.1.2. See Table 1 for typical Reporting Limits.

5.2. Sample Collection, Preservation and Storage

5.2.1. Aqueous samples are collected in 1L amber glass bottles with Teflon lined screw cap. A suitable amount of 1:1HCl (5mL) to produce a sample pH of 2 or less and cooling to 4C are the necessary preservation requirements. Aqueous samples must be extracted

within 14 days of collection and analyzed within 40 days of extraction. Samples must be preserved with HCl within 2 hours of collection. Samples may also be frozen per the requirements in the MADEP method.

5.2.2. Soil and sediment samples are collected in 4 oz. amber wide mouth glass jars with Teflon lined screw caps. Preservation consists of cooling to 4C. Solid samples must be extracted within 14 days of collection and analyzed within 40 days of extraction. Solid samples may also be frozen per the requirements in the MADEP method.

5.3. Calibration Procedure

5.3.1. Aromatic and Aliphatic Hydrocarbon calibration curve standards are prepared at a minimum of five concentration levels. These levels are made by serially diluting the appropriate calibration standard into methylene chloride. One of the calibration standards must be at or below the reporting limit. The other concentrations correspond to the expected range of concentrations found in samples or as defined by the linear range of the detector. The following calibration levels are recommended: 1, 5, 10, 50, 100, and 200 ug/mL for each analyte.

5.3.2. Each calibration standard is injected at the instrument in the same way that is used to analyze samples. Once the run is complete the chromatograms are reviewed to confirm proper integration and surrogate areas are subtracted from the range areas. The resulting range areas are plotted against the assigned concentrations and used to generate a calibration curve. The analyst's discretion is used in selecting the curve fit and weighting which best represents the data, paying particular attention to the low end of the calibration range. Usually, the inverse of concentration is most appropriate weighting. If linear regression is used as the curve fit, the r^2 value must be ≥ 0.995. For curve fits utilizing average response factor, the relative standard deviation of the response factors must be $\leq 25\%$.

5.3.3. The curve is saved and ready to be verified with a second source (ICV). The suggested format for saving the curve is S1EPHmmddyyARO.M where S1 is the instrument, EPH is the analysis, mmddyy is the date the curve was run and ARO or ALI is to distinguish the compounds used for calibration.

5.4. Calibration Verification Requirements

5.4.1. An initial calibration verification (ICV) is analyzed immediately after the calibration standards to verify the calibration curve. This ICV standard must be from a different source or lot number than the calibration standards. This criterion must be met for both

aliphatic and aromatic calibration curves. If the ICV is above acceptance criteria, the curve can not be used for analysis and requires corrective action. Corrective action could include instrument maintenance, verification of standard concentrations, checking expiration dates, re-making the standard fresh, etc. Once problem is located a new 5 point calibration curve can be analyzed. For additional guidance and suggestions concerning QC failure refer to the QA Manual.

5.4.2. On a continual basis the calibration is verified with a CCV standard, which is a standard made up from the same standard source that is used for the curve. The concentration is generally a mid point in the calibration curve and should be varied throughout a run. A CCV is run at the beginning of an instrument session, after every ten samples, and at the end of an instrument session, or after 24 hours which ever comes first.

5.5. Interferences per the MADEP EPH Method

5.5.1. Method interferences are reduced by washing all glassware with hot soapy water and then rinsing with warm tap water, DI water, and methylene chloride.

5.5.2. High purity reagents are used to minimize interference problems.

5.5.3. Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. Whenever an unusually concentrated sample is analyzed, it must be followed by the analysis of a system solvent blank to check for cross-contamination.

5.5.4. Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interference will vary considerably from one source to another depending upon the nature and diversity of the site being sampled. A silica gel cleanup procedure is used to overcome many of these interferences, but some samples may require additional cleanup approaches which are beyond the scope of this method.

5.5.5. Certain organic compounds not associated with releases of petroleum products, including chlorinated hydrocarbons, phenols, and phthalate esters, could be quantitated as Extractable and Total Petroleum Hydrocarbons. If necessary and/or desirable, additional sample cleanup and/or analytical procedures may be employed to minimize or document the presence of such compounds upon request of the customer.

5.5.6. Because of their weakly polar nature, naphthalene and substituted naphthalenes are readily fractionated into the aliphatic extract if excessive amounts of hexane are used to

elute the silica gel cartridge/column. Because these compounds constitute a significant percentage of the water-soluble fraction of fuel oils, this occurrence is especially problematic in the analysis of water samples. For this reason, the method requires the use of fractionation surrogate standards with properties similar to naphthalene to monitor and document problems of this nature.

6. 6.0 Procedure

6.1. Sample Preparation

6.1.1. Aqueous Sample Extraction and Concentration

6.1.1.1. Assign a new quality control number, generated by the LIMS, to the preparation blank and the laboratory control sample and duplicate (LCS & LCSD). Record this information in the EPH Extraction Logbook. A preparation blank, LCS, and an LCSD will be extracted every 24 hours or every 20 samples, whichever is more frequent.

6.1.1.2. Set up scrupulously cleaned separatory funnels that have been washed with Alconox, rinsed with DI water, and rinsed with methylene chloride.

6.1.1.3. Mark the level of the sample on the sample container and while working in the hood, pour the sample into the separatory funnel. For the quality control samples add one liter of DI water to the associated separatory funnel. Using pH paper, verify that the pH of the sample is less than 2 and note this in the logbook. If the sample is above 2, note the deficiency in the logbook and add 1:1 HCl. Also add HCl to the quality control samples to reduce their pH to less than 2.

6.1.1.4. Using a gas-tight syringe, add 0.1 mL of the current EPH Extraction Surrogate Solution to the samples and all associated QC. Add 0.3mL each of the current EPH Aliphatic Hydrocarbon and EPH Aromatic Hydrocarbon spiking solutions to the LCS and LCSD and matrix spikes if they have been requested by the customer.

6.1.1.5. Add 40mL of methylene chloride to the sample container to extract any residue that may have adhered to the sides of the container and pour the solvent into the separatory funnel.

6.1.1.6. Shake the separatory funnel for three minutes, venting pressure often. Allow the two layers to separate for a minimum of ten minutes.

6.1.1.7. Collect the organic layer (bottom layer) into a previously washed and solvent

rinsed glass jar labeled with a piece of masking tape denoting the appropriate lab number and "eph" to distinguish the analysis. Repeat extraction two more times using 30mL of methylene chloride for the second extraction and 20mL for the final extraction.

6.1.1.8. Add sodium sulfate to the jars of solvent to remove any residual water that may remain in the extract. Cap the jars with previously cleaned and solvent rinsed Teflon lined lids and agitate the jar to thoroughly trap all the moisture in the sodium sulfate. If excessive water is present you may need to pipette off the aqueous layer before the addition of sodium sulfate.

6.1.1.9. Place a plug of glass wool into a previously cleaned and solvent rinsed glass funnel and rinse the glass wool and funnel with methylene chloride. Place the funnel atop the previously cleaned and solvent rinsed Rapid-Vap glassware that has been labeled with the appropriate lab number and "eph."

6.1.1.10. Quantitatively transfer the extract through the funnel into the Rapid-Vap glassware. Rinse the inside of the jar with methylene chloride and pour rinseate through funnel, repeat one more time.

6.1.1.11. Place glassware into Rapid-Vap and run "Program 1" (see Table 2 for settings) to concentrate the sample to approximately 2mL. Add a minimum of 10mL of hexane to the Rapid-Vap vial and swirl to mix the two solvents. Return the vial to the Rapid-Vap and concentrate to 1mL.

6.1.1.12. Remove the glassware from the Rapid-Vap and using a disposable pipette transfer the extract to a 1.5mL glass vial with a Teflon lined cap. Rinse the sides of the Rapid-Vap glassware with a small portion of hexane. Using this rinse solvent, bring the final extract up to 1mL in the vial using a newly made 1mL reference vial of hexane for comparison.

6.1.1.13. Dispose of the remaining aqueous sample from the separatory funnel into the satellite waste receiver located in the hood.

6.1.1.14. Fill the empty sample container with water up to the mark (6.1.2b) and determine the volume, to the nearest five milliliters, with a graduated cylinder. Record this value in the extraction notebook.

6.1.2. Solid Sample Extraction and Concentration by Microwave

6.1.2.1. Assign a new quality control number, generated by the LIMS, to the preparation blank and the laboratory control sample and duplicate (LCS & LCSD). Record this information in the EPH Extraction Logbook. A preparation blank, LCS, and an LCSD will be extracted every 24 hours or every 20 samples, whichever is more frequent. Homogenize the sample by mixing it with a scoopula. An adequate amount of soil to meet the required analytical detection limits is measured into a microwave vessel. Add a similar amount of sodium sulfate and mix by shaking vessel. (The typical amount of soil is 10 grams to 10 grams of sodium sulfate.) For the quality control samples (PB,LCS,LCSD) previously cleaned sand is measured into coinciding vessels as well as a similar amount of sodium sulfate.

6.1.2.2. Using a gas tight micro-liter syringe, 0.1mL of the extraction surrogate is added directly to the soil/sodium sulfate mixture inside the microwave vessel. Add 0.3mL each of the current EPH Aliphatic Hydrocarbon and EPH Aromatic Hydrocarbon spiking solutions to the LCS and LCSD and matrix spikes if they have been requested by the customer.

6.1.2.3. Add 30mL of methylene chloride to each vessel.

6.1.2.4. Place white plug and cap on each vessel. Tighten each cap making sure that no solvent can escape. The cap should be finger tight and then rotate an additional 30 degrees to tighten the cover to the proper torque range (proper torque is 1.5 ft-lbs or 18 in-lbs). Shake tube to distribute the extraction solvent.

6.1.2.5. Each vessel is placed in a composite sleeve in the microwave turntable. Make sure vessels are placed evenly around the turntable. (Note: NO LESS THAN 8 SAMPLES IN THE MICROWAVE). Place the turntable with the samples on the turntable drive lug. Close the door and select appropriate program.

6.1.2.6. Once the program has concluded. Remove the turntable from the microwave. Remove each vessel from the composite sleeves and place the vessels in a rack to cool. The vessels must be brought to room temperature before loosening the caps to avoid loosing any solvent.

6.1.2.7. A plug of glass wool is added to a glass funnel, rinse with methylene chloride and set on top of a RV tube that is labeled with the lab number ID.

6.1.2.8. The extract is poured through the funnel. Tap the soil from the vessel in to the

funnel. The sample can be poured through a small amount of sodium sulfate in the funnel if there is an excess of water. Rinse the vessel twice with methylene chloride pouring through the funnel. The funnel is also rinsed with methylene chloride into the RV tube and the sample is concentrated to approximately 2 mL. Add a minimum of 10mL of hexane to the Rapid-Vap vial and swirl to mix the two solvents. Return the vial to the Rapid-Vap and concentrate to 1mL.

6.1.2.9. Remove the glassware from the Rapid-Vap and using a disposable pipette transfer the extract to a 1.5mL glass vial with a Teflon lined cap. Rinse the sides of the Rapid-Vap glassware with a small portion of hexane. Using this rinse solvent, bring the final extract up to 1mL in the vial using a newly made 1mL reference vial of hexane for comparison.

6.1.2.10. The vessel and funnel are allowed to air dry in the hood and then washed.

6.1.3. Solid Sample Extraction and Concentration by Soxhlet

6.1.3.1. Assign a new quality control number, generated by the LIMS, to the preparation blank and the laboratory control sample and duplicate (LCS & LCSD). Record this information in the EPH Extraction Logbook. A preparation blank, LCS, and an LCSD will be extracted every 24 hours or every 20 samples, whichever is more frequent. Homogenize the sample by mixing it with a scoopula. An adequate amount of soil to meet the required analytical detection limits is measured into a 50mL beaker labeled with the lab ID number. A similar amount of sodium sulfate is mixed in until a free-flowing homogenous sample is created. (The typical amount of soil is 10 grams to 10 grams of sodium sulfate.) For the quality control samples (PB,LCS,LCSD) previously cleaned sand is measured into labeled beakers and sodium sulfate is added to each of the QC samples.

6.1.3.2. The soil and sodium sulfate mixture is transferred to a disposable cellulose extraction thimble which is then set in the same small beaker until the rest of the soxhlet extractor apparatus is ready to be assembled.

6.1.3.3. Using a gas tight micro-liter syringe, 0.1mL of the extraction surrogate is added directly to the soil/sodium sulfate mixture inside the extraction thimble. It is at this time that 0.3mL of the EPH Aromatic Hydrocarbon and the EPH Aliphatic Hydrocarbon spiking solutions is added to the LCS and LCSD and matrix spikes if they have been requested by the customer.

6.1.3.4. A round bottom flask is filled with 120-150 mL of Methylene Chloride and three to five Teflon boiling stones are added. The appropriate lab ID is written on the flask.

6.1.3.5. The thimble is lowered to the bottom of a soxhlet extraction tube which is attached to the labeled flask. This set-up is then placed on the soxhlet extractor hot plate with a condenser tube attached to the top which is allowed to rest freely on top of the extraction tube. All associated glassware has been washed with Alconox, rinsed with DI water and solvent rinsed with methylene chloride.

6.1.3.6. The attached chiller/re-circulator is turned on and allowed to cool to approximately 12 degrees Celsius.

6.1.3.7. The hot plate is turned on and the time at which the solvent in the flask begins to boil is recorded in the EPH extraction notebook. Make sure that the heating controls are set at a level to produce 4-6 complete cycles per hour. Check that the ground glass connections are tight, the water level in the chiller is adequate, and there is enough solvent in the flask to cycle through the apparatus without going to dryness.

6.1.3.8. Sixteen hours later the hot plate is turned off and once the entire system has cooled the chiller/re-circulator can be turned off. (this time is also recorded in the notebook) The condenser is rinsed with methylene chloride back into the flask.

6.1.3.9. The condenser is removed and the extraction tube and flask are tipped to one side to siphon the remaining solvent into the flask. Consult SOP QA-5305 for additional information regarding the set-up and use of the soxhlet extraction apparatus.

6.1.3.10. A plug of glass wool is added to a glass funnel and approximately ten grams of sodium sulfate is added to the funnel. This set-up is rinsed with methylene chloride and set on top of a RV tube that is labeled with the lab number ID.

6.1.3.11. The extract is poured through the funnel. The flask and funnel are rinsed with methylene chloride into the RV tube and the sample is concentrated to approximately 2 mL. Add a minimum of 10mL of hexane to the Rapid-Vap vial and swirl to mix the two solvents. Return the vial to the Rapid-Vap and concentrate to 1mL.

6.1.3.12. Remove the glassware from the Rapid-Vap and using a disposable pipette

transfer the extract to a 1.5mL glass vial with a Teflon lined cap. Rinse the sides of the Rapid-Vap glassware with a small portion of hexane. Using this rinse solvent, bring the final extract up to 1mL in the vial using a newly made 1mL reference vial of hexane for comparison.

6.1.3.13. The soil and extraction thimble are allowed to air dry in the hood and then disposed of in the solid waste disposal stream.

6.1.4. Silica Gel Fractionation

6.1.4.1. Note: All samples should be fractionated unless there are no ranges expected to be detected above the reporting limit. If samples do have reportable concentrations of ranges, they **must** be fractionated along with **all** associated quality control samples.

6.1.4.2. Prepare the column by adding a plug of glass wool into the column just above the stopcock. Rinse with hexane. Add approximately 30 mL of hexane to the column with the stopcock closed. Add 4 grams of silica gel to the bulbed top of the column which is tilted horizontally. Swirl bulb until all bubbles have escaped. Slowly tip the column vertical swirling continuously. The column will fill up with the gel.

6.1.4.3. Drain off the hexane until the solvent level is just above the silica gel. Discard the hexane into solvent waste.

6.1.4.4. Load a 1-mL gas tight syringe with 0.5 mL of the fractionation surrogate then add 0.5 mL of the sample. Add this combination to the column and follow with a 1mL syringe rinse of hexane.

6.1.4.5. Label two collection vessels per sample. One will be labeled with the lab number and “Aromatics” and the other will be labeled with the lab number and “Aliphatics.” Pre-rinse the “Aromatic” vessels with methylene chloride and the “Aliphatic” with hexane.

6.1.4.6. Run the sample on to the column until it is just above the silica gel. Pour 19 mL of hexane into the column and collect into the vessel labeled "Aliphatic". Stop the drain just above the silica gel.

6.1.4.7. Pour 30 mL of methylene chloride into the column and collect into the vessel labeled "Aromatic.” At this point it is okay for the column to run dry.

6.1.4.8. Quantitatively transfer the two extracts to the Rapid-Vap glassware, one labeled

"Aliphatic" and one labeled "Aromatics." Rinse the inside of the vessel with methylene chloride (or hexane depending on which fraction you are working with) and pour rinseate into Rapid-Vap vial, repeat one more time.

6.1.4.9. Place glassware into Rapid-Vap and run "Program 1" for the Aromatic fraction and "Program 2" for the Aliphatic fraction (see Table 2 for settings) to concentrate the sample to just below 1mL.

6.1.4.10. Remove the glassware from the Rapid-Vap and using a disposable pipette transfer the extract to a 1.5mL glass vial with a Teflon lined cap. Rinse the sides of the Rapid-Vap glassware with a small portion of the appropriate solvent, either hexane or methylene chloride. Using this rinse solvent, bring the final extract up to 1mL in the vial using a newly made 1mL reference vial of hexane or methylene chloride for comparison.

6.1.4.11. The vials are placed in the EPH tray in the organic department refrigerator for later analysis.

6.2. Instrument Setup For GC FID Range Determination:

6.2.1. The GC method "S1EPHA" or most current acquisition method is loaded into the HP3365 software (see Table 3 for typical GC FID instrument conditions). Every 24 hours, or the start of a new sequence, a continuing calibration verification sample (CCV) is analyzed to verify the current calibration curve. All analytes of interest must meet the acceptance criteria outlined in section 7.2.4, or the samples cannot be run. The CCV can be remade and re-analyzed, otherwise a new calibration curve must be prepared. See the ARA QA Manual for addition guidance concerning failing QC samples. In the opening mid-point aliphatic standard (ICV or CCV), the C28 to C20 ratio must be calculated (Section 7.2.4). This ratio must be calculated for each opening aliphatic CCV Additionally, the n-nonane (n-C_9) peak and surrogates COD and OTP must be adequately resolved from the solvent front and any individual components in the standards. Upon any major instrument changes, the resolution of naphthalene and dodecane must also be scrutinized by analyzing an unfractionated LCS sample containing both analytes.

6.2.2. Once the CCV has been verified, an instrument blank (CCB) is analyzed. The CCB is checked for contamination in which all compounds of interest must be less than the reporting limit. The CCB often contains the surrogates of interest prepared as a fresh dilution to confirm that the surrogate spiking solution has not degraded and is not contaminated.

6.2.3. Once the CCV and CCB have been evaluated a preparation blank is analyzed, if available. The PB is checked for contamination in which all PAH targets of interest must be less than the reporting limit and all ranges being reported are less than 10% of the most stringent applicable MCP standard. For projects requiring DoD protocol, refer to the QAM for additional evaluation criterion for method blanks If any compound does not meet these criteria, the samples will be re-extracted or if this is not possible refer to guidelines provided in the QA Manual.

6.2.4. If an LCS & LCSD is available, they follow the PB. The LCS & LCSD is used to evaluate the thoroughness of the extraction. All compounds that will be reported for the associated samples must be recovered within the acceptance criteria outlined in 7.2.1. For projects requiring DoD protocol, refer to the DoD Quality Systems Manual for Environmental Laboratories for required LCS/D acceptance limits. The relative percent difference between the LCS and the LCSD must meet the acceptance criteria in 7.2.1. If the LCS & LCSD does not meet the acceptance criteria the samples will be re-extracted or if this is not possible the data will be qualified accordingly, as described in the QA Manual.

6.2.5. Samples can now be analyzed and are interspersed with CCVs and CCBs every ten samples and the sequence ends with a CCV and CCB.

6.2.6. The analytical sequence is recorded in the run-log and is transcribed into the ChemStation Sequence Table

6.2.7. After the run has completed, the data files are copied and stored on an additional computer. The sample sequence is saved under the date in which it began (mmddyy).

6.2.8. The vials are then returned to trays in the organics refrigerator and placed on a shelf designated for post-run samples. They are stored there for 40 days before they are disposed of in designated waste drums.

6.3. Instrument Setup For Target Analyte Determination:

6.3.1. After fractionation of the initial extract, there is 0.5mL remaining that is analyzed by GCMS for PAH targets as per the EPA Method 8270C and the ARA SOP QA-5515. Samples analyzed for projects associated with the Maine DEP require the analysis of PAH targets to be performed from the aromatic fraction of all samples and associated quality control samples.

6.3.2. The instrument is prepared and calibrated as stated in the 8270C SOP QA-5515 (see Table 4 for typical GCMS instrument conditions). In addition to the 8270C criteria, a CCV is also run at the end of the 12 hour dfttp tuning window and must pass the acceptance criteria outlined in section 7.2.4. If the CCV does not meet these criteria the samples are either re-run or appropriately qualified. See the QA Manual for addition information.

6.4. Analysis

6.4.1. After the chromatograph of the sample has been completed, the data are evaluated by someone experienced with GC data evaluation and the EnviroQuant software.

6.4.2. Data files are quantitated using the appropriate data analysis method prepared from the most recent calibration. Using the Qedit function, each analyte is reviewed for qualitative and quantitative accuracy using retention time and integration settings. Surrogate recoveries are verified to be acceptable. In the case of failure a sample may be reanalyzed. If there is not enough sample for re-extraction, the data are qualified with an appropriate qualifier. A quant report is printed for each sample.

6.4.3. The aliphatic fractions of the LCS & LCSD are also evaluated using the AROMATIC calibration curve to evaluate naphthalene and 2-methylnaphthalene break through that may have occurred during the fractionation process. The raw areas of each peak are compared to the area seen in the aromatic fraction of the LCS & LCSD. See section 7.2.1 for acceptable breakthrough limits. If the criteria are not met, re-fractionation may be required or appropriate qualification of the data if re-fractionation is not possible.

6.4.4. All three instrument runs produce separate quantitation reports that are attached together and used to generate the final report that will be sent to the customer. The quant reports are kept in a project specific folder.

6.5. Sample Calculations

6.5.1. As an external standard method, response factors are determined by:

$$RF = \frac{(A_C)}{(C_C)}$$

where,
A_C = Area of compound of interest
C_C = Concentration of analyte of interest

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RF = Response Factor

6.5.2. When average response factor is used, the RF is determined for each calibration level. The response factors are averaged. This average is used to determine the sample concentration:

$$C_C = \frac{(A_C)}{(\text{avgRF})}$$

where,
A_C = Area of compound of interest
C_C = Concentration of analyte of interest
avgRF = average relative Response Factor

6.5.3. In cases where a curve fit is employed, a plot of concentration v. RF is performed using the calibration standard data. Using typical statistical calculations (method of least squares) a curve fit is determined. The equation of this curve is used to determine the sample concentrations.

6.5.4. In all cases, the Agilent EnviroQuant software is employed to perform the above calculations.

6.5.5. The generated quantitation report shows results in ug/mL units, which need to be converted to ug/L to be reported for aqueous samples and ug/g dry wt. to be reported for solid samples. The follow equations are used to convert the data:

6.5.6. To determine the reported result:

6.5.7. Aqueous Samples:

$$\text{Concentration (ug/L)} = \frac{\text{quant sheet concentration(ug/mL)} * \text{extract volume (mL)}}{\text{Amount fractionated (mL)} * \text{Volume Sample Extracted (L)}}$$

6.5.8. Solid Samples:

$$\text{Concentration (ug/g)} = \frac{\text{quant sheet concentration(ug/mL)} * \text{extract volume (mL)}}{\text{Amount fractionated (mL)} * \text{Amount sample extracted (g)} * \text{dry fraction}}$$

7. Quality Control Requirements

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7.1. Method Performance

7.1.1. Initial Demonstration of Performance: An initial demonstration of capability is performed to demonstrate method and analyst performance. The procedure includes the analysis of (4) LCS samples. Acceptable recovery of the LCS samples must be achieved prior to use of this method. See ARA QA Manual for more details. The analytes of interest should be spiked at approximately 50ug/L for water, and 5 mg/kg for solids. For each analyte, the mean accuracy should be 40-140%. For each analyte the %RSD must be less than or equal to 25%. Evaluate the Fractionation Efficiency of (4) of these LCS to meet the IDC requirement for the Factionation Check Solution.

7.1.2. Method Detection Limits: MDLs are not required by the MADEP EPH Method, but may be performed if required by other regulatory bodies. The MDL data are stored electronically, by method, in the QA/MDL file folder. The reporting limit is established at the lowest standard in the curve. Initial demonstration is performed per the method.

7.1.3. LODs/LOQs: Refer to the QAM *Analytical Procedures* section for specific LOD/LOQ requirements. LOD/LOQ data are stored electronically in the QA/LOD&LOQ folder.

7.1.4. IDC/CDC: Proficiency of the analyst is ensured by documentation of an Initial or Continuing Demonstration of Capability (IDC or CDC). Typically, the analyst analyzes LCS's (see method requirement) with acceptable precision and accuracy or shows acceptable performance of a blind sample analysis (usually a PT). See QAM and method for additional information.

7.1.5. Control Charts: Control Charts are quality control tools which graphically display QC data over time. The data required for generation of accuracy and precision control charts is maintained by the laboratory. See QAM for additional information.

7.1.6. Inter-laboratory performance: Proficiency Test studies, conducted by approved providers, constitute the majority of inter-laboratory performance comparisons. Proficiency tests are performed at a frequency as required by method, regulation, or accrediting body. See QAM for additional information.

7.2. Ongoing Method QC Demonstrations and Corrective Actions

7.2.1. Laboratory Control Samples(LCS)/LCSD: LCS or Lab fortified blank and LCS Duplicate is prepared one per 20 samples or one per day, whichever is more frequent.

The acceptable spike recovery range is 40-140% for waters and solids. The relative percent difference between the LCS and the LCSD should be less than 25% for the ranges. For projects requiring DoD protocol, refer to the DoD Quality Systems Manual for Environmental Laboratories for required LCS/D acceptance limits. If spike recoveries are not within this range, all samples associated with the batch are considered suspect. The associated samples must be qualified accordingly on the report or whenever possible, re-extract and re-analyze the samples. The laboratory control samples are also analyzed for breakthrough of naphthalene and 2-methylnaphthalene into the aliphatic fractions during sample fractionation. Naphthalene and 2-methylnaphthalene recoveries in the aliphatic fraction should not exceed 5% of the quantities found in the aromatic fraction.

7.2.2. Surrogates: The compound chloro-octadecane is used to evaluate the extraction of the aliphatic portion. The compounds 2-fluorobiphenyl and 2-bromonapthalene are used as fractionation separation surrogate recoveries quantitated in the aromatic portion. O-terphenyl is the extraction surrogate for the aromatic portions. Acceptance recoveries for the sample surrogates are 40-140%. For projects requiring DoD protocol, refer to the DoD Quality Systems Manual for Environmental Laboratories for required surrogate acceptance limits. Acceptable recoveries for the fractionation surrogates are 40-140%. For projects requiring DoD protocol, refer to the DoD Quality Systems Manual for Environmental Laboratories for required surrogate acceptance limits. Samples with unacceptable surrogate recoveries should be re-extracted or re-fractionated, if sample is available, unless obvious chromatographic interferences exist. See method for guidance on reporting samples with failing surrogate values. Any sample data associated with a failing surrogate is flagged in the data report.

7.2.3. Prep Blanks: For every 20 samples or once every 24 hours whichever is more frequent, a method blank is performed. The blank must be less than the reporting limit for any applicable PAH target and less than 10% of the most stringent applicable MCP standard for all ranges being reported. For projects requiring DoD protocol, refer to the QAM for additional evaluation criterion for method blanks. If there is contamination in the blank, the source of the contamination must be determined and the impact on the sample data evaluated. If the problem cannot be resolved or the samples re-extracted, the associated sample data will be qualified in the report.

7.2.4. Initial/Continuing Calibration Verification: The CCV must be within 25% recovery for all ranges and FID analytes and 20% for GC/MS targets. The ICV percent recovery for all analytes must be within 20% of the true value. If the CCV performed at the beginning of a run (in order to verify a curve) is out of control, either remake and re-analyze the CCV. If it passes the batch can be continued, or build a new calibration

curve. If the CCV performed after 10 samples is out of control stop running. Locate the source of the problem and re-analyze the CCV. If the CCV is not within the acceptance limits, re-analyze all the samples run after the last passing CCV. For target analysis, the closing CCV may have up to four compounds outside these criteria, but not more than 40%. Throughout the sequence, aliphatic and aromatic verification standards must also be analyzed for mass discrimination across the aliphatic range. The following explains these criteria in the EPH method: "In order to demonstrate the absence of aliphatic mass discrimination, the response ratio of C_{28} to C_{20} must be at least 0.85. If <0.85, this nonconformance must be noted in the laboratory case narrative. The chromatograms of Continuing Calibration Standards for aromatics must be reviewed to ensure that there are no obvious signs of mass discrimination." Alternatively, the instrument related issue can be resolved and samples can be rerun.

7.2.5. Continuing Calibration Blank: The CCB is analyzed at the same frequency as the CCV. The CCB must be below the reporting limit for the analyte. If the CCB is not below the reporting limit steps must be taken to determine the source of the problem. Corrective actions may include re-analysis of the CCB with fresh reagent water to trace the contamination. If there is suspected data impact (discuss with the QAO), the samples may be reanalyzed or the data qualified to disclose the suspected impact to the customer.

7.2.6. Fractionation Check Solution: To demonstrate proper performance of the fractionation step the LCS and LCSD are monitored to ensure that fractionation is complete and appropriate. During the fractionation check evaluation of the LCS & LCSD each analyte must have a percent recovery of 40-140%. When a new lot of silica gel is received at the lab an LCS & LCSD are prepared and fractionated to ensure proper separation before samples are analyzed.

7.2.7. Matrix Spike & Matrix Spike Duplicate: Performed only when requested by the customer. A known amount of analyte is added to an aliquot of a randomly chosen sample. The calculation of the percent recovery is as follows:

$$R = (Cs - C)/s * 100$$

R = percent recovery
Cs = Fortified sample concentration
C = Sample background concentration
s = Concentration equivalent of analyte added to the sample

The percent recovery must be 40-140%. The RPD criteria for the MSD is +/- 50%. If

recovery limits are not met and the LCS is in control, no further action necessary. Note in report.

7.2.8. Matrix Duplicate: Performed only when requested by the customer. The relative percent difference must be ±50%. If RPD fails, check calculations, re-extract as needed to confirm. Note in report. The RPD is calculated as follows:

RPD = (D1-D2)/((D1+D2)/2) *100

D1 = The initial result of the analyte
D2 = The duplicate result of the analyte

7.3. Retention Times

7.3.1. C9-C18 aliphatic hydrocarbon retention time window starts 0.1 min before n-Nonane and ends 0.1 min before n-Nonadecane

7.3.2. C19-C36 aliphatic hydrocarbon retention time window starts 0.1 min before n-Nonadecane and ends 0.1 min after N-Hexatriacontane

7.3.3. C11-C22 aromatic hydrocarbon retention time window starts 0.1 min before Naphthalene and ends 0.1 min after Benzo(g,h,i)Perylene

8. Responsibilities

8.1. The analyst uses the Quality Control Criteria outlined in this SOP to evaluate and validate the data. It is the analyst's or technician's responsibility is to follow this SOP and record all the data in the lab notebook. Any irregularities within the test or samples should be recorded in the lab notebook and reported to the lab manager who is responsible for reviewing the data.

9. Health and Safety

9.1. Normal, accepted laboratory safety practices should be followed during reagent preparation and instrument operation. All technicians shall be familiar with the Chemical Hygiene Plan. Refer to Chemical Hygiene Plan (SOP QA604) and SDS sheets.

10. Pollution Prevention and Waste Management

10.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Where possible, reduced sample and solvent volumes are used to minimize waste.

10.2. The EPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Waste management in this laboratory is regulated by the Safety director. The water sample extract waste, extracts, and solid residue is segregated into a waste streams and handled by a contractor in accordance with regulations.

Title: Extractable Petroleum Hydrocarbons –MADEP 2004-1.1

Table 1

Typical/CAM Required Reporting Limits

	Water(ug/L)	Solid (ug/g)
Naphthalene	0.5-1.0	0.1-0.2
2-Methylnaphthalene	0.5-1.0	0.1-0.2
Acenaphthylene	0.5-1.0	0.1-0.2
Acenaphthene	0.5-1.0	0.1-0.2
Fluorene	0.5-1.0	0.1-0.2
Phenanthrene	0.5-1.0	0.1-0.2
Anthracene	0.5-1.0	0.1-0.2
Fluoranthene	0.5-1.0	0.1-0.2
Pyrene	0.5-1.0	0.1-0.2
Benzo(a)Anthracene	0.5-1.0	0.1-0.2
Chrysene	0.5-1.0	0.1-0.2
Benzo(b)Fluoranthene	0.5-1.0	0.1-0.2
Benzo(k)Fluoranthene	0.5-1.0	0.1-0.2
Benzo(a)Pyrene	0.2-0.4	0.1-0.2
Indeno(123 cd)Pyrene	0.5-1.0	0.1-0.2
Dibenzo(ah)Anthracene	0.5-1.0	0.1-0.2
Benzo(ghi)Perylene	0.5-1.0	0.1-0.2
C9-18 Aliphatic Hydrocarbons	200/100	100/20
C19-C36 Aliphatic Hydrocarbons	200/100	100/20
C11-C22 Aromatic Hydrocarbons	200/100	100/20

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Table 2
Labconco Rapid-Vap Settings for "Program 1"

PROGRAM 1

Speed= 55%
Heat= 42º C
NO_2= 15 psi
Time= 12 minutes to start
Sample No.= Number of Rapid-Vap glassware to be concentrated at one time

PROGRAM 2

Speed=55%
Heat=62 º C
NO2=15psi
Time= 10 minutes to start
Sample No.= Number of Rapid-Vap glassware to be concentrated at one time

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Table 3: Typical GC FID Instrument Conditions (subject to change)

INSTRUMENT CONTROL PARAMETERS

Sample Inlet: GC
Injection Source: GC ALS

7673 Injector

Front Injector:

Sample Washes	1
Sample Pumps	3
Injection Volume	2.0 microliters
Syringe Size	10.0 microliters
On Column	Off
Nanoliter Adapter	Off
PostInj Solvent A Washes	3
PostInj Solvent B Washes	0
Viscosity Delay	1 seconds
Plunger Speed	Fast

Back Injector:

No parameters specified

HP5890 Temperature Parameters

Zone Temperatures:	State	Setpoint
Inlet A:	On	285 C
Inlet B:	Off	250 C
Detector A:	On	320 C
Detector B:	Off	280 C
Auxiliary:	Off	50 C

Oven Parameters:

Oven Equib Time:	0.00 minutes
Oven Max:	450 C
Oven State:	On
Cryo State:	Off
Cryo Blast:	Off
Ambient:	25 C

Title: Extractable Petroleum Hydrocarbons –MADEP 2004-1.1

Oven Program:

Initial Temperature: 40 C
Initial Time: 1.00 minutes

Level	Rate (C/minute)	Final Temperature (C)	Final Time (minutes)
1	50.0	140	0.00
2(A)	5.0	165	0.00
3(B)	30.0	340	9.50

Next Run Time: 23.33 minutes

HP5890 Inlet Temperature Programs

Inlet A:

Oven Tracking: Off
Inlet Zone: On
Initial Temperature: 285 C
Initial Time: 650.00 minutes

Level	Rate (C/minute)	Final Temperature (C)	Final Time (minutes)
1	0	50	0.00
2(A)	0	50	0.00
3(B)	0	50	0.00

Total Program Time: 650.00 minutes

Inlet B:

Oven Tracking: Off
Inlet Zone: Off
Initial Temperature: 250 C
Initial Time: 480.00 minutes

Level	Rate (C/minute)	Final Temperature (C)	Final Time (minutes)
1	0	50	0.00
2(A)	0	50	0.00
3(B)	0	50	0.00

Total Program Time: 480.00 minutes

Title: Extractable Petroleum Hydrocarbons –MADEP 2004-1.1

HP5890 Inlet Pressure Programs

GC Pressure Units: psi

Inlet A:

Constant Flow: Off
Constant Flow Pressure: 0.0 psi
Constant Flow Temperature: 50 C
Initial Pressure: 0.0 psi
Initial Time: 650.00 minutes

Level	Rate (psi/minute)	Final Pressure (psi)	Final Time (minutes)
1	0.00	0.0	0.00
2(A)	0.00	0.0	0.00
3(B)	0.00	0.0	0.00

Total Program Time: 650.00 minutes

Column Length: 30.00 m
Column Diameter: 0.530 mm
Gas: He
Vacuum Compensation: Off

Inlet B:

Constant Flow: On
Constant Flow Pressure: 5.8 psi
Constant Flow Temperature: 40 C
Initial Pressure: 25.0 psi
Initial Time: 480.00 minutes

Level	Rate (psi/minute)	Final Pressure (psi)	Final Time (minutes)
1	0.00	0.0	0.00
2(A)	0.00	0.0	0.00
3(B)	0.00	0.0	0.00

Total Program Time: 480.00 minutes

Column Length: 30.00 m

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Column Diameter: 0.250 mm
Gas: He
Vacuum Compensation: Off

HP5890 Detector Information

Detector	Type	State
A	FID	On
B	ECD	Off

HP5890 Signal Information

Save data for signal 1 only.

Signal	Source	Peak Width	Data Rate	Start Data	Stop Data
1	Det A	0.013	20.000	0.00	1.00
2	Testplot	0.053	5.000	0.00	1.00

Title: Extractable Petroleum Hydrocarbons –MADEP 2004-1.1

Table 4: Typical GCMS Instrument Conditions (subject to change)

Sample Inlet: GC
Injection Source: GC ALS
Mass Spectrometer: Enabled

7673 Injector

Front Injector:
No parameters specified

Back Injector:

Sample Washes	1
Sample Pumps	4
Injection Volume	1.0 microliters
Syringe Size	10.0 microliters
On Column	Off
Nanoliter Adapter	Off
PostInj Solvent A Washes	4
PostInj Solvent B Washes	0
Viscosity Delay	1 seconds
Plunger Speed	Fast

HP5890 Temperature Parameters

Zone Temperatures:	State	Setpoint
Inlet A:	Off	50 C
Inlet B:	On	250 C
Detector A:	Off	50 C
Detector B:	On	280 C
Auxiliary:	Off	50 C

Oven Parameters:

Oven Equib Time:	0.01 minutes
Oven Max:	340 C
Oven State:	On
Cryo State:	Off
Cryo Blast:	Off
Ambient:	25 C

Title: Extractable Petroleum Hydrocarbons –MADEP 2004-1.1

Oven Program:
Initial Temperature: 50 C
Initial Time: 1.00 minutes

Level	Rate (C/minute)	Final Temperature (C)	Final Time (minutes)
1	15.0	275	0.00
2(A)	50.0	310	5.00
3(B)	70.0	330	8.10

Next Run Time: 30.09 minutes

HP5890 Purge Valve Settings

Inlet Purge	Init Value	On Time	Off Time	Splitless Injection
A	Off	0.75	40.00	Yes
B	Off	0.75	40.00	Yes

HP5890 Valve and Relay Information

Initial Setpoints:
5890 Valves:
Valve 1: Off Valve 2: Off Valve 3: Off Valve 4: Off
19405 Valves:
Valve 5: Off Valve 6: Off Valve 7: Off Valve 8: Off
19405 Relays:
Relay 1: Off Relay 2: Off Relay 3: Off Relay 4: Off

HP5890 Detector Information

Detector	Type	State
A	---	Off
B	---	Off

HP5890 Signal Information

Not saving signal data.

Title: Extractable Petroleum Hydrocarbons –MADEP 2004-1.1

Signal	Source	Peak Width	Data Rate	Start Data	Stop Data
1	Testplot	0.053	5.000	0.00	1.00
2	Testplot	0.053	5.000	0.00	1.00

MS ACQUISITION PARAMETER

General Information
------- -----------
Tune File: dftpp.u
Acquistion Mode: Scan

MS Information
-- -----------
Solvent Delay: 3.25 min
EM Absolute: False
EM Offset: 0
Resulting EM Voltage: 2105.9

[Scan Parameters]
Low Mass: 40
High Mass: 450
Threshold: 10
Sample #: 2 A/D Samples 4

Title: **Preparation and analysis of PAHs, Base/Neutrals and Acids by EPA Method 8270D**

QA Officer: Jennifer Guenette Date: 8/3/17

Laboratory Director: Date: 8/3/17

Author: Date: 8/3/17

Analyst: Date:

By signing this SOP, the analyst has read and understood the latest version of the test method; has read and understood this SOP; acknowledges receipt of this SOP; and is responsible for the implementation of this SOP.

Revision History:

Revision	Changes	Date
1	Method prep, MDL and QA/QC updates	4/04
2	Added QC Prep information, changed final volume amount for PAH aqueous	8/07
3	Added method 3546, added solid QLs, CCV changes for update to 8270D	2/08
4	Added Changes to Acid prep for ABN	5/09
5	DoD Language	12/10
6	Solvent mixture for 3546 extraction updated and extraction procedures moved to separate SOPs, Co name updated, clarified curve fit weighting	8/11
7	System degradation updates, MDL/LOQ/LOD location, formating	3/14
8	DOD QSM 5.0 modifications	2/15
9	Revised RV program settings, revised CEM microwave settings, added Method performance sec 7.1	3/16
10	bis(2-chloroisopropyl)ether name change; added table with typical GCMS instrument conditions	2/17
11	Update tuning requirements sec6.2.2	7/17

Title: Method 8270D – PAHs, Base/Neutrals and Acids

Preparation and analysis of PAHs, Base/Neutrals and Acids by EPA Method 8270D

1. Purpose and Applicability

1.1. This method covers the determination of a number of organic compounds that are partitioned into an organic solvent and are amenable to gas chromatography. The parameters listed in Tables 1, 2, and 3 may be qualitatively and quantitatively determined using this method. The compounds that are commonly reported are based on historical requests of clients and their needs.

1.2. This is a gas chromatographic/mass spectrometry (GC/MS) method applicable to the determination of the compounds listed in Tables 1 and 2 in municipal and industrial aqueous discharges, groundwater and solids.

1.3. This method is for use only by or under the supervision of analysts experienced in the use of gas chromatograph/mass spectrometer and in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method.

2. Definitions

2.1. Initial Calibration Verification (Instrument Performance Check) (ICV): A standard at a mid-point in the calibration curve to verify the integrity of the curve. The ICV is made from a second source standard. All analytes must have a different lot number than the one that made the calibration curve. Acceptable recovery for the ICV is ± 30% of the expected concentration.

2.2. Continuing Calibration Verification (CCV): A CCV is analyzed every 12 hours and at the start of a batch (if verifying a previous curve). A CCV is a standard at a mid-point of the calibration curve. The concentration of the CCV is varied periodically in order to reveal any concentration specific biases.

2.3. Calibration Standard (ICal): Standard solutions prepared from the stock standard at levels corresponding to the calibration curve. These are used to calibrate the instrument

response with respect to analyte concentration.

2.4. Field Duplicates: A duplicate sample taken in the field. These are done at the request of a customer.

2.5. Laboratory Control Sample (Laboratory Fortified Blank) (LCS): An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly as a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate measurements. The acceptance criterion for recovery varies among compounds. The acceptance limits for the Acids and Base/Neutrals catagories can be found in Table 4.

2.6. Prep Blank (Laboratory Reagent Blank) (PB): An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, and reagents that are used with field samples. The PB is used to determine if the method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. The analytes found in the PB must be below the reporting limit for each analyte. For projects requiring DoD protocol, refer to the QAM for additional evaluation criterion for method blanks.

2.7. Linear Calibration Range (LCR): The concentration range over which the instrument response is linear and calibrated.

2.8. Quality Control Sample (QCS): See definition for ICV.

2.9. Stock Standard Solution (SSS): A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source, i.e. Absolute Standards or Ultra Scientific. Documentation of the purity of the SSS are retained for traceability purposes in the organic standards binder.

2.10. Matrix Spike: An aliquot of a sample spiked with a known amount of a standard.

2.11. Surrogate Solution: A solution containing a known concentration of compounds that are not found in environmental samples, but behave similar to compounds of interest. Surrogates are added to all field and QC samples to determine the thoroughness of the

extraction. See Table 8.

2.12. Internal Standard Solution: Solution of known concentration of compounds that are not found in environmental samples which is spiked into all extracts prior to analysis. The internal standards are used to correct for variability in injection volume in the quantitation of analytes in the sample extract.

2.13. DFTPP Tune: 2uL of a 50ug/mL solution of deca-fluoro-tri-phenyl-phosphine is analyzed prior to sample analysis, to observe its mass spectrum. The ion abundances must meet the acceptance criteria as shown in Table 5. The DFTPP tune is valid for 12 hours.

2.14. Degradation Standard: A standard that contains DDT, Benzidine, and Pentachlorophenol to evaluate the inertness of the injection port, liner, and system. This combination of analytes may be combined with the DFTPP tuning solution.

3. Applicable Documents/References

3.1. 40 CFR 136, Appendix A, Method 625
3.2. Test Methods for Evaluation Solid Waste, Physical/Chemical Methods, USEPA SW 846, 3rd Edition, 1986, Method 8270D
3.3. US EPA Contract Laboratory Program, Statement of Work for Organic Analysis, 2/88 and 3/91
3.4. Methods for Chemical Analysis of Water and Wastes, US EPA 600 – 4/79/020, 1979 revised 1983, Method 625
3.5. ARA SOP QA-400 Sample Receiving and Identification
3.6. ARA QA Manual
3.7. ARA Manual Integration SOP QA5000
3.8. MADEP MCP Guidance, CAM, July 2010
3.9. ARA SOP# 5524 for extraction procedure
3.10. ARA SOP# 5520 for extraction procedure
3.11. ARA SOP# 5522 for extraction procedure
3.12. ARA SOP# 5520 for extraction procedure
3.13. Current DoD Quality Systems Manual for Environmental Laboratories

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4. Materials and Apparatus

4.1. Equipment:

4.1.1. Hamilton syringes (or equivalent) in 250, 500, and 1000 uL sizes
4.1.2. 1.5 mL screw cap vials with Teflon-faced silicon septa
4.1.3. Glass Wool
4.1.4. Hewlett Packard Series II 5890 GC
4.1.5. Hewlett Packard 5972 Series Mass Selective Detector
4.1.6. Autosampler

4.2. Reagents/Standards:

4.2.1. Methylene Chloride: HPLC grade

4.2.2. SVOC Calibration Standards: Purchased from Absolute Standards (or equivalent) and containing all compounds of interest at a concentration of 2000ug/mL. See Table 1 & 2 for constituents.

4.2.3. ABN Surrogate Solution: Purchased from Environmental Consulting Services (or equivalent) See Table 8 for constituents. The surrogate is diluted into acetone.

4.2.4. PAH Surrogate Solution: Purchased from ChemServe (or equivalent) and prepped in acetone. See Table 8 for constituents.

4.2.5. Internal Standard: Purchased from Environmental Consulting Services (or equivalent) at a concentration of 4000ug/mL. See Table 9 for constituents.

4.2.6. Sodium Sulfate: (granular)

4.2.7. Sodium hydroxide: 10M solution prepared from sodium hydroxide pellets.

4.2.8. Sulfuric acid: 1:1 solution prepared from stock acid.

4.2.9. SVOC Spiking Solution: Purchased from Environmental Consulting Services (or equivalent) This contains the full list of target compounds.

4.2.10. De-ionized Water: Reagent grade purified/Deionized water

4.2.11. PAH Spiking Solution: (polycyclic aromatic hydrocarbons) Compounds whose structure contains multiple fused carbon rings.

5. Method/Calibration/Interferences

5.1. Method Summary

5.1.1. Samples are extracted either by a separatory funnel, microwave, or probe sonication. These extracts are quantitatively concentrated in a Labconco Rapid-Vap. Internal Standard is added prior to GCMS analysis. After GCMS analysis, the analytes in the sample extract are compared to a calibration that is generated by analysis of standards.

5.1.2. Sample Collection, Preservation and Storage

5.1.2.1. Aqueous samples are collected in previously cleaned 1000mL amber glass bottles and solid samples are collected in 4oz amber glass jars. Both matrices are stored on ice at 4±2˚C. See SOP for sample storage procedures.

5.1.2.2. Samples are unpreserved and extracted within 7 days from the sampling date for waters and 14 days for solids.

5.1.2.3. Shipment of samples must follow the requirements of the method and SOP.

5.2. Calibration Procedure

5.2.1. The SSS is made using the compounds of interest purchased from Absolute

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Standards at a concentration of 2000ug/mL. Upon receipt the standards are logged into the organic standards logbook where they are given a unique identifying code. The organic standards logbook contains the supplier information such as lot number, date received, concentration, etc. The standards are stored in the organic extract refrigerator between 2 and 6 degrees Celsius. The purchased standards are allowed to come to room temperature, shaken, and diluted by ten times to create a 200 ppm SSS for calibration. The SSS is then serially diluted to create the standards necessary to create a calibration curve. Each dilution is spiked with 10uL of a 4000ug/mL internal standard stock solution such that the final concentration of internal standard in the extract is 40ug/mL.

5.2.2. (5.2a) Suggested standard CAL levels are:

Concentration Level (ug/mL)	Vol. Stock Standard (mL)	Vol. MeCl2 (mL)	Dilution
0.1	0.5 of 0.2 ppm std	0.5	2
0.2	0.1 of 2 ppm std	0.9	10
0.5	0.1 of 5 ppm std	0.9	10
2	0.1 of 20 ppm std	0.9	10
5	0.1 of 50 ppm std	0.9	10
10	0.1 of 100 ppm std	0.9	10
20	0.1 of SSS	0.9	10
50	0.2 of SSS	0.8	4
100	0.5 of SSS	0.5	2
200	1.0 of SSS	0.0	0

The reporting limits vary for each compound depending on each analyte's chemical properties and regulatory needs. The reporting limit for each of the compounds can be found in Table 7.

5.2.3. Calibration Verification Requirements

5.2.3.1. The analyst's discretion is used in selecting the curve fit and weighting which best represents the data, paying particular attention to the low end of the calibration range. Usually, the inverse of concentration is most appropriate

weighting. If linear fit is to be used to create the curve then the calibration coefficient needs to be 0.99 or greater. If average of response factors is used to create the curve, then the RSD of the response factors must not exceed 20%. If these approaches fail to be adequate, a quadratic fit may be used if the curve is not too visually skewed. If quadratic line fit is to be used all integrations in all levels of the curve for that particular compound should be checked for errors. An ICV must be analyzed following the calibration prior to the analysis of samples. The ICV must show recovery of ± 20% the expected concentration. These requirements must be met before using a new calibration curve.

5.3. Interferences

5.3.1. Interferant 1: Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/ or elevated baselines in the chromatogram. The preparation blank is utilized to detect such contamination. If contamination is found, its source is found and is replaced with uncontaminated materials.

5.3.2. Interferant 2: Glassware must be scrupulously cleaned and rinsed with appropriate solvent prior to the extraction to minimize contamination.

6. Procedure

6.1. Sample Preparation

6.1.1. A batch is defined as a 24 hour window in which up to 20 samples can be prepped. Every batch must be accompanied by a preparation blank and a laboratory control sample. Every tenth sample should be matrix spiked and every twentieth sample should be done in duplicate and matrix spiked if sample volume exists. If sample volume does not exist a duplicate LCS can be substituted.

6.1.2. Aqueous PAH, ABN, Acid, or BN Sample Preparation

6.1.2.1. See ARA SOP# 5524 for extraction procedure.

6.1.3. Solid ABN Sample Preparation by Sonication

6.1.3.1. See ARA SOP# 5520 for extraction procedure.

6.1.4. Solid ABN Extraction and Concentration by Microwave

6.1.4.1. See ARA SOP# 5522 for extraction procedure.

6.1.5. Solid PAH Sample Preparation

6.1.5.1. See ARA SOP# 5520 for extraction procedure.

6.2 Instrument Setup

6.2.1 See Table 11 for typical GC MS instrument conditions.

6.2.2 Tuning

6.2.2.1 Per the manufacturer's instructions, instrument parameters are set to achieve the method requirements for tuning as outlined in Table 5. These parameters are reviewed and optimized after instrument maintenance and prior to calibration.

6.2.2.2 Before analysis begins, a dftpp tune is analyzed to establish a valid tune window. The tuning standard is at a concentration of 50ug/mL. It is prepared by taking 1 ml of a CLP Semi-Volatile tuning standard at a concentration of 500ug/mL and bringing it to a final volume of 10mls with MeCl2. The ion abundances are compared to the method requirements (see Table 5). These criteria must be met before samples may be analyzed. The dftpp should be evaluated using "autofind". If the resulting scans are not acceptable, a manual evaluation of an average peak spectrum, including the apex, is performed. The mass spectrum of the dftpp should be acquired by selecting the apex of the peak, the scan before the apex and scan after the apex. A background subtraction, using a single scan within 20 scans of the dftpp, is required to remove interfering ions. The tune is valid for 12 hours.

6.2.2.3 Passing scan results for the tune are saved as TUNEVAL.TXT in the sample's directory. If the tune does not pass, the DFTPP solution is re-injected. If it still fails the criteria, the mass spectrometer is re-tuned and DFTPP is reanalyzed until all criteria are met.

For full list ABN analysis, degradation of DDT to DDE and DDD is also evaluated in the injection of this standard. DDT breakdown should not exceed 20%. Tailing factors for benzidine and pentachlorophenol should be evaluated by the following formula and not exceed a tailing factor of 2. Tailing factor = BC/AB
Where: AC is the width at 10% height
DE is the height of the peak
B is the height at 10% of DE

6.2.3 All samples, standards and quality control samples are spiked with an internal standard solution at 4000ug/mL. The samples are spiked with a volume of internal standard equal to 1/100 of the extracts final volume resulting in a concentration of 40ug/mL in the extract. i.e. a 1mL extract is spiked with 10uL internal standard solution.

6.2.4 Every 12 hours, or the start of a new sequence, a continuing calibration verification sample (CCV) is analyzed to verify the current calibration curve. The percent recovery for the target analytes should be within 20% of their true value. Up to 20% of the full-list of analytes may exhibit percent recoveries outside of this criterion. If only PAH analytes are to be reported, then all PAH compounds must be within 20% of the true value. If the compounds are not within 20%, the CCV can be remade and reanalyzed, otherwise a new calibration curve must be prepared. If some compounds exhibit percent recoveries beyond the 20%, samples can still be analyzed as long as those particular compounds are not detected in the samples. For projects requiring DoD protocol, all compounds of interest in the CCV must be recovered between 80% and 120%. Project level approval would be needed for any analytes that do not meet this criterion. For projects requiring DoD protocol, a CCV is also analyzed at the end of the 12 hour window. All compounds of interest in the ending CCV must be recovered between 50% and 150%.

6.2.5 Once the tune and CCV have been verified a PB is analyzed, if available. The PB is checked for contamination in which all compounds of interest must be less than the reporting limit. If any compound is above the reporting limit the samples will be re-extracted or if this is not possible refer to guidelines provided in the QA Manual.

6.2.6 If an LCS is available, it follows the PB. The LCS is used to evaluate the thoroughness of the extraction. All compounds of interest should be within the acceptance criteria as seen in Table 4. If the LCS does not meet the acceptance criteria the samples will be re-extracted or if this is not possible the data will be qualified accordingly, as described in the QA Manual.

6.2.7 Samples can now be analyzed as long as they fit within the 12 hour window provided by the DFTPP tune.

6.2.8 The analytical sequence is recorded in the GC/MS run-log and is transcribed into the ChemStation Sequence Table.

6.2.9 After the run has completed, the data files are copied and stored on an additional computer. The Sample sequence is saved under the date in which it began (mmddyy).

6.2.10 The vials are then returned to trays in the organics refrigerator and placed on a shelf designated for post-run samples. They are stored there for 40 days before they are disposed of in designated waste drums.

6.2 Analysis

6.2.1 After the chromatograph of the sample has been completed, the data are evaluated by someone experienced with GCMS data evaluation and the EnviroQuant software.

6.2.2 Data files are quantitated using the appropriate data analysis method prepared from the most recent calibration. Using the Qedit function, each analyte is reviewed for qualitative and quantitative accuracy using retention time, integration, mass spectrum, library references, and secondary ion ratios. Internal standard areas and surrogate recoveries are verified to be acceptable. In the case of failure a sample must be reanalyzed. If there is not enough sample for re-extraction, the data are qualified with an appropriate qualifier. A quant report is printed for each sample.

6.2.3 The reduced quant results are used to generate the final report that will be sent to the customer. The quant report is kept in a project specific folder.

6.3 Sample Calculations

6.3.1 Results are obtained from the GCMS system and associated software. If the extract is diluted, multiply the result and the reporting limit by that dilution factor. The result that appears on the final reports is calculated based on the

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amount of sample used, the final volume of the extract and the result provided from the GCMS The calculation is shown below.

$$\text{Extract Concentration (ug/mL)} = \frac{(A_s)(I_s)}{(A_{is})(RF)}$$

where: As= Response for the parameter to be measured
Ais= Response for the internal standard
Is= Amount of internal standard added to each extract (ug)

As an internal standard method, response factors are utilized to determine concentration of compounds. Response factors are determined by the following equation:

$$RF = \frac{(A_C)(C_{IS})}{(A_{IS})(C_C)}$$

where,
A_C = Area of compound of interest
A_{IS} = Area of Internal Standard
C_C = Concentration of analyte of interest
C_{IS} = Internal Standard Concentration
RF = Response Factor

To determine the reported result:

Aqueous Samples:

$$\text{Concentration (ug/L)} = \frac{\text{extract concentration(ug/mL) X extract volume (mL)}}{\text{Volume Sample Extracted (L)}}$$

Solid Samples:

$$\text{Concentration (ug/g)} = \frac{\text{extract concentration(ug/mL) X extract volume (mL)}}{\text{Amount sample extracted (g) X dry fraction}}$$

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7.0 Quality Control Requirements

7.1 Method Performance

7.1.1 Initial Demonstration of Capability: This is used to characterize instrument performance (calibration and analysis of the ICV) and MDLs prior to performing analyses by this method.

7.1.1.1 Initial Calibration Verification: The ICV must be from a second source or separate lot number and be within ±20% of the expected concentration.

7.1.1.2 Method Detection Limit: MDLs are established for all analytes. Reagent water is fortified at a concentration of two to five times the estimated method detection limit. To Determine an MDL, a minimum of seven replicates of the fortified reagent water are processed through entire analytical system. Calculate the MDL for each analyte as follows:

MDL = (t)*(s)

t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. For seven replicates t = 3.14.

s = standard deviation of the replicate analyses.

MDLs should be determined initially or after significant change to instrument or procedure. See the QA Manual for guidance on preparing a method detection limit standard. The MDL data are stored electronically in the QA/MDL file folder.

7.1.2 LOD/LOQ: Refer to QAM *Analytical Procedures* section, for specific LOD/LOQ requirements. LOD/LOQ data are stored electronically in the QA/LOD&LOQ file folder**.**

7.1.3 IDC/CDC: Proficiency of the analyst is ensured by documentation of an Initial or Continuing Demonstration of Capability (IDC or CDC). Typically, the analyst analyzes four LCS's with acceptable precision and accuracy or shows acceptable performance of a blind sample analysis (usually a PT). See QAM for additional information.

7.1.4 Control Charts: Control Charts are quality control tools which graphically display QC data over time. The data required for generation of accuracy and precision control charts is maintained by the laboratory. See QAM for additional information.

7.1.5 Inter-laboratory performance: Proficiency Test studies, conducted by approved providers, constitute the majority of inter-laboratory performance comparisons. Proficiency tests are performed at a frequency as required by method, regulation, or accrediting body. See QAM for additional information.

7.2 Laboratory Performance Quality Control and Corrective Actions

7.2.1 Continuing Calibration Verification: The CCV result must be within ± 20% of the expected concentration. Up to 20% of the full-list of analytes may exhibit percent recoveries outside of this criterion. If only PAH analytes are to be reported, then all PAH compounds must be within 20% of the true value. If the compounds are not within 20%, the CCV can be remade and reanalyzed, if acceptable the batch can be continued, otherwise a new calibration curve must be prepared. If some compounds exhibit percent recoveries beyond the 20%, samples can still be analyzed as long as those particular compounds are not detected in the samples. For projects requiring DoD protocol, all compounds of interest in the CCV must be recovered between 80% and 120%. Project level approval would be needed for any analytes that do not meet this criterion. For projects requiring DoD protocol, a CCV is also analyzed at the end of the 12 hour window. All compounds of interest in the ending CCV must be recovered between 50% and 150%. Refer to QAM and DoD QSM for additional guidance.

7.2.2 Prep Blank: The PB is prepared once every batch or 24hours, whichever is reached first. The PB undergoes the sample preparation process. The PB

must be below the reporting limit of the analyte, if not refer to the quality SOP for corrective action. For projects requiring DoD protocol, the prep blank should not have any detectable concentrations above half the reporting limit.

7.2.3 Laboratory Control Sample (LCS): The LCS is prepared once every batch or 24 hours, whichever is reached first. The percent recovery is calculated and must meet the acceptance criteria seen in Table 4. Up to 20% of the full-list of analytes may exhibit percent recoveries outside of this criterion. If only PAH analytes are to be reported, then all PAH compounds must be within 20% of the true value. If some compounds exhibit percent recoveries beyond the 20%, samples can still be analyzed as long as those particular compounds are not detected in the samples. If the LCS is determined to be out of control, the source of the problem must be identified and the batch associate samples should be re-extracted and re-analyzed. If re-extraction is not possible, the results would need to be qualified accordingly. Refer to QAM and DoD QSM for additional guidance.

7.2.4 Matrix Spike: Two spikes should be analyzed per 20 samples as sample volume allows. A known amount of analyte is added to an aliquot of a randomly chosen sample. Refer to QAM and DoD QSM for additional guidance. The calculation of the percent recovery is as follows:

$$R = (Cs - C)/s * 100$$

R = percent recovery
Cs = Fortified sample concentration
C = Sample background concentration
s = Concentration equivalent of analyte added to the sample

If the sample matrix spike fails, the LCS results are reviewed to determine whether the failure is matrix specific or an analytical problem. If found to be matrix specific, the report for the spiked sample is noted with this failure.

LCS/MS acceptance criteria: See Table 4

7.2.5 Matrix Duplicate: One matrix duplicate should be extracted per 20

samples. The relative percent difference must be ±20% for waters and ±30% for solids. The RPD is calculated as follows.

RPD = (D1-D2)/((D1+D2)/2) *100

D1 = The initial result of the analyte
D2 = The duplicate result of the analyte

7.2.6 Surrogates: Surrogates are added to all samples. Surrogate recovery criteria can be found in Table 8. Samples failing these criteria are reanalyzed. If the reanalysis shows acceptable recoveries, these results are reported. If the sample reanalysis shows a similar failure, the sample is re-extracted. If no additional sample remains for re-extraction, the failing results are reported, with a notation of the failure and possible cause.

7.3 Retention Times

7.3.1 The retention times in the analytical system should be stable such that peaks can be accurately identified. Variability in the system is reduced by using the relative retention times from the closest internal standard. The width of the retention time window used to make identifications can be based upon the measurements of actual retention times variations of standards over the course of several days. These retention times and windows are estimates. Three times the standard deviation of a retention time can be used to calculate a suggested window size for each analyte. As a default, 0.3 minutes is used. The experience of the analyst should weigh heavily in the interpretation of GCMS data.

8.0 Responsibilities

8.1 The analyst uses the Quality Control Criteria outlined in this SOP to evaluate and validate the data. The analyst or technician's responsibility is to follow this SOP and record all the data in the lab notebook. Any irregularities within the test or samples should be recorded in the lab notebook and reported to the lab manager who is

responsible for reviewing the data.

9.0 Health and Safety

9.1 Refer to the Chemical Hygiene Plan (SOP QA604 and applicable MSDS. Normal, accepted laboratory safety practices should be followed during reagent preparation, extraction and instrument operation. The calibration standards contain compounds that are suspected carcinogens and should be handled accordingly. Every sample in the lab should be handled as if it is hazardous waste. All technicians shall be familiar with the Chemical Hygiene Plan.

10.0Pollution Prevention and Waste Management

10.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. All waste generated as a result of this method is disposed of in labeled drums that are stored until full and picked up by a company dealing in disposal of hazardous material.

10.2 The EPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Waste management in this laboratory is regulated by the Safety director. The aqueous waste left over from the extraction is stored in drums with labels describing the contents. The standards are generally used in their entirety, but in the instances where they expire before they are gone, they are stored in labeled drums to be picked up by a disposal company. The 1.5mL vials containing the extracts are kept under refrigeration for 40 days after analysis. After the 40 days they are disposed of in labeled drums to be picked up by a disposal company.

List of Figures and tables

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Table 1
Base/Neutral Extractables

N-nitroso-dimethylamine	hexachlorocyclopentadiene	anthracene
aniline	2-chloronaphthalene	carbazole
bis-(2-choroethyl)ether	2-nitroanaline	di-n-butyl phthalate
1,3-dichlorobenzene	acenaphthylene	fluoranthene
1,4-diclorobenzene	dimethyl phthalate	benzidine
1,2-dichlorobenzene	2,6-dinitrotoluene	pyrene
benzyl alcohol	2,4-dinitrotoluene	butyl benzyl phthalate
2,2'-oxybis(1-chloropropane)	acenaphthene	benzo(a)anthracene
hexachloroethane	3-nitroanaline	chrysene
N-nitroso-di-n-propylamine	dibenzofuran	3,3'-dichlorobenzidine
nitrobenzene	fluorene	bis-2-ethylhexyl phthalate
isophorone	diethyl phthalate	di-n-octyl phthalate
bis(2-chloroethoxy)methane	4-chlorophenyl phenyl ether	Benzo(b)fluoranthene
1,2,4-trichlorobenzene	4-nitroanaline	benzo(k)fluoranthene
naphthalene	azobenzene	benzo(a)pyrene
benzoic acid	N-nitrosodiphenylamine	indeno(1,2,3-cd)pyrene
4-chloroanaline	4-bromophenyl phenyl ether	dibenzo(a,h)anthracene
hexachloro-1,3-butadiene	hexachlorobenzene	benzo(g,h,i)perylene
2-methylnaphthalene	phenanthrene	

Table 2
Acid Extractables

phenol	2,4-dimethylphenol	2,4-dinitrophenol
2-chlorophenol	2,4-dichlorophenol	4-nitrophenol
2-methylphenol	4-chloro-3-methylphenol	4,6-dinitro-2-methylphenol
4-methylphenol	2,4,6-trichlorophenol	pentachlorophenol
2-nitrophenol	2,4,5-trichlorophenol	

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Table 3
PAH Extractable Compounds

naphthalene	**phenanthrene**	**Benzo(b)fluoranthene**
2-methylnaphthalene	**anthracene**	**benzo(k)fluoranthene**
acenaphthylene	**fluoranthene**	**benzo(a)pyrene**
acenaphthene	**pyrene**	**indeno(1,2,3-cd)pyrene**
dibenzofuran	**benzo(a)anthracene**	**dibenzo(a,h)anthracene**
fluorene	**chrysene**	**benzo(g,h,i)perylene**

Table 4

ABN SPIKING CATEGORIES AND ACCEPTANCE CRITERIA

Analyte Category	LOW (%)	HIGH (%)
ACID COMPOUNDS	30	130
BN COMPOUNDS	40	140

Note: For projects requiring DoD protocol, refer to the DoD Quality Systems Manual for Environmental Laboratories for required LCS/D acceptance limits.

Table 5—DFTPP Key Masses and Abundance Criteria

Mass	m/z Abundance criteria
51	30-60 percent of Mass 198.
68	Less than 2 percent of Mass 69.
70	Less than 2 percent of Mass 69.
127	40-60 percent of Mass 198.
197	Less than 1 percent of Mass 198.
198	Base peak, 100 percent relative abundance.
199	5-9 percent of Mass 198.
275	10-30 percent of Mass 198.
365	Greater than 1 percent of Mass 198.
441	Present but less than Mass 443.
442	Greater than 40 percent of Mass 198.
443	17-23 percent of Mass 442

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Table 6 : Labconco Rapid-Vap Settings for "Program 1"
PROGRAM 1

Speed= 55%
Heat= 42° C
NO_2= 15 psi
Time= 12 minutes to start
Sample No.= Number of Rapid-Vap glassware to be concentrated at one time

Table 7
Typical Quantitation Limits for Reported Compounds

	Aqueous Quant Limit ug/L	Solid Quant Limit ug/g		Aqueous Quant Limit ug/L	Solid Quant Limit ug/g
N-nitrosodimethylamine	2	0.2	2,4-dinitrotoluene	2	0.2
aniline	2	0.2	acenaphthene	0.5	0.05/0.5*
phenol	2	0.2	3-nitroaniline	2	0.2
2-chlorophenol	5	0.5	2,4-dinitrophenol	50	5
bis-(2-chloroethyl)ether	2	0.2	dibenzofuran	0.5	0.05/0.5*
1,3-dichlorobenzene	2	0.2	4-nitrophenol	10	1
1,4-dichlorobenzene	2	0.2	fluorene	0.5	0.05/0.5*
1,2-dichlorobenzene	2	0.2	diethyl phthalate	5	0.5
benzyl alcohol	2	0.2	4-chlorophenyl phenyl ether	5	0.5
2-methylphenol (o-cresol)	2	0.2	4-nitroaniline	5	0.5
2,2'-oxybis(1-chloropropane)	2	0.2	4,6-dinitro-2-methylphenol	20	2
acetophenone	2	0.2	azobenzene	2	0.2
hexachloroethane	2	0.2	N-nitrosodiphenylamine	2	0.2
N-nitroso-di-n-propylamine	2	0.2	4-bromophenyl phenylether	2	0.2
4-methylphenol (p-cresol)	2	0.2	hexachlorobenzene	2	0.2
nitrobenzene	2	0.2	pentachlorophenol	10	1
isophorone	5	0.5	phenanthrene	0.5	0.05/0.5*
2-nitrophenol	2	0.2	anthracene	0.5	0.05/0.5*
2,4-dimethylphenol	2	0.2	carbazole	2	0.2
bis(2-chloroethoxy)methane	5	0.5	di-n-butyl phthalate	5	0.5
2,4-dichlorophenol	5	0.5	fluoranthene	0.5	0.05/0.5*
1,2,4-trichlorobenzene	5	0.5	benzidine	30	3
naphthalene	0.5	0.05/0.5*	pyrene	0.5	0.05/0.5*
benzoic acid	50	5	butyl benzyl phthalate	5	0.5
4-chloroaniline	2	0.2	benzo(a)anthracene	0.5	0.05/0.5*

Title: Method 8270D – PAHs, Base/Neutrals and Acids

hexachloro-1,3-butadiene	2	0.2	chrysene	0.5	0.05/0.5*
4-chloro-3-methylphenol	2	0.2	3,3'-dichlorobenzidine	30	3
2-methylnaphthalene	0.5	0.05/0.5*	bis-2-ethylhexyl phthalate	5	0.5
hexachlorocyclopentadiene	10	1	di-n-octyl phthalate	2	0.2
2,4,6-trichlorophenol	2	0.2	benzo(b)fluoranthene	0.5	0.05/0.5*
2,4,5-trichlorophenol	2	0.2	benzo(k)fluoranthene	0.5	0.05/0.5*
2-chloronaphthalene	5	0.5	benzo(a)pyrene	0.2	0.02/0.2*
2-nitroaniline	2	0.2	indeno(1,2,3-cd)pyrene	0.5	0.05/0.5*
acenaphthylene	0.5	0.05/0.5*	dibenz(a,h)anthracene	0.5	0.05/0.5*
dimethyl phthalate	5	0.5	benzo(g,h,i)perylene	0.5	0.05/0.5*
2,6-dinitrotoluene	2	0.2			

Note: All solid quantitation limits are based on 100% dry weight.
* = The first quantitation limit is for full-list ABNs. The second quantitation limit is for the PAH ONLY list.

Table 8 Surrogate Compounds and Acceptance Criteria

Parameter	Compound	Acceptance Limits (%)
abn	2-fluorophenol	21-100
abn	phenol-d5	10-102
abn	2,4,6-tribromophenol	10-123
abn	nitrobenzene-d5	35-114
abn,pah	2-fluorobiphenyl	43-116
abn	terphenyl-d14	33-141
pah	o-terphenyl	33-141

Note: For projects requiring DoD protocol, refer to the DoD Quality Systems Manual for Environmental Laboratories for required surrogate acceptance limits.

Table 9 Internal Standard Components

1,4-Dichlorobenzene-d4
Naphthalene-d8
Chrysene-d12
Phenanthrene-d10
Acenaphthene-d10
Perylene-d12

Title: Method 8270D – PAHs, Base/Neutrals and Acids

Table 10 CEM Microwave Settings

Wattage: 1600W 100% power
Time: 30 minutes
Warm up: Ramp up to 100 degrees C, 10 minutes
Run Time: Hold 10 minutes
Cool down: 10 minutes

Table 11: Typical GCMS Instrument Conditions (subject to change)

Sample Inlet: GC
Injection Source: GC ALS
Mass Spectrometer: Enabled

7673 Injector

Front Injector:
No parameters specified
Back Injector:

Sample Washes	1
Sample Pumps	4
Injection Volume	1.0 microliters
Syringe Size	10.0 microliters
On Column	Off
Nanoliter Adapter	Off
PostInj Solvent A Washes	4
PostInj Solvent B Washes	0
Viscosity Delay	1 seconds
Plunger Speed	Fast

HP5890 Temperature Parameters

Zone Temperatures:	State	Setpoint
Inlet A:	Off	50 C
Inlet B:	On	250 C
Detector A:	Off	50 C
Detector B:	On	280 C
Auxiliary:	Off	50 C

Title: Method 8270D – PAHs, Base/Neutrals and Acids

Oven Parameters:

Oven Equib Time: 0.01 minutes
Oven Max: 340 C
Oven State: On
Cryo State: Off
Cryo Blast: Off
Ambient: 25 C

Oven Program:

Initial Temperature: 50 C
Initial Time: 1.00 minutes

Level	Rate (C/minute)	Final Temperature (C)	Final Time (minutes)
1	15.0	275	0.00
2(A)	50.0	310	5.00
3(B)	70.0	330	8.10

Next Run Time: 30.09 minutes

HP5890 Purge Valve Settings

Inlet Purge	Init Value	On Time	Off Time	Splitless Injection
A	Off	0.75	40.00	Yes
B	Off	0.75	40.00	Yes

HP5890 Valve and Relay Information

Initial Setpoints:

5890 Valves:

Valve 1: Off Valve 2: Off Valve 3: Off Valve 4: Off

19405 Valves:

Valve 5: Off Valve 6: Off Valve 7: Off Valve 8: Off

Title: Method 8270D – PAHs, Base/Neutrals and Acids

19405 Relays:
Relay 1: Off Relay 2: Off Relay 3: Off Relay 4: Off

HP5890 Detector Information

Detector	Type	State
A	---	Off
B	---	Off

HP5890 Signal Information

Not saving signal data.

Signal	Source	Peak Width	Data Rate	Start Data	Stop Data
1	Testplot	0.053	5.000	0.00	1.00
2	Testplot	0.053	5.000	0.00	1.00

MS ACQUISITION PARAMETER

General Information
------- -----------
Tune File: dftpp.u
Acquistion Mode: Scan

MS Information
-- -----------
Solvent Delay: 3.25 min
EM Absolute: False
EM Offset: 0
Resulting EM Voltage: 2105.9

[Scan Parameters]
Low Mass: 40
High Mass: 450
Threshold: 10
Sample #: 2 A/D Samples 4

Title: **Microwave Extraction by EPA Method 3546**

QA Officer: Jenny Griffith Date: 3/16/16

Laboratory Director: Date: 3/16/16

Author: Date: 3/16/16

Analyst: Date:

By signing this SOP, the analyst has read and understood the latest version of the test method; has read and understood this SOP; acknowledges receipt of this SOP; and is responsible for the implementation of this SOP.

Revision History:

Revision	Changes	Date
1	Procedural clarifications	1/13
2	Prep info for blank sand	7/13
3	RV Program 2, Solvent Mixture for PCB/Pest	3/14
4	Pest Surrogate and Spike amounts revised	2/15
5	Procedure for pesticides revised, high level extraction volume specified for PCBs, RV settings revised, Method Perf. referenced	3/16

Title: Microwave Extraction by EPA Method 3546

1. **Purpose and Applicability**
   1.1 This method covers the extraction of a number of organic compounds that are partitioned into an organic solvent and are amenable to gas chromatography. The determinative methods that may utilize this extraction method are listed below:

Method	SOP#	Extraction Type
EPA8270	5200	Low Level
EPA8082	5303	High Level
EPA8081	5304	Low Level
EPA8015	5501	Low Level

   1.2 This method is for use only by or under the supervision of analysts experienced in Microwave extraction. Each analyst must demonstrate the ability to generate acceptable results with this method.

2. **Definitions**
   2.1 Field Duplicates: A duplicate sample taken in the field. These are done at the request of a customer.
   2.2 Laboratory Control Sample (Laboratory Fortified Blank) (LCS): An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly as a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate measurements. The acceptance criteria for recovery varies among compounds.
   2.3 Preparation Blank (Laboratory Reagent Blank) (PB): An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, and reagents that are used with field samples. The PB is used to determine if the method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. The analytes found in the PB must be below the reporting limit for each analyte. For projects requiring DoD protocol, refer to the QAM for additional evaluation criterion for method blanks.
   2.4 Stock Standard Solution (SSS): A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source, i.e. Absolute Standards or Ultra Scientific. Documentation of the purity of the SSS are retained for traceability purposes in the organic standards binder.
   2.5 Matrix Spike: An aliquot of a sample spiked with a known amount of a standard.
   2.6 Surrogate Solution: A solution containing a known concentration of compounds that are

not found in environmental samples, but behave similar to compounds of interest. Surrogates are added to all field and QC samples to determine the thoroughness of the extraction.

3. **Applicable Documents/References**
   - 3.1 EPA Method 3546.
   - 3.2 US EPA Contract Laboratory Program, Statement of Work for Organic Analysis, 2/88 and 3/91
   - 3.3 ARA SOP QA-400 Sample Receiving and Identification
   - 3.4 ARA QA Manual
   - 3.5 MADEP MCP Guidance, CAM, July 2010
   - 3.6 Current DoD Quality Systems Manual for Environmental Laboratories

4. **Materials and Apparatus**
   - 4.1 **Equipment:**
     - 4.1.1 Hamilton syringes (or equivalent) in 250, 500, and 1000 uL sizes
     - 4.1.2 1.5 mL screw cap vials with Teflon-faced silicon septa
     - 4.1.3 Rapid-Vap Concentrator and nitrogen
     - 4.1.4 Rapid-Vap Concentrator glassware
     - 4.1.5 Laboratory Scale
     - 4.1.6 Scoopulas
     - 4.1.7 Glass Wool
     - 4.1.8 Glass funnels
     - 4.1.9 Microwave vessels with vent plugs and caps
   - 4.2 **Reagents/Standards:**
     - 4.2.1 Methylene Chloride: HPLC grade
     - 4.2.2 50/50 Mix Acetone/Methylene Chloride: Pesticide residue grade or equivalent.
     - 4.2.3 50/50 Mix Hexane/Acetone: Pesticide residue grade or equivalent. Prepared fresh daily.
     - 4.2.4 Hexane: Pesticide residue grade or equivalent.
     - 4.2.5 Surrogate Solution: See determinative method SOP for specific compounds and concentrations.
     - 4.2.6 Spiking Solution: See determinative method SOP for specific compounds and concentrations.
     - 4.2.7 Sodium Sulfate: Purchased from a reputable supplier such as UCT, Inc. The sodium sulfate should be purified by heating at 400^{o}C for four hours or extraction with methylene chloride. The purification step may be skipped as long as routine analysis of preparation blanks demonstrates the absence of contamination.
     - 4.2.8 Sodium hydroxide: 10M solution prepared from sodium hydroxide pellets.

Title: Microwave Extraction by EPA Method 3546

4.2.9 Sulfuric acid: 1:1 solution prepared from stock acid.
4.2.10 De-ionized Water: Reagent grade purified/Deionized water
4.2.11 Sand for Blanks: Play sand purchased from a hardware store. Place several cups of sand in a fine mesh bag. Rinse with DI Water to remove fine particles. Bake in a foil loaf pan in the muffler furnace at 500 degrees Celsius for a few hours until the sand is dry. Allow to cool completely. Assign an Organic Prep number.

5. Method/Calibration/Interferences

5.1 Method Summary

5.1.1 Samples are extracted by microwave. These extracts are quantitatively concentrated in a Labconco Rapid-Vap. The extracts are then analyzed by the appropriate determinative method.

5.2 Sample Collection, Preservation, and Storage

5.2.1 See determinative method SOP for specific sample collection, preservations, and storage requirements. In general, solid matrix samples should be delivered on ice 4±2˚C and extracted within 14 days from sampling.
5.2.2 Shipment of samples must follow the requirements of the method and SOP.

5.3 Calibration Procedure

5.3.1 See determinative method SOP

5.4 Interferences

5.4.1 Interference 1: Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/ or elevated baselines in the chromatogram. The preparation blank is utilized to detect such contamination. If contamination is found, its source is found and is replaced with uncontaminated materials.
5.4.2 Interference 2: Glassware must be scrupulously cleaned and rinsed with appropriate solvent prior to the extraction to minimize contamination.
See determinative method SOP for additional interference.

6. Sample Preparation

6.1 A batch is defined as a 24 hour window in which up to 20 samples can be prepped. Every batch must be accompanied by a preparation blank and a laboratory control sample. Every tenth sample should be matrix spiked and every twentieth sample should be done in duplicate and matrix spiked. Based on specified protocols or specific quality control plans, a duplicate LCS can be substituted for matrix specific QC.
6.2 Low-Level Sample Extraction
6.2.1 Homogenize the sample by mixing it with a scoopula. Add a small amount of sodium sulfate to the bottom of a clean microwave vessel before taring balance. Measure an adequate amount of soil to meet the required analytical detection limits

into a numbered microwave vessel. Add a similar amount of sodium sulfate. Place white plug onto vessel and shake to integrate sodium sulfate with sample.

6.2.2 (The typical amount of soil is 10g to 10g of sodium sulfate for ABN and 5g of soil to 5g of sodium sulfate for pesticides and PCBs.) Note: If the sample has standing water, pour off the water before mixing the soil. If the sample contains large rocks or pieces of sample, attempt to get a representative aliquot of sample without the large pieces.

6.2.3 Be sure to change scoopula between each sample and gloves as needed, if they become contaminated with soil. Brush any fallen pieces off balance before recording weight.

6.2.4 For the quality control samples (PB,LCS,LCSD) previously rinsed and baked sand is measured into coinciding vessels. An equivalent volume of sodium sulfate is added.

6.2.5 Immediately after measuring out the appropriate sample volume, an additional aliquot of the sample should be prepared for dry weight determination. Refer to SOP QA5834 for procedure.

6.2.6 If the samples are damp or wet, let sit for 10-30 minutes to allow moisture to be absorbed by the sodium sulfate.

6.2.7 Using a gas tight micro-liter syringe, add surrogate. The extraction surrogate is added directly to the soil/sodium sulfate mixture inside the microwave vessel. It is at this time that appropriate amount of the spiking solutions are added to the LCS and LCSD and matrix spikes if they have been requested by the customer. See applicable determinative method SOP for standard solutions to be used. The appropriate volumes of standards to add are listed in Table 4.

6.2.8 See Table 3 for the appropriate amount and type of solvent to add to extraction vessel.

6.2.9 Place white plug and cap on each vessel. Tighten each cap making sure that no solvent can escape. The cap should be finger tight and then rotate an additional 30 degrees to tighten the cover to the proper torque range.

6.2.10 Shake vessel to incorporate solvent with solid sample. Note start time in Prep log.

6.2.11 Each vessel in placed in a composite sleeve in the microwave turntable. Make sure vessels are placed evenly around the turntable. (Note: NO LESS THAN 8 SAMPLES IN THE MICROWAVE). Place the turntable with the samples on the turntable drive lug. Close the door and select appropriate program (see table 2).

6.2.12 Once the program has concluded. Remove the turntable from the microwave. Remove each vessel from the composite sleeves and place the vessels in a rack to cool. The vessels must be brought to room temperature before loosening the caps to avoid loosing any solvent.

6.2.13 As caps are removed, check to ensure solvent was not lost from the vessel. This

would be evident by signs of solvent under the cap or around the plug, as well as a reduced volume in the vessel.

6.2.14 A plug of glass wool is added to a glass funnel, and a few grams of sodium sulfate are added on top of the glass wool. Rinse with extraction solvent and set on top of a clean RV tube that is labeled with the lab number ID.

6.2.15 The extract is poured through the funnel. Tap the soil from the vessel in to the funnel. Rinse the vessel twice with appropriate solvent, pouring through the funnel. The funnel is also rinsed with solvent into the RV tube. See table 1 for RV settings.

6.2.16 Check RV at intervals and watch sample volume to ensure too much does not evaporate. Unless extracting pesticides or use of solvent exchange is required, concentrate to just below 1mL.

6.2.17 If extracting pesticides, concentrate to just below 5mLs and rinse the Rapid-Vap glassware with a small portion of n-Hexane. Use this rinse to bring the extract to a final volume of 5mL. Pesticide solids are subjected to Florisil clean-up. For details on this procedure refer to QA-5304, section 6.0.

6.2.18 If solvent exchange is required, concentrate to around 5mLs, then add approximately 10mLs of hexane and continue to concentrate down to just below 1mL.

6.2.19 Remove the glassware from the Rapid-Vap and using a disposable pipette transfer the extract to a 1.5mL glass vial with a Teflon lined cap. Rinse the sides of the Rapid-Vap glassware with a small portion of final solvent. Using this rinse solvent, bring the final extract up to 1mL. This can be done by using a newly made 1mL reference vial of MeCl2 for comparison or using a 1mL syringe to remove extract and bring to the final volume with the rinse solvent.

6.2.20 The vessel and funnel are allowed to air dry in the hood and then washed with soap and water, rinsed with DI water and rinsed with solvent before each use.

6.3 High-Level Extraction Procedure

6.3.1 Homogenize the sample by mixing it with a scoopula. An adequate amount of soil to meet the required analytical detection limits is measured into a microwave vessel. Add a similar amount of sodium sulfate to samples as well as the QC, and mix by shaking vessel. (The typical amount of soil for High-Level PCBs is 3 grams to 3 grams of sodium sulfate.)

6.3.2 For the quality control samples (PB, LCS, LCSD), previously rinsed and baked sand is measured into coinciding vessels. An equivalent volume of sodium sulfate is also added. Place white plug onto vessel and shake to integrate sodium sulfate with sample.

6.3.3 If the samples are damp or wet, let sit for 10-30 minutes to allow moisture to be absorbed by the sodium sulfate.

Title: Microwave Extraction by EPA Method 3546

6.3.4 Immediately after measuring out the appropriate sample volume, an additional aliquot of the sample should be prepared for dry weight determination. Refer to SOP QA5834 for procedure.

6.3.5 Using a gas tight micro-liter syringe, add the extraction surrogate directly to the soil/sodium sulfate mixture inside the microwave vessel. It is at this time that the appropriate amount of the spiking solution is added to the LCS and LCSD and matrix spikes if they have been requested by the customer. See applicable determinative method SOP for standard solutions to be used. The appropriate volumes of standards to add are listed in Table 4.

6.3.6 Using a graduated cylinder, add 10mL of solvent (See Table 3) to each vessel. This solution must be prepared fresh daily.

6.3.7 Place white plug and cap on each vessel. Tighten each cap making sure that no solvent can escape. The cap should be finger tight and then rotate an additional 30 degrees to tighten the cover to the proper torque range. Shake vessel to incorporate solvent with solid sample. Note start time in prep log.

6.3.8 Each vessel is placed in a composite sleeve in the microwave turntable. Make sure vessels are placed evenly around the turntable. (Note: NO LESS THAN 8 SAMPLES IN THE MICROWAVE). Place the turntable with the samples on the turntable drive lug. Close the door and select method 3546. See Table 2 for microwave operating parameters.

6.3.9 Once the program has concluded, remove the turntable from the microwave. Remove each vessel from the composite sleeves and place the vessels in a rack to cool. The vessels must be brought to room temperature before loosening the caps to avoid losing any solvent.

6.3.10 As caps are removed, check to ensure solvent was not lost from the vessel. This would be evident by signs of solvent under the cap or around the plug, as well as a reduced volume in the vessel.

6.3.11 Remove 1mL of the extract directly from the microwave vessel and place in a 1mL vial labeled with the appropriate lab number.

6.3.12 Slowly add approximately 1mL concentrated Sulfuric acid to the extract and shake well. Place vials in the centrifuge at1500 RPM for 2 minutes to allow the distinct layers to form between the acid and the extract. Remove 0.5mL and transfer to a new vial labeled with the appropriate lab number. Make sure to only get the extract and none of the acid.

6.3.13 The final extract volume is recorded. This volume is 5mLs since a twofold concentration step occurs when the acid is mixed with the hexane and acetone solution. This is due to the miscible properties of acetone and sulfuric acid, and immiscible properties of the acetone/sulfuric acid and hexane.

6.3.14 The vials with the sulfuric acid are stored in the refrigerator for later disposal.

Title: Microwave Extraction by EPA Method 3546

7. **Instrument Setup**
   7.1 See determinative method SOP.

8. **Analysis**
   8.1 See determinative method SOP.

9. **Sample Calculations**
   9.1 See determinative method SOP.

10. **Quality Control Requirements**
    10.1 **Method Performance**
       10.1.1 **Initial Demonstration Requirements-** See determinative method SOP.

11. **Laboratory Performance Quality Control and Corrective Actions**
    11.1 Prep Blank: The PB is prepared once every batch or 24hours, whichever is reached first. The PB undergoes the sample preparation process. The PB must be below the reporting limit of the analyte, if not refer to the quality SOP for corrective action. For projects requiring DoD protocol, refer to the QAM for additional evaluation criterion for method blanks.
    11.2 Laboratory Control Sample (LCS): The LCS is prepared once every batch or 24 hours, whichever is reached first. The percent recovery is calculated and must meet the acceptance criteria seen in Table 4. If the LCS is out of control, the source of the problem must be identified and the LCS re-analyzed before continuing onto samples. For projects requiring DoD protocol or when QAPPs require, the full list of analytes are spiked into LCS/Ds and MS/Ds. Refer to QAM and DoD QSM for additional guidance.
    11.3 Matrix Spike: Refer to determinative method SOP for frequency and acceptance criteria. A known amount of analyte is added to an aliquot of a randomly chosen sample. For projects requiring DoD protocol or when QAPPs require, the full list of analytes are spiked into LCS/Ds and MS/Ds. Refer to QAM and DoD QSM for additional guidance. The calculation of the percent recovery is as follows:

$$R = (Cs - C)/s * 100$$

R = percent recovery
Cs = Fortified sample concentration
C = Sample background concentration
s = Concentration equivalent of analyte added to the sample

       11.3.1 If the sample matrix spike fails, the LCS results are reviewed to determine whether the failure is matrix specific or an analytical problem. If found to be matrix specific, the report for the spiked sample is noted with this failure.

Title: Microwave Extraction by EPA Method 3546

11.4 Matrix Duplicate: Refer to determinative method SOP for frequency and acceptance criteria. RPD is calculated as follows:

RPD = (D1-D2)/((D1+D2)/2) *100
D1 = The initial result of the analyte
D2 = The duplicate result of the analyte

11.5 Surrogates: Surrogates are added to all samples. Samples failing these criteria are reanalyzed. If the reanalysis shows acceptable recoveries, these results are reported. If the sample reanalysis shows a similar failure, the sample is re-extracted. If no additional sample remains for re-extraction, the failing results are reported, with a notation of the failure and possible cause.

12. **Retention Times**

See determinative method SOP

13. **Responsibilities**

13.1 The analyst uses the Quality Control Criteria outlined in this SOP to evaluate and validate the data. It is the analyst's or technician's responsibility to follow this SOP and record all the data in the lab notebook. Any irregularities within the test or samples should be recorded in the lab notebook and reported to the lab manager who is responsible for reviewing the data.

14. **Health and Safety**

14.1 Normal, accepted laboratory safety practices should be followed during reagent preparation, extraction and instrument operation. The calibration standards contain compounds that are suspected carcinogens and should be handled accordingly. Every sample in the lab should be handled as if it is hazardous waste. Refer to Chemical Hygiene Plan (SOP QA604), and SDS sheets.

15. **Pollution Prevention and Waste Management**

15.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. All waste generated as a result of this method is disposed of in labeled drums that are stored until full and picked up by a company dealing in disposal of hazardous material.

15.2 The EPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Waste management in this laboratory is regulated by the Hazardous Waste Coordinator/Manager. The aqueous waste left over from the extraction is stored in drums with labels describing the contents. The standards are generally used in their entirety, but in the instances where they expire before they are gone, they are stored in labeled drums to be picked up by a disposal company. The

Title: Microwave Extraction by EPA Method 3546

1.5mL vials containing the extracts are kept under refrigeration for 40 days after analysis. After the 40 days they are disposed of in labeled drums to be picked up by a disposal company.

Table 1 : Labconco Rapid-Vap Settings for "Program 1"

PROGRAM 1

Speed= 55%
Heat= 42° C
NO_2= 15 psi
Time= 12 minutes to start
Sample No.= Number of Rapid-Vap glassware to be concentrated at one time

PROGRAM 2

Speed=55%
Heat=62° C
NO2=15psi
Time= 4 minutes to start
Sample No.= Number of Rapid-Vap glassware to be concentrated at one time

Table 2 CEM Microwave Settings

Wattage: 1600W 100% power
Total Time: 30 minutes
Warm up: 10 minute ramp up to 100 degrees C
Run Time: Hold 10 minutes
Cool down: 10 minutes

Table 3 Solvent Specifications

Method	Amount	Type
8270	30mL	1:1 Methylene Chloride:Acetone
8081	25mL	1:1 Hexane:Acetone
8082	10mL	1:1 Hexane:Acetone
8015	30mL	Methylene Chloride

Title: Microwave Extraction by EPA Method 3546

Table 4 Surrogate and Spike amounts

Method	SS	Spike
EPA8270	0.1mL	0.4mL
EPA8082	0.1mL	0.1mL
EPA8081	0.2mL	0.2mL
EPA8015	0.1mL	0.1mL

Note: Table 4 contains typical volumes of solutions added to samples and QC. Depending on concentrations of stock spikes and surrogates, volumes used should be discussed with the determinative method analyst and recorded in the appropriate extraction logbooks.

Title: **Separatory Funnel Liquid-Liquid Extraction by EPA Method 3510C**

QA Officer: Jennifer Guerrette Date: 8/1/17

Laboratory Director: Leonard Hybl Date: 8/2/17

Author: [signature] Date: 8/1/17

Analyst: _____ Date: _____

By signing this SOP, the analyst has read and understood the latest version of the test method; has read and understood this SOP; acknowledges receipt of this SOP; and is responsible for the implementation of this SOP.

Revision History:

Revision	Changes	Date
1	QC frequency to ref. determinative SOP	1/13
2	RV Methods and spike amounts. Solvent Exchange for single pH samples. Salt addition for PCB/Pest Extraction	3/14
3	Added salt to reagents, revised pH adjustment with sodium hydroxide, revised salting out quantity for PCB/Pest, revised RV programs, revised 8270 amounts in table 2, Method Perf Referenced	3/16
4	Added requirement to check pH of samples prior to extraction; added Table 3 pH Requirements	7/17

Title: **Separatory Funnel Liquid-Liquid Extraction by EPA Method 3510C**

1. **Purpose and Applicability**
   1.1 This method covers the determination of a number of organic compounds that are partitioned into an organic solvent and are amenable to gas chromatography. The determinative methods that may be qualitatively and quantitatively determined using this extraction method are as follows:

Method	SOP#
EPA8270	5200
EPA8082	5303
EPA8081	5304
EPA8015	5501

   1.2 This method is for use only by or under the supervision of analysts experienced in the use of gas chromatograph/mass spectrometer and in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method.

2. Definitions
   2.1 Field Duplicates: A duplicate sample taken in the field. These are done at the request of a customer.
   2.2 Laboratory Control Sample (Laboratory Fortified Blank) (LCS): An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly as a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate measurements.
   2.3 Prep Blank (Laboratory Reagent Blank) (PB): An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, and reagents that are used with field samples. The PB is used to determine if the method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. The analytes found in the PB must be below the reporting limit for each analyte. For projects requiring DoD protocol, refer to the QAM for additional evaluation criterion for method blanks.
   2.4 Stock Standard Solution (SSS): A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source, i.e. Absolute Standards or Ultra Scientific. Documentation of the purity of the SSS are retained for traceability purposes in the organic standards binder.
   2.5 Matrix Spike: An aliquot of a sample spiked with a known amount of a standard.

Title: **Separatory Funnel Liquid-Liquid Extraction by EPA Method 3510C**

2.6 Surrogate Solution: A solution containing a known concentration of compounds that are not found in environmental samples, but behave similar to compounds of interest. Surrogates are added to all field and QC samples to determine the thoroughness of the extraction.

3. **Applicable Documents/References**
   - 3.1 Test Methods for Evaluation Solid Waste, Physical/Chemical Methods, USEPA SW 846, 3rd Edition, 1986, Method 3510C
   - 3.2 US EPA Contract Laboratory Program, Statement of Work for Organic Analysis, 2/88 and 3/91
   - 3.3 ARA SOP QA-400 Sample Receiving and Identification
   - 3.4 ARA QA Manual
   - 3.5 MADEP MCP Guidance, CAM, July 2010
   - 3.6 Current DoD Quality Systems Manual for Environmental Laboratories

4. **Materials and Apparatus**
   - 4.1 **Equipment:**
     - 4.1.1 Hamilton syringes (or equivalent) in 250, 500, and 1000 uL sizes
     - 4.1.2 1.5 mL screw cap vials with Teflon-faced silicon septa
     - 4.1.3 Teflon separatory funnels
     - 4.1.4 Glass receiving jars with Teflon lined lids
     - 4.1.5 Rapid-Vap Concentrator and nitrogen
     - 4.1.6 Rapid-Vap Concentrator glassware
     - 4.1.7 Glass Wool
     - 4.1.8 Glass funnels
     - 4.1.9 pH paper
   - 4.2 **Reagents/Standards:**
     - 4.2.1 Methylene Chloride: HPLC grade
     - 4.2.2 Hexane: Pesticide Grade
     - 4.2.3 Surrogate Solution: See determinative method SOP for specific compounds and concentrations.
     - 4.2.4 Spiking Solution: See determinative method SOP for specific compounds and concentrations.
     - 4.2.5 Sodium Sulfate: Purchased from a reputable supplier such as UCT, Inc. The sodium sulfate should be purified by heating at 400°C for four hours or extraction with methylene chloride. The purification step may be skipped as long as routine analysis of preparation blanks demonstrates the absence of contamination.
     - 4.2.6 Sodium hydroxide: 10M solution prepared from sodium hydroxide pellets. 400 g

Title: **Separatory Funnel Liquid-Liquid Extraction by EPA Method 3510C**

NaOH pellets diluted to 1000 ml with DI water. Add water slowly and allow sufficient cool down time.

4.2.7 Sulfuric acid: 1:1 solution prepared from stock acid.

4.2.8 NaCl: laboratory grade salt from a reputable provider such as Fischer Scientific

4.2.9 De-ionized Water: Reagent grade purified/Deionized water

5. **Method/Calibration/Interferences**

5.1 Method Summary

5.1.1 Samples are extracted by separatory funnel extraction. These extracts are quantitatively concentrated in a Labconco Rapid-Vap. The extracts are then analyzed by the appropriate determinative method.

5.2 Sample Collection, Preservation and Storage

5.2.1 See determinative method SOP for specific sample collection, preservations, and storage requirements. In general, aqueous matrix samples should be delivered on ice and extracted within 7 days from sampling.

5.2.2 Shipment of samples must follow the requirements of the method and SOP.

5.3 Calibration Procedure

5.3.1 See determinative method SOP.

5.4 Interferences

5.4.1 Interference 1: Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/ or elevated baselines in the chromatogram. The preparation blank is utilized to detect such contamination. If contamination is found, its source is found and is replaced with uncontaminated materials.

5.4.2 Interference 2: Glassware must be scrupulously cleaned and rinsed with appropriate solvent prior to the extraction to minimize contamination.

5.4.3 See determinative method SOP for additional interferences.

5.5 Calibration Procedure

See determinative method SOP.

6. **Procedure**

6.1 A batch is defined as a 24 hour window in which up to 20 samples can be prepped. Every batch must be accompanied by a preparation blank and a laboratory control sample. Every tenth sample should be matrix spiked and every twentieth sample should be done in duplicate and matrix spiked if sample volume exists. If sample volume does not exist a duplicate LCS can be substituted.

Title: **Separatory Funnel Liquid-Liquid Extraction by EPA Method 3510C**

6.2 Aqueous ABN, Acid, or BN Sample Preparation

6.2.1 Set up scrupulously cleaned separatory funnels that have been washed with Alconox, rinsed with DI water, and rinsed with methylene chloride.

6.2.2 Mark the level of the sample on the sample container.

6.2.3 Check the pH of the sample with wide-range pH paper and adjust the pH, if necessary, to the pH indicated in Table 3, using 1:1 (v/v) sulfuric acid or 10 N sodium hydroxide. Document initial sample pH and any adjustments made in the associated lab prep log.

6.2.4 While working in the hood, use a gas-tight syringe to add the appropriate amount of surrogate solution to all sample bottles and mix thoroughly. If matrix spike samples are to be performed, add the appropriate spiking solution to the sample bottle at this time. See applicable determinative method SOP for standard solutions to be used. The appropriate volumes of standards to add are listed in Table 2.

6.2.4.1 Note 1: If sample bottle is filled to the top and there is a risk of overflowing the bottle by adding surrogate/spike directly to the sample bottle, transfer sample to clean separatory funnel and then add the respective surrogate/spike amounts directly into the separatory funnel. Note in the prep log if surrogate and spike were not added directly to the sample bottle.

6.2.4.2 Note 2: If the sample bottle has a large amount of sediment on the bottom of the bottle, transfer the sample into a clean separatory funnel and then add the respective surrogate/spike amounts directly into the separatory funnel. Do not rinse bottle with methylene chloride. Note in the prep log if the bottle could not be rinsed due to heavy sediment.

6.2.5 Pour the sample into the separatory funnel and fill each of the required QC separatory funnels with 1L of laboratory grade water. Add the appropriate surrogate and spiking solution to the QC samples. IF ONLY ACID EXTRACTABLES ARE REQUESTED, SKIP AHEAD TO STEP 6.2.11.

6.2.6 Adjust the pH of the samples and QC to >11 by addition of the sodium hydroxide solution. (4.2.6)

6.2.7 Add 60mLs methylene chloride to the sample container to extract any residue that may have adhered to the sides of the container. Shake the container briefly and pour the solvent into the separatory funnel.

6.2.8 In an effort to reduce solvent usage and environmental impact, alternative solvent volumes are permissible for projects other than DoD or where project specific criteria exists that would prohibit modifications. The alternate solvent volumes employed are three extractions utilizing 40mLs, 30mLs, and 20mLs. Note in prep log the date and start time of each QC set.

6.2.9 Shake each separatory funnel for 2 minutes, venting pressure often. Allow the

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two layers to separate for a minimum of 10 minutes.

6.2.10 Collect the organic layer (bottom layer) into a previously washed and solvent rinsed glass jar labeled with a piece of masking tape denoting the appropriate lab number and analysis type.

6.2.11 Repeat extraction two more times using 60mLs of methylene chloride. If reduced solvent usage is employed, repeat extraction two mores times with 30mLs and 20mLs or methylene chloride. IF ONLY BASE-NEUTRAL EXTRACTABLES ARE REQUESTED, SKIP TO STEP 6.2.15.

6.2.12 Adjust the pH of the sample and QC to <2 with the 1:1 sulfuric acid solution (4.2.7) and add 60mLs methylene chloride to the separatory funnel. If reduced solvent usage is employed, add 40mLs methylene chloride to the separatory funnel.

6.2.13 Shake each separatory funnel for 2 minutes, venting pressure often. Allow the two layers to separate for a minimum of 10 minutes.

6.2.14 Collect the organic layer in a separate previously washed and solvent rinsed glass jar labeled with a piece of masking tape denoting the appropriate lab number and analysis type.

6.2.15 Repeat extraction two more times using 60mLs of methylene chloride. If reduced solvent usage is employed, repeat extraction two mores times with 30mLs and 20mLs or methylene chloride.

6.2.16 Add sodium sulfate to the sample and QC jars of solvent to remove any residual water that may remain in the extract. Cap the jars with previously cleaned and solvent rinsed Teflon lined lids and agitate the jar to thoroughly trap all the moisture in the sodium sulfate.

6.2.17 Place a plug of glass wool into a previously cleaned and solvent rinsed glass funnel and rinse the glass wool with methylene chloride. Place the funnel atop previously cleaned and solvent rinsed Rapid-Vap glassware that has been labeled with the appropriate lab number and analysis type.

6.2.18 Quantitatively transfer the extract through the funnel into the Rapid-Vap glassware. Rinse the inside of the jar with methylene chloride and pour the rinsate into the funnel, repeat one more time.

6.2.19 Place glassware into Rapid-Vap and run “Program 1” (see Table 1 for settings) to concentrate the sample to the appropriate final volume and solvent (typically 1mL).

6.2.20 Remove the glassware from the Rapid-Vap and using a disposable pipette transfer the extract to a 1.5mL glass auto-sampler vial with a Teflon lined cap. Rinse the sides of the glassware with a small volume of methylene chloride. Using this rinse solvent, bring the final extract up to 1mL in the vial using a newly made 1mL reference vial of the same solvent for comparison.

6.2.21 Place the extract in the appropriate tray in the organics refrigerator and alert the

analyst to the completion of the extraction.

6.2.22 Fill the empty sample container with water up to the mark (6.2.2) and determine the volume with a graduated cylinder. Record this volume in the extraction notebook.

6.2.23 Dispose of the remaining aqueous sample from the separatory funnel into the satellite waste receiver located in the hood.

6.2.24 Verify that all necessary information concerning the extraction has been documented in the appropriate extraction log book.

6.3 Single pH Sample Preparation

6.3.1 Set up scrupulously cleaned separatory funnels that have been washed with Alconox, rinsed with DI water, and rinsed with methylene chloride.

6.3.2 Mark the level of the sample on the sample container.

6.3.3 Check the pH of the sample with wide-range pH paper and adjust the pH, if necessary, to the pH indicated in Table 3, using 1:1 (v/v) sulfuric acid or 10 N sodium hydroxide. Document initial sample pH and any adjustments made in the associated lab prep log. This step does not apply to samples under 8100 (TPH) or 8015(DRO) because they are extracted at the "as received" pH per the EPA 3510c extraction method.

6.3.4 While working in the hood, use a gas-tight syringe to add the appropriate amount of surrogate solution to all sample bottles and mix thoroughly. If matrix spike samples are to be performed, add the appropriate spiking solution to the sample bottle at this time. See applicable determinative method SOP for standard solutions to be used. The appropriate volumes of standards to add are listed in Table 2.

6.3.4.1 Note 1: If sample bottle is filled to the top and there is a risk of overflowing the bottle by adding surrogate/spike directly to the sample bottle, transfer sample to clean separatory funnel and then add the respective surrogate/spike amounts directly into the separatory funnel. Note in the prep log if surrogate and spike were not added directly to the sample bottle.

6.3.4.2 Note 2: If the sample bottle has a large amount of sediment on the bottom of the bottle, transfer the sample into a clean separatory funnel and then add the respective surrogate/spike amounts directly into the separatory funnel. Do not rinse bottle with methylene chloride. Note in the prep log if the bottle could not be rinsed due to heavy sediment.

6.3.4.3 Note3: If extracting PCB and or Pesticide samples add roughly10g of salt to each sample and corresponding QC.

6.3.5 Pour the sample into the separatory funnel and fill each of the required QC

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separatory funnels with 1L of laboratory grade water. Add the appropriate surrogate and spiking solution to the QC samples.

6.3.6 Add 60mLs methylene chloride to the sample container to extract any residue that may have adhered to the sides of the container. Shake the container briefly and pour the solvent into the separatory funnel. In an effort to reduce solvent usage and environmental impact, alternative solvent volumes are permissible for projects other than DoD or where project specific criteria exists that would prohibit modifications. The alternate solvent volumes employed are three extractions utilizing 40mLs, 30mLs, and 20mLs. Document in prep log start time and date or each QC set.

6.3.7 Shake each separatory funnel for 2 minutes, venting pressure often. Allow the two layers to separate for a minimum of 10 minutes.

6.3.8 Collect the organic layer (bottom layer) into a previously washed and solvent rinsed glass jar labeled with a piece of masking tape denoting the appropriate lab number and analysis type.

6.3.9 Repeat extraction two more times using 60mLs of methylene chloride. If reduced solvent usage is employed, repeat extraction two mores times with 30mLs and 20mLs or methylene chloride.

6.3.10 Add sodium sulfate to the sample and QC jars of solvent to remove any residual water that may remain in the extract. Cap the jars with previously cleaned and solvent rinsed Teflon lined lids and agitate the jar to thoroughly trap all the moisture in the sodium sulfate.

6.3.11 Place a plug of glass wool into a previously cleaned and solvent rinsed glass funnel and rinse the glass wool with methylene chloride. Place the funnel atop the previously cleaned and solvent rinsed Rapid-Vap glassware that has been labeled with the appropriate lab number and analysis type.

6.3.12 Quantitatively transfer the extract through the funnel into the Rapid-Vap glassware. Rinse the inside of the jar with methylene chloride and pour rinsate through funnel, repeat one more time.

6.3.13 Place glassware into Rapid-Vap and run “Program 1” (see Table 1 for settings) to concentrate the sample to the appropriate final volume and solvent (typically 1mL). Remove the glassware from the Rapid-Vap and using a disposable pipette transfer the extract to a 1.5mL glass vial with a Teflon lined cap. Rinse the sides of the Rapid-Vap glassware with a small portion of methylene chloride. Using this rinse solvent, bring the final extract up to 1.0mL in the vial using a newly made 1.0mL reference vial of methylene chloride for comparison. For samples requiring solvent exchange, concentrate the sample to approximately 2mLs. Add a minimum of 10mLs of hexane to the Rapid-Vap vial and swirl to mix the two solvents. Return the vial to the Rapid-Vap on “Program 2” (see Table 1 for settings) and concentrate to just below 1mL. Rinse the side

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of the RV tube with hexane to bring the final extract to 1mL.

6.3.14 Place the completed extract in the appropriate tray in the organics refrigerator and alert the analyst to the completion of the extraction.

6.3.15 Dispose of the remaining aqueous sample from the separatory funnel into the satellite waste receiver located in the hood.

6.3.16 Fill the empty sample container with water up to the mark (6.3.2) and determine the volume with a graduated cylinder. Record this value in the extraction notebook.

6.3.17 Verify that all necessary information concerning the extraction has been documented in the appropriate extraction log book.

7. **Instrument Setup**
   7.1 See determinative method SOP.
8. **Analysis**
   8.1 See determinative method SOP.

9. **Sample Calculations**
   9.1 See determinative method SOP.

10. **Quality Control Requirements**
    10.1 Method Performance
    10.1.1 Initial Demonstration Requirements- See determinative method SOP.

11. **Laboratory Performance Quality Control and Corrective Actions**
    11.1 Prep Blank: The PB is prepared once every batch or 24hours, whichever is reached first. The PB undergoes the sample preparation process. The PB must be below the reporting limit of the analyte, if not refer to the quality SOP for corrective action. For projects requiring DoD protocol, refer to the QAM for additional evaluation criterion for method blanks.
    11.2 Laboratory Control Sample (LCS): The LCS is prepared once every batch or 24 hours, whichever is reached first. The percent recovery is calculated and must meet the acceptance criteria found in the determinative method SOP. If the LCS is out of control, the source of the problem must be identified and the LCS re-analyzed before continuing onto samples. For projects requiring DoD protocol or when QAPPs require, the full list of analytes are spiked into LCS/Ds and MS/Ds. Refer to QAM and DoD QSM for additional guidance.
    11.3 Matrix Spike: Refer to determinative method SOP for frequency and acceptance criteria. A known amount of analyte is added to an aliquot of a randomly chosen sample. For projects requiring DoD protocol or when QAPPs require, the full list of analytes are

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spiked into LCS/Ds and MS/Ds. Refer to QAM and DoD QSM for additional guidance. The calculation of the percent recovery is as follows:

$R = (Cs – C)/s * 100$

R = percent recovery
Cs = Fortified sample concentration
C = Sample background concentration
s = Concentration equivalent of analyte added to the sample

11.3.1 If the sample matrix spike fails, the LCS results are reviewed to determine whether the failure is matrix specific or an analytical problem. If found to be matrix specific, the report for the spiked sample is noted with this failure.

11.4 Matrix Duplicate: Refer to determinative method SOP for frequency and acceptance criteria. The RPD is calculated as follows:

$RPD = (D1-D2)/((D1+D2)/2) *100$

D1 = The initial result of the analyte
D2 = The duplicate result of the analyte

11.5 Surrogates: Surrogates are added to all samples. Samples failing these criteria are reanalyzed. If the reanalysis shows acceptable recoveries, these results are reported. If the sample reanalysis shows a similar failure, the sample is re-extracted. If no additional sample remains for re-extraction, the failing results are reported, with a notation of the failure and possible cause.

12. **Retention Times**

12.1 Refer to determinative method SOP.

13. **Responsibilities**

13.1 The analyst uses the Quality Control Criteria outlined in this SOP to evaluate and validate the data. It is the analyst's or technician's responsibility to follow the quality control criteria in this SOP and record all the data in the lab notebook. Any irregularities within the test or samples should be recorded in the lab notebook and reported to the lab manager who is responsible for reviewing the data.

14. **Health and Safety**

14.1 Normal, accepted laboratory safety practices should be followed during reagent preparation and extraction. The spiking solutions contain compounds that are suspected carcinogens and should be handled accordingly. Every sample in the lab should be handled as if it is hazardous waste. Refer to Chemical Hygiene Plan (SOP QA604), and SDS sheets.

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15. **Pollution Prevention and Waste Management**

15.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. All waste generated as a result of this method is disposed of in labeled drums that are stored until full and picked up by a company dealing in disposal of hazardous material.

15.2 The EPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Waste management in this laboratory is regulated by the Safety director. The aqueous waste left over from the extraction is stored in drums with labels describing the contents. The standards are generally used in their entirety, but in the instances where they expire before they are gone, they are stored in labeled drums to be picked up by a disposal company. The 1.5mL vials containing the extracts are kept under refrigeration for 40 days after analysis. After the 40 days they are disposed of in labeled drums to be picked up by a disposal company.

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Table 1 : Labconco Rapid-Vap Settings for "Program 1"

PROGRAM 1

Speed= 55%
Heat= 42° C
NO_2= 15 psi
Time= 25 minutes to start
Sample No.= Number of Rapid-Vap glassware to be concentrated at one time

PROGRAM 2

Speed=55%
Heat=62°C
NO2=15psi
Time= 10 minutes to start
Sample No.= Number of Rapid-Vap glassware to be concentrated at one time

Table 2: Surrogate and Spike Amounts

Method	Surrogate	Spike
EPA8270	0.1mL	0.4mLABN/ 0.2mL PAH
EPA8082	0.1mL	0.1mL
EPA8081	0.1mL	0.1mL
EPA8015	0.1mL	0.05mL

Note: Table 2 contains typical volumes of solutions added to samples and QC. Depending on concentrations of stock spikes and surrogates, volumes used should be discussed with the determinative method analyst and recorded in the appropriate extraction logbooks.

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Table 3: pH Extraction Conditions for Various Determinative Methods

Determinative Method	Intial Extraction pH	Secondary Extraction pH
8081	5-9	none
8082	5-9	none
8270	<2	>11

ABSOLUTE RESOURCE ASSOCIATES
Standard Operating Procedure QA-5600

Title: Mercury Analysis by Cold Vapor Methods 245.1, 7470A, 7471B

QA Officer: Jennifer Guerrette Date: 8/1/17

Laboratory Director: [signature] Date: 8/2/17

Author: [signature] Date: 8/1/17

Analyst: _____ Date: _____

By signing this SOP, the analyst has read and understood the latest version of the test method; has read and understood this SOP; acknowledges receipt of this SOP; and is responsible for the implementation of this SOP.

Revision History:

Revision	Changes	Date
2	New format	
3	Technical Corrections on Reagents	9/17/03
4	Reference to Corrective Action in QA Manual	1/04
5	Minor Edits, matrix specific QC	8/05
6	Minor edits and clarifications; updates on use of Aspen/LIMS .	9/07
7	Added method reference 7470A, Use of LL verification, IDL definition	5/09
8	Procedure for Cetac Instrumentation	2/10
9	Added method reference 7471B, and sample calculations for CETAC	12/10
10	Updated Co name, Sample volume 7471 and conc. acids daily calibration	7/11
11	Minor edits and clarifications	1/13
12	LOD/Q and MDL location added, Updated blank info for solid matrices.	3/14
13	Updated balance sensitivity (4.1.1), updated sections: 6.3.2 (corrected reagent reference), 6.3.7 (added agitate samples), removed references to LL verification	1/15
14	6.3.1 added to clarify the balance requirements, clarified 6.7, added MS acceptance criteria 7.2.6, clarified LCSD 6.1.1, added MSD to 7.2.7, added method performance to 7.1	3/16
15	Definition fixes, MDL frequency clarification	2/17
16	Added DOD requirement: LLCCV shall be analyzed daily	7/17

Title: Mercury Analysis by Cold Vapor Methods 245.1/7470A/7471B

1.0 Purpose and Applicability

1.1 This SOP is designed to determine levels of mercury present in a variety of matrices.

1.2 This method is applicable to aqueous and solid samples. Aqueous samples include industrial wastewater, surface water, groundwater, etc. Solid samples include soil, sediment, sludge, etc.

1.2.1 This method is for use only by or under the supervision of analysts experienced in the use of Cold Vapor AA and in the interpretation of the resulting data. Each analyst must demonstrate the ability to generate acceptable results with this method.

1.3 The reporting limit for water samples is 0.0002mg/L and approximately 0.04 µg/g for solid samples. Solid sample reporting limits are dependent on the percent moisture of the sample.

2.0 Definitions

2.1 Continuing Calibration Blank (CCB) or Initial Calibration Blank (ICB): A volume of reagent water treated with the same reagents as the calibration standards and samples. The blank is used to determine if there are interferences in the laboratory environment, reagents, or equipment. The blank immediately following the calibration curve is identified as the ICB. The blank analyzed periodically during sample analysis is identified as CCB.

2.2 Initial Calibration Verification (Instrument Performance Check) (ICV): A standard analyzed at a mid-point in the calibration curve to verify the integrity of the curve. The ICV is made from a second source standard or at the least, have a different lot number than the one that made the calibration curve.

2.3 Continuing Calibration Verification (CCV): A QC sample spiked with the calibration standard which is used to validate the calibration throughout the sequence. It concentration should be varied throughout the sequence.

2.4 Calibration Standard (ICal): Standard solutions prepared from the stock standard at levels corresponding to the calibration curve. These are used to calibrate the instrument response with respect to analyte concentration.

2.5 Field Duplicates: A duplicate sample taken in the field. These are done at the customer's request.

2.6 Laboratory Control Sample/Duplicate (Laboratory Fortified Blank) (LCS/D): An aliquot of reagent water to which known quantities of the mercury are added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

2.7 Certified Reference Material/Duplicate (CRM/D): A solid standard purchased from a reputable supplier that contains a documented concentration mercury. This standard is used as the LCS and LCSD for Method 7471B. The CRM/D is analyzed exactly as a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate measurements.

2.8 Prep Blank (Laboratory Reagent Blank) (PB): An aliquot of reagent water or other blank matrices (such as SiO_2) that is treated as a sample including exposure to all glassware, equipment, and reagents that are used with other samples. The PB is used to determine if the method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. The PB results must be below the reporting limit for the analyte.

2.9 Linear Calibration Range (LCR): The concentration range over which the instrument response is linear and calibrated.

2.10 Quality Control Sample (QCS): See definition for ICV.

2.11 Stock Standard Solution (SSS): A concentrated solution of mercury prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source, i.e. LabChem or Ultra Scientific. Documentation of the purity of the SSS is retained for traceability purposes in the Metals Certificate of Analysis Binder.

2.12 Matrix Spike: An aliquot of a sample to which a known amount of mercury from the second source standard is added. It is analyzed exactly like a sample and is used to determine whether the sample matrix contributes to bias in the analytical results.

2.13 Instrument Detection Limit (IDL): A useful means to evaluate the instrument noise level and response changes over time. It can be estimated by calculating the average of the standard deviations of three runs on three consecutive days from the analysis of a reagent blank with seven consecutive measurements per day.

3.0 Applicable Documents/References

3.1 Test Methods for Evaluating Solid Waste Physical/Chemical Methods, US EPA SW 846, 3rd edition: Method 7470A and Method 7471B.

3.2 40 CFR 136 Method 245.1.

3.3 QuickTrace M-6100 Mercury Analyzer Operator's Manual Version 1.1

3.4 CETAC ASX-260 AutoSampler Operator's Manual

3.5 QuickTrace Mercury Analyzer Software Manual

3.6 ARA SOP QA-400

3.7 ARA QA Manual

4.0 Materials and Apparatus

Title: Mercury Analysis by Cold Vapor Methods 245.1/7470A/7471B

4.1 **Equipment:**

- 4.1.1 Analytical balance, capable of accurately weighing to the nearest 0.01g.
- 4.1.2 Scoopula or clean wood popsicle sticks
- 4.1.3 Environmental Express 50mL disposable digestion tubes with screw caps
- 4.1.4 CPI International ModBlock digestion system heated to 95 degrees Celsius
- 4.1.5 CETAC QuickTrace Mercury Analyzer M-6100
- 4.1.6 Medicine cups
- 4.1.7 Kim-Wipes
- 4.1.8 Glass or disposable pipettes
- 4.1.9 0.1µL to 10,000µL Eppendorf Pipettors
- 4.1.10 Class "A" volumetric flasks, assorted volumes
- 4.1.11 Environmental Express SC0408 FilterMate Filtration devices
- 4.1.12 Compressed nitrogen gas cylinder providing ultra-high purity, dry, research grade N2.

4.2 **Reagents/Standards:**

- 4.2.1 DI Water: Supplied by Hydro UV Plus pump.
- 4.2.2 Concentrated Nitric Acid: trace metals grade purchased from Fisher Scientific (or equivalent);
- 4.2.3 2% Nitric Acid: 20 mL of concentrated nitric acid diluted to 1000mL of DI Water.
- 4.2.4 Concentrated Sulfuric Acid (H_2SO_4): Purchased from Fisher Scientific or equivalent
- 4.2.5 Mercury Standard: purchased from VHG Labs (or equivalent) at a concentration of 1000µg/mL in 2 – 10% nitric acid solution.
- 4.2.6 Potassium permanganate solution: stock purchased from Fisher Scientific (or equivalent) from which a working solution is prepared by dissolving 25g into 500mL DI water.
- 4.2.7 Potassium persulfate solution: stock purchased from Fisher Scientific (or equivalent) from which a working solution is prepared by dissolving 40g into 800mL of DI water in a hot water bath.
- 4.2.8 Hydroxylamine hydrochloride solution: stock purchased from Fisher Scientific (or equivalent) from which a working solution is prepared by dissolving 60g into 500mL of DI water.
- 4.2.9 Concentrated Hydrochloric Acid: trace metals grade purchased from Fisher Scientific (or equivalent)
- 4.2.10 Stannous chloride: stock purchased from Fisher Scientific (or

equivalent) made into a reagent solution prepared by dissolving 10g $SnCl_2$ and 7mL HCl into a partially filled 100mL volumetric flask, then brought up to 100mL with DI Water.

4.2.11 Hg Media solution is made by adding 2mL H_2SO_4, 1mL HNO_3, 6mL of prepared $KMnO_4$ reagent, 3.2 mL prepared K_2SO_8, and 2.4 mL prepared Hydroxylamine Hydrochloride to 40mL DI Water in a 50mL DigiPrep tube

4.2.12 QuickTrace rinse solution: 1% HCl / 2% HNO_3 solution made by diluting 20mL HCl and 40mL HNO_3 to 2 L DI Water

4.2.13 Aqua Regia: Prepare immediately before using by carefully adding 3 volumes of concentrated HCl to 1 volume concentrated HNO_3 in a fume hood. Discard after the days use.

5.0 Method Summary

5.1 Samples are digested in a potassium permanganate solution and oxidized at 95 degrees Celsius. Once cool and following the addition of Hydroxylamine Hydrochloride, stannous chloride is introduced to the sample in the QuickTrace Analyzer which reduces any mercury present to elemental mercury, which is transferred to a sample cell in the path of light from a mercury vapor lamp. The resulting absorbance of the mercury vapor is compared to a curve of absorbance v. concentrations to determine the concentration in the sample.

5.1.1 Sample Collection, Preservation and Storage: Aqueous samples are collected in previously cleaned polyethylene bottles which are preserved with HNO_3 to bring their pH below 2. Aqueous samples do not require refrigeration. Solid samples are collected in glass jars and stored at 4 degrees Celsius.

5.1.2 The holding time for mercury analysis is 28 days from the date of collection.

5.1.3 Shipment of samples must follow the preservation requirements of the method.

5.2 Calibration Procedure

5.2.1 The instrument must be calibrated each day it is in use.

5.2.2 The SSS-**A** (see 2.10) is made by diluting the stock mercury standard by a factor of 100. This is done by adding 50µL of the standard to 4.95mL of 2% nitric acid.

5.2.3 The SSS-**B** is made by diluting SSS-**A** by a factor of 10 (making a final overall dilution factor of 1000), done by adding 0.5mL of SSS-**A** to 4.5mL of 2% nitric acid. The SSSs are serially diluted to create the

Title: Mercury Analysis by Cold Vapor Methods 245.1/7470A/7471B

calibration curve using the Hg media.

5.2.4 Suggested standard CAL levels are:

Level	ICal(µg/L)	Vol. SSS (µL)	Vol. Hg Media (mL)	Dilution
1	10.0	40 µL **(A)**	40	1000
2	5.0	20 µL **(A)**	40	2000
3	2.5	100 µL **(B)**	40	4000
4	1.0	40 µL **(B)**	40	10,000
5	0.2	8 µL **(B)**	40	50,000

5.2.5 The lowest non-zero point in the curve is at the reporting limit. The absorbance value provided by the instrument is measured by the software against the calibration curve generated by the above calibration standards. The current method and the curve used for calculating the results are stored in the CETAC software as the present analysis date. Linear regression is used.

5.2.6 Calibration Verification Requirements: The calibration curve must have a correlation coefficient (r^2) of 0.998 or greater. An ICV and an ICB must be run immediately following the calibration curve. The ICV must be recovered at ±10% the expected value. The ICB result must be below the reporting limit. These requirements must be met before using a new calibration curve.

5.3 **Interferences**

5.3.1 Interferant 1: Samples containing high levels of sulfide, chloride, copper and tellurium have been shown to affect readings since they readily absorb around the same wavelength. (253.7nm). The QuickTrace Mercury Analyzer has fixed optical interference filters to prevent or minimize this interference

5.3.2 Interferant 2: Laboratory environment conditions with high background levels of mercury can lead to low level false positives. Be cautious of prepping mercury immediately following the prep of chemical oxygen demand which uses mercuric sulfate as a reagent.

6.0 **Procedure**

6.1 **Sample Preparation**

6.1.1 Each batch of up to 20 samples (aqueous or solid) of mercury digestions must contain a matrix matched preparation blank and a matrix-matched

LCS and LCSD. A matrix spiked sample is prepared every 10 samples and a sample duplicate every 20 samples. A batch is valid for 24 hours.

6.2 Methods 245.1 and 7470A:

6.2.1 Obtain 50mL pre-cleaned disposable digestion tubes with screw caps and add 40mL of aqueous sample to the bottle. Label the tube with the appropriate lab ID designation. Record exact volumes/weights and all other pertinent information in the mercury prep logbook.

6.2.2 Add 2.0mL of concentrated sulfuric acid to each tube.

6.2.3 Add 1.0mL of concentrated nitric acid to each tube.

6.2.4 Freshly prepared spiking solution is added to the LCS, LCSD, and matrix spike samples. Add 80µL SSS-**(B)** prepared with the second source standard to the LCS/D and MS. (True Value = 2.0 ug/L)

6.2.5 Add 6.0 mL of prepared potassium permanganate to each tube.

6.2.6 Add 3.2 mL of prepared potassium persulfate to each tube.

6.2.7 Cap the tubes and place in the previously preheated ModBlock digester to digest the samples at 95 degrees Celsius for 2 hours.

6.2.8 Remove from ModBlock, immediately loosen the caps, and allow samples to cool to room temperature.

6.2.9 Add 2.4 mL Hydroxylamine Hydrochloride to each tube, recap and shake until potassium permanganate clears. (Caution should be taken that sample tubes don't leak due to the increased internal pressure of the reaction.)

6.2.10 The samples are ready for analysis. Sample filtration maybe required based on the analyst's judgement as to whether sufficient suspended solids are present that may clog the instrument tubes or lines. If any samples are filtered, all QC samples must also be filtered.

6.3 Method 7471B:

6.3.1 Place the 0.1g calibration weight on the balance and confirm that the value is within 10% of the true value.

6.3.2 In 50mL labeled digestion tubes, prepare solid samples. Use 0.25g. SiO_2 for the PBs, and 0.25g CRM and CRMD. Use 0.2-0.25g well homogenized sample for solid samples.

6.3.3 Add 2mL DIH2O to each tube

6.3.4 Add 2mL aqua regia (reagent 4.2.13) to each tube

6.3.5 Cover and heat 2 minutes in the previously preheated ModBlock set to 95 $\pm$ 3 degrees C, then cool

6.3.6 Add 20mL DIH_20 and 6mL potassium permanganate (4.2.6) solution to each sample.
6.3.7 Recap and mix thoroughly, then heat for 30 min. at 95 $\pm$ 3 degrees.
6.3.8 Remove from ModBlock and immediately loosen the caps.
6.3.9 Allow samples to cool in a fume hood, then add 2.4mL hydroxylamine hydrochloride (4.2.8) to reduce the excess permanganate.
6.3.10 Add 20mL DIH2O and agitate samples to mix and complete reaction (Caution should be taken that sample tubes don't leak due to the increased internal pressure of the reaction.)
6.3.11 Solid samples should be allowed to settle prior to analysis. Alternatively the tubes may be centrifuged at the lowest setting for a few minutes.
6.3.12 Sample filtration maybe required based on the analyst's judgement as to whether sufficient suspended solids are present that may clog the instrument tubes or lines. If any samples are filtered, all QC samples must also be filtered. Use the SC0408 FilterMate Filtration devices.
6.3.13 The samples are ready for analysis.

6.4 **Instrument Setup** (see manual for additional instructions and details)
6.4.1 Turn on the nitrogen tank (should be ~40 psi)
6.4.2 At least ½ hour before use, power on the mercury analyzer, peristaltic pump, and the autosampler. Verify that the Gas-Liquid Separator (GLS) line is disconnected.
6.4.3 Check volumes of rinse solution, waste container, $SnCl_2$ and DI Water. (If the $SnCl_2$ reagent has precipitated, crystallized, yellowed or oxidized, replace with a fresh solution.) Ensure waste container has adequate room to receive the instrument waste.
6.4.4 Launch the CETAC software
6.4.5 Wet the GLS. Place the sipper tubing in the DI Water, and attach the top 2 channels of the pump. Wait for the bubble to rise to the top of the GLS, then quickly attach bottom 2 channels to start draining the GLS. When the water is drained completely down, carefully attach the black GLS tubing to the glass post.
6.4.6 In the QuickTrace software, open the template labeled "MASTER" and rename and save as the current date in mmddyy format. Enter analytical sequence.
6.4.7 Uncap the calibration standards.
6.4.8 Place the stannous chloride line in the stannous chloride reagent.
6.4.9 Press the "Go" button to begin calibrating the instrument. Once the

calibration and verification standards have been evaluated, analysis of samples may begin.

6.5 **Analysis**

6.5.1 To export the data acquired by the CETAC Mercury Analyzer

6.5.1.1 Click on the report icon on the top of the screen; choose the worksheet to be reported, and choose the "save to disk" icon on the right of the screen. This will export the saved file to an export folder.

6.5.1.2 Save the resulting file as the run date in mmddyy format

6.5.1.3 Open the Hg CETAC file. Run the macro "CETAC copier" on the previously exported file when prompted.

6.5.1.4 Copy appropriate data onto the import tab and remove the instrument QC (CCV, CCB, etc)

6.5.1.5 Save the Excel spreadsheet as the prep batch number and matrix, if multiple prep batches were run, there should be a file for each prep batch. IMPORTANT: highlight the QC code cells that are <u>empty</u> and delete them all.

6.5.1.6 Import the data into the Aspen LIMS system for generating reports.

6.6 Instrument Shut-Down

6.4.1 Move sipper from $SnCl_2$ reagent to DI Water, and in "Instrument" raise the sipper

6.4.2 Run ~ 10 minutes to flush, then take sipper out of the water to dry the lines for ~ 10 minutes.

6.4.3 Turn off the lamp, pump and autosampler. Cap standards and waste jug

6.4.4 Unhook the GLS tubing and peristaltic pump tubing

6.4.5 Turn off nitrogen gas.

6.7 **Sample Calculations**

6.7.1 Dilutions are noted where they are done either in the prep worksheet or the analysis worksheet. (If there is a dilution, the result and the reporting limit are multiplied by that factor in the report.) For solid samples, the concentration is calculated (in LIMS) based on the dry weight of the sample. The following equation is used to calculate the concentration of an analyte in a solid:

$$\text{Concentration } (\mu g/g) = \frac{C \times V}{W \times S}$$

Where, C = concentration of analyte in extract (µg/mL)
V = volume used in digestion (mL)
W = weight of material (g, as received)
S = fraction dry matter

7.0 Quality Control Requirements

7.1 Method Performance

7.1.1 Initial Demonstration of Performance: This is used to characterize instrument performance (linear calibration ranges and analysis of the ICV), analyst performance and laboratory performance (MDLs) prior to use of this method. The Demonstration of Capability as described in the QA Manual is followed prior to use of this method by an analyst.

7.1.1.1 Linear Calibration Range: This is the criteria of the calibration curve. Must be a minimum of five standards and verified at ±10% by a second source. The correlation coefficient must be 0.998 or higher.

7.1.1.2 Initial Calibration Verification: The ICV must be from a second source and show recovery within ±10% of the true value.

7.1.1.3 Low-level Calibration Check Standard: For DoD compliant samples, an LLCCV must be analyzed daily. The LLCCV recovery must be within ±20% of the expected value. The concentration should be less than or equal to the LOQ. If the concentration of the lowest calibration standard is less than or equal to the LOQ, the lowest standard may be re-quantified against the calibration curve as a LLCCV. Otherwise, a separate standard must be analyzed as the LLCCV prior to the analysis of any samples.

7.1.1.4 Method Detection Limit: The MDL must be established for all analytes. Reagent water is fortified at a concentration of two to three times the estimated method detection limit. To determine the MDL, take seven replicates of the fortified reagent water and process through entire analytical procedure (the same preparation process as the LCS). Calculate the values of the MDL for each analyte as follows:

MDL = (t)*(s)

t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. For seven replicates

t = 3.14.

s = Standard deviation of the replicate analysis.

The aqueous and solid MDLs should be determined whenever there is a significant modification to the procedure or analytical system. The MDL data are stored electronically in the QA/MDL file folder.

7.1.2 LOD/LOQ: Refer to QAM *Analytical Procedures* section, for specific LOD/LOQ requirements. LOD/LOQ data are stored electronically in the QA/LOD&LOQ file folder.

7.1.3 IDC/CDC: Proficiency of the analyst is ensured by documentation of an Initial and Continuing Demonstration of Capability (IDC or CDC). Typically, the analyst analyzes four LCS's with acceptable precision and accuracy or shows acceptable performance of a blind sample analysis (usually a PT). See QAM for additional information.

7.1.4 Control Charts: Control Charts are quality control tools which graphically display QC data over time. The data required for generation of accuracy and precision control charts is maintained by the laboratory. See QAM for additional information.

7.1.5 Inter-laboratory performance: Proficiency Test studies, conducted by approved providers, constitute the majority of inter-laboratory performance comparisons. Proficiency tests are performed at a frequency as required by method, regulation, or accrediting body. See QAM for additional information.

7.2 **Laboratory Performance Quality Control and Corrective Actions**

7.2.1 Corrective action guidance is provided in the SOP as follows. Additional corrective action guidance can be found in the QA Manual.

7.2.2 Continuing Calibration Verification: The CCV recovery must be within ±10% of the expected value. The CCV is analyzed after ten samples and at the end of a sequence. If the CCV performed after 10 samples is out of control, stop running samples. Locate the source of the problem and re-analyze the CCV. If the CCV is now within ±10% of the expected value, re-analyze all the samples run after the last passing CCV.

7.2.3 Continuing Calibration Blank: The CCB is analyzed at the same frequency as the CCV. The CCB must be below the reporting limit for the analyte. If the CCB is not below the reporting limit, steps must be taken to determine the source of the problem. Corrective actions may

include re-analysis of the CCB with fresh reagent water to trace the contamination. If there is suspected data impact (discuss with the QAO), the samples may be reanalyzed or the data qualified to disclose the suspected impact to the customer.

7.2.4 Prep Blank: One PB is analyzed per matrix in every set of up to 20 samples or 24 hours, whichever is reached first. The PB undergoes the sample preparation process. The PB must be below the reporting limit. Follow the same corrective action steps as the CCB.

7.2.5 Laboratory Control Sample/Duplicate (LCS/D): One LCS and LCSD is analyzed per matrix in every set of up to 20 samples or 24 hours, whichever is reached first. The percent recovery is calculated and must be within ±20% of the expected value for fortified reagent water and within the vendor control limits (95% confidence limit) for solid reference materials (CRM). The relative percent difference between the LCS and LCSD must be ±20%. The relative percent difference between the CRM and CRMD must be ±30%. If the LCS/D or CRM/D is out of control, the source of the problem must be identified and the QC re-analyzed before continuing onto samples.

7.2.6 Matrix Spike: A matrix spike is prepared and analyzed every 10 samples. A known amount of analyte is added to an aliquot of a randomly chosen sample. The percent recovery for the MS should be 80-120%. Samples outside of this range should be documented and investigated. The calculation of the percent recovery is as follows:

$$R = 100 * (Cs - C)/S$$

R = percent recovery
Cs = Fortified sample concentration
C = Sample native concentration
S = Concentration equivalent of analyte added to the sample

7.2.7 Duplicate: A sample duplicate is prepared and analyzed every 20 samples and may also be run upon request of the customer. Matrix Spike Duplicates are run per customer request and can be substituted for a duplicate sample. The relative percent difference must be ±20% for waters and ±30% for solids. The RPD is calculated as follows:

$$RPD = 100 * (D1-D2)/((D1+D2)/2)$$

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D1 = The initial result of the analyte
D2 = The duplicate result of the analyte

8.0 **Responsibilities**

8.1 The analyst uses the Quality Control Criteria outlined in this SOP to evaluate and validate the data. The analyst or technician's responsibility is to follow this SOP and record all the data in the lab Mercury Prep and Mercury Run-Log notebooks. Any irregularities within the test or samples should be recorded in the lab notebooks and reported to the lab manager who is responsible for reviewing the data. Regular maintenance of the Cold Vapor AA includes wiping up all spills and replacing any tubing that is fatigued, worn or appreciably "flattened". The Nafion dryer cartridge should be replaced if there is a loss of mercury absorbance sensitivity and an increase in the baseline of more than 1000uabs during a short run of 30 minutes or less. A record is kept of all other instrument problems or necessary maintenance in the CVAA Maintenance Logbook stored under the Mercury Analyzer.

9.0 **Health and Safety**

9.1 Normal, accepted laboratory safety practices should be followed during reagent and sample preparation and instrument operation. Every sample in the lab should be handled as if it is hazardous waste. Refer to Chemical Hygiene Plan (SOP QA604), and SDS sheets.

10.0 **Pollution Prevention and Waste Management**

10.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Waste generated from the analysis of mercury is collected by itself in a satellite waste container which must remain capped at all times unless in a fume hood. Once full, the satellite waste container is held in the Hazardous Waste room until it is picked up by an approved company for the disposal of the waste.

10.2 The EPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Waste management in this laboratory is regulated by the Safety director. Stannous chloride is added to any standards that are left over from the analysis of mercury when they are disposed of in the satellite waste container along with the analyzed samples.

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Typical Analytical Sequence after Calibration, With Acceptance Criteria
Mercury Analysis

Sample Number	Instrument Run Log	Acceptance Limit
	Blank	
	Ical (5 point min)	R2=0.998
	ICV	± 10%
	ICB	<RL
	LLCCV (for DoD samples)	± 20%
	PB	<RL
	PBs	<RL
	LCS (aqueous)	80-120%
	LCSD (aqueous)	RPD ≤20%
	CRM (solid)	95% vendor confidence limits
	CRMD (solid)	RPD ≤30%
1	Sample 1	
2	Sample 2	
3	Sample 3	
4	Sample 4	
5	Sample 5	
6	Sample 6	
7	Sample 7	
8	Sample 8	
9	Sample 9	
10	Sample 10	
10ms	Sample 10 spike	± 20%
	CCB	<RL
	CCV	± 10%
11	Sample 11	
12	Sample 12	
13	Sample 13	
14	Sample 14	
15	Sample 15	
16	Sample 16	
17	Sample 17	
18	Sample 18	
19	Sample 19	
20	Sample 20	
20dup	Sample 20 Duplicate	RPD ≤20%W / ≤30%
20ms	Sample 20 spike	± 20%
	CCB	<RL
	CCV	± 10%

Title: Determination of Metals and Trace Elements in Water, Solids and Wastes by Inductively Coupled Plasma-Mass Spectrometry by 200.8/6020A

QA Officer: _____ Date: 8/3/17

Laboratory Director: _____ Date: 8/3/17

Author: _____ Date: 8/3/17

Analyst: _____ Date: _____

By signing this SOP, the analyst has read and understood the latest version of the test method; has read and understood this SOP; acknowledges receipt of this SOP; and is responsible for the implementation of this SOP.

Revision History:

Revision	Changes	Date
1	Updated DD calibration requirement, clarified 200.8 analysis titles, updated definition of ICV (different manufacturer), changed title of Table 1, added solid reporting limits to Table 2, 6.1.3.2 added to clarify the balance requirements, clarified dilution test requirements 7.2.7, updated RLs	1/16
2	LCSD RPD added to 7.2.4, clarified method performance sec 7.1, updated solid reporting limits	3/16
3	Internal standard monitoring clarification. Addition and clarification of 200.8, 6020a(Rev1), manufacturer, and internal requirements throughout the body of the SOP. RL and LDR updates.	02/17
4	IS criteria chngd and clarified for each reg req. (DoD, MCP).See sec 7.2.5. Ending ccv clarification for 200.8 in 7.2.1.	7/17

Title: Trace Metals By ICPMS EPA 200.8/6020A

Purpose and Applicability

1.1 This method covers the determination of the metals by Inductively Coupled Plasma-Mass Spectrometry. See Table 1.0 for the list of metals analyzed by this method and the associated reporting limits. Other metals may be added based on customer request.

1.2 The matrices applicable are as follows: Drinking Water, Surface Water, Mixed Domestic and Industrial Wastewaters, Groundwater, Reagent Waters, and Solids (soils, sludges, and sediments after digestion), Paint Chips, Dust Wipes, and Air Filters.

1.3 This method is recommended for use only by or under the supervision of analysts experienced in the use of ICPMS for trace metals and in the interpretation of the results. Each analyst must demonstrate the ability to generate acceptable results with this method.

1.4 The table below shows the possible digestion and analysis methods for samples applicable to this SOP. The primary digestion method is the most routinely utilized method, however, the secondary digestion method may also be utilized as necessary or as requested by the customer or contract.

Matrix	Primary Digestion	Secondary Digestion	Analysis Method
Discharge Water	SW3005A		200.8
Drinking Water	None*	3005A	200.8
Groundwater	SW3005A		6020A
Soil/Sludge	SW3051A	3050B	6020A
Paint Chips	SW3051A		6020A
Air Filters	NIOSH 7303	SW3051A	6020A
Dust Wipes	SW3051A		6020A

*Direct Analysis of total recoverable analytes is performed on samples when turbidity is verified to be <1 NTU.

2.0 Definitions

2.1 Batch: A group of at most 20 samples of similar matrix (aqueous, solid, paint chips, etc.) which are extracted and analyzed together with the same method and the same lots of reagents within a 24 hour time frame.

2.2 Calibration Standard: Solutions prepared from the stock standard solutions. These are used to calibrate the instrument response with respect to analyte concentration.

2.3 Calibration Blank (ICB or CCB): A volume of reagent water acidified with the same matrix as the calibration standards. The blank is used to determine if there are interferences in the laboratory environment, reagents, or equipment.

2.4 Continuing Calibration Verification (CCV): A QC sample spiked with the calibration standard which is used to validate the calibration throughout the sequence, within the Linear Dynamic Range. It must be varied throughout as appropriate in relation to the sample concentrations.

Title: Trace Metals By ICPMS EPA 200.8/6020A

2.5 Dilution Test: A second analysis of samples with sufficiently high concentrations, at a 1:5 dilution to check precision, and confirm that there is no interference effect.

2.6 Dissolved Analyte: The concentration of an analyte in an aqueous sample that will pass through a 0.45 micron membrane filter assembly prior to sample acidification.

2.7 Initial Calibration Verification (ICV)(QCS for 200.8): A QC sample spiked with the analytes of interest from a second source standard. This sample is used to verify the integrity of the curve. The concentration is near the mid-point in the calibration curve. The second source standard is, at a minimum, from a standard with a different lot number than the one used to make the calibration curve and typically is from a different vendor. This standard also satisfies the requirement for analyzing a Quality Control Sample (QCS) to verify calibration set forth in the 200.8 method.

2.8 Field blank: an aliquot of reagent water or blank matrix collected by field personnel in a sample container and analyzed at the customer's request to determine contamination in the field.

2.9 Field Duplicates: A duplicate sample taken in the field. These are analyzed at the customer's request.

2.10 Instrument Detection Limit (IDL): A tool to evaluate the instrument noise level and response changes over time for each analyte from a series of reagent blank analyses to obtain a calculated concentration.

2.11 Instrument Performance Check (IPC) Solution: the analysis of the calibration blank and at least one calibration standard immediately following each calibration routine, to determine whether the calibration remains valid. This is a requirement for method 200.8.

2.12 Internal Standard (IS): Pure analyte added to standard solutions and samples in known amounts and used to measure the relative responses of other method analytes that are components of the same sample or solution. The internal standard must be an analyte that is not a sample component.

2.13 Interference Check Sample (ICS): a QC sample that contains known concentrations of interfering elements that will demonstrate the magnitude of interferences and provide an adequate test of any corrections. Typically two solutions are prepared, an "A" and "B".

2.14 Laboratory Control Sample (LCS) (Lab Fortified Blank in 200.8): An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly like a sample, and 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 of the LCS should match the matrix of the samples (i.e.: aqueous, solid, paint chips, etc) whenever a reasonable standard is available.

2.15 Laboratory Duplicates (DUP): Two aliquots of the same sample that is analyzed separately with identical procedures. Analysis of sample and DUP indicates precision

associated with the laboratory procedures. The result is not an indicator of the acceptability of sample collection, preservation, and storage procedures.

2.16 Linear Dynamic Range (LDR): The concentration range over which the instrument response is linear and calibrated for.

2.17 Low Level Check Standard (LLCS) (LLICV and LLCCV in 6020A, IPC 200.8): A QC sample made from the Calibration standard with a concentration at one to two times the lower limit of quantitation. This standard is analyzed after initial calibration and at the end of the run at a minimum to verify accuracy at the reporting limit.

2.18 High Level Check Standard (HLCS): Standard made from the calibration SSS that is run at the analyst's discretion to verify quantitation at a higher level than the calibration curve. This standard is typically run at the end of the analytical sequence.

2.19 Lower Limit of Calibration Check Sample (LLQC) (requirement of 6020A- Same as the LOQ verification): A QC sample spiked with a verified standard at one to two the quantitation limit. It is carried through the entire preparation and analytical procedure and is used to both establish and confirm the lowest quantitation limit.

2.20 Low Level Continuing Calibration Verification (LLCCV) (requirement of 6020A- same as LLCS) : A QC sample made from the calibration standard with a concentration at one to two times the lower limit of quantitation, analyzed during the analytical batch to verify accuracy at the reporting limit.

2.21 Matrix Spike (MS): An aliquot of a sample to which known amounts of target analytes from the second source standard are added. It is analyzed exactly like a sample and is used to determine whether the sample matrix contributes to bias in the analytical results.

2.22 Method Detection Limit (MDL): The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero. The reporting limit is generally 2-5 times the MDL.

2.23 Post Digestion Spike: A sample where spike is added after the digestion process, in response to an initial failure of a digested matrix spike on that same sample. It serves to help in the confirmation of matrix effects.

2.24 Preparation Blank (PB)(Laboratory Reagent Blank or Method Blank in 200.8): An aliquot of reagent water or other blank matrices that are treated exactly as a sample, including exposure to all glassware, equipment, and reagents that are used with other samples. The PB is used to determine if the method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

2.25 Quality Control Sample : See ICV for this 200.8 requirement. The mean concentration from three QCS/ICV analyses is determined.

2.26 Rinse Solution: Solution prepared as a rinse blank, which is introduced by the instrument, to flush the system between standards and samples.

2.27 Stock Standard Solution (SSS): A concentrated solution containing one or more target

analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source. Documentation of the purity of the SSS must be retained for traceability purposes in the Metals Certificates of Analysis Binder.

2.28 Total Recoverable Analyte: The concentration of an analyte determined either by direct analysis of an unfiltered acid preserved drinking water sample with a turbidity of <1NTU, or by analysis of the solution extract of solid sample or an unfiltered aqueous sample following digestion.

2.29 Tuning Solution: A solution used to for instrument tuning and mass calibration and resolution checks prior to calibration and sample analysis.

3.0 Applicable Documents/References

3.1 EPA Method 200.8 Determination of Trace Elements in Water and Wastes by ICPMS. Rev. 5.4, EMMC Version.

3.2 EPA SW846, Method 6020A ICPMS Test Methods for Evaluating Solid Waste, Physical/Chemical Methods

3.3 EPA SW846, Method 6020A Inductively Coupled Plasma-Mass Spectrometry Spectrometry. Rev.1

3.4 EPA SW846, Method 3005A. Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by ICP Spectroscopy.

3.5 EPA SW846, Method 3050B. Acid Digestion of Sediments, Sludges, and Soils. (H_2O_2 Method).

3.6 EPA SW846, Method 3051A. Microwave Assisted Acid Digestion of Sediments, Soils and Sludges.

3.7 Shell for Analytical Chemistry Requirements EM 200-1-3, 1 Feb 01

3.8 Standard Methods for Examination of Water and Wastewater, Method 2340B-2011 Hardness by calculation

3.9 ARA SOP QA-400, Sample Receiving and Identification.

3.10 ARA SOP QA-402 Bottle Order Preparation.

3.11 ARA SOP QA-604 Chemical Hygiene Plan

3.12 ARA SOP QA-5001, Laboratory and Sample Waste Characterization and Disposal.

3.13 Perkin Elmer Syngistix Software User Guide, July 2014

3.14 ARA ICPMS Maintenance Log

3.15 ARA QA Manual

3.16 CEM Corporation MARS Microwave Operation Manual, February 2006.

3.17 NIOSH [1993]. Method 7303: Elements by ICP, NIOSH Manual of Analytical Methods (NMAM), Fourth Edition, Issue 1, March 15, 2003.

3.18 ARA SOP QA-5862, Turbidity by SM 2130B and EPA Method 180.1.

3.19 ARA SOP QA-801, Sample Readiness

3.20 ARA SOP QA-5834, Total Solids SM2540B,G

Title: Trace Metals By ICPMS EPA 200.8/6020A

4.0 Materials and Apparatus

4.1 Equipment:

- 4.1.1 Perkin Elmer Nexion 350-X ICPMS
- 4.1.2 Perkin Elmer S-10 Autosampler
- 4.1.3 Analytical Balance capable of accurately weighing to the nearest 0.01g.
- 4.1.4 Graduated Cylinders, 100mL
- 4.1.5 Class "A" pipettes, assorted volumes
- 4.1.6 Class "A" volumetric flasks, assorted volumes
- 4.1.7 Disposable Short Stem Funnels and Whatman #5 Filter paper (or equivalent)
- 4.1.8 Environmental Express FilterMates
- 4.1.9 50mL pre-cleaned disposable digestion tubes
- 4.1.10 50mL acid washed glass digestion tubes
- 4.1.11 Microwave vessels
- 4.1.12 Disposable watch glass covers
- 4.1.13 Liquid Argon with Gas Valve (99.99% pure) stored outside in a MultiBulk tank.
- 4.1.14 CPI International Mod Block, Block Digestion System (temperature to be calibrated quarterly)
- 4.1.15 MARS Microwave Digestion System with manufacturer approved acid washed vessels for 3051A.
- 4.1.16 HF Scientific DRT-15CE Portable Turbidimeter (Nephelometer) and Glass sample cells
- 4.1.17 Centrifuge

4.2 Reagents/Standards:

- 4.2.1 DI Water: Supplied by Hydro UV Plus pump.
- 4.2.2 Trace Metal Grade Hydrochloric Acid: Purchased from a reputable supplier. (Note: all diluted acids are to be numbered and tracked in the Metals Standards Logbook.)
- 4.2.3 Trace Metal Grade Nitric Acid: Purchased from a reputable supplier. (Note: all diluted acids are to be numbered and tracked in the Metals Standards Logbook.)
- 4.2.4 2:5:3 Acid Solution: 2 parts reagent 4.2.3:5 parts reagent 4.2.2:3 parts DI Water. Solution is made daily.
- 4.2.5 1% Hydrofluoric Acid Solution: Purchased from a reputable supplier. Used for cleaning ICPMS glass cyclonic spray chamber.
- 4.2.6 10%HNO_3 Acid Wash: 280mL HNO_3 per gallon of DI Water.
- 4.2.7 30% Hydrogen Peroxide: Use only under supervision.
- 4.2.8 Stock Standard Solutions (SSS): All stock metal solutions are purchased from

a reputable vendor.

4.2.9 Prep Blank (PB): A solution that must contain all the reagents in the same volumes as used in the processing of the samples. The PB must be carried throughout the preparation process.

4.2.10 Calibration Blank (CB): Solution is made daily.

4.2.10.1 6020a/200.8WW: Partially fill with container with DI H2O. Add 25 mL reagent 4.2.2 and 10 mL 4.2.3. Bring to 500 mL with DI water.

4.2.10.2 200.8DW: A 100x dilution of reagent 4.2.3 with DI water (1%).

4.2.11 Internal Standard:

4.2.11.1 6020a: Partially fill with DI H2O. Add 25 mL reagent 4.2.2 and 10 mL 4.2.3. In addition, add 2.5mL multi-element (Li, Sc, Tb, Y, In, Bi, and Ho) IS concentrate, 0.1mL Yttrium Concentrate, and 0.25 mL of Scandium concentrate. Bring to 500 mL with DI water.

4.2.11.2 200.8 WW: Same as 6020A IS

4.2.11.3 200.8 DW: 1.25mL multi-element (Li, Sc, Tb, Y, In, Bi, and Ho) IS concentrate and 2.5mL concentrated nitric acid and bring to a final volume of 250mL with DI water.

4.2.12 Initial Calibration Verification (ICV):

4.2.12.1 6020a: Add 0.5mL second source multi-element and 10mL reagent 4.2.4 to minimum DI water and bring to 100mL final volume with DI water.

4.2.12.2 200.8 WW: Same as 6020A

4.2.12.3 200.8 DW: Add 0.5mL second source multi-element and 1.0mL concentrated nitric acid and bring to 100mL final volume with DI water.

4.2.13 Inter-Element Check Standard (ICS): Made from multi-element standards to achieve the final concentrations listed in Table 1.

4.2.14 LLCS, LLCCV: Made by diluting calibration standards to the level of one to two times the QL.

4.2.15 Laboratory Control Samples (LCS/D), Limit of Detection (LOD), Limit of Quantitation (LOQ): Add 0.25ml of second source multi-element SSS, 0.1ml of Calcium SSS to 50ml of DI water and add appropriate reagents for preservation and digestion. LODs and LOQs are spiked with second source SSS to achieve concentrations of 0.0001ppm, 0.001ppm, 0.01ppm, 0.02ppm, 0.1ppm, and 0.5ppm for both solids and waters.

4.2.16 High Level Check Standard (HLCS): Add 1 mL of calibration standard to 19 mL of 4.2.10 to achieve a concentration of 5ppm.

4.2.17 Standard Performance Check, Mass Cal, and KED Standard Performance Check Solutions: Standard and KED Setup solutions are purchased from Perkin Elmer for the purpose of optimizing and tuning the instrument.

4.2.18 Dual Calibration Standards: Standards with concentrations of analyte between 0.2ppm and 1.2ppm are made using SSS and 4.2.10. Internal standards can also be added near concentrations found in 4.2.11. Dual Calibration Standards can be matrix matched to the calibration standards and samples run. This has been shown to improve overall performance for some analytes. 0.2ml Cal standard, 0.1ml Ca std, 0.25ml IS multielement std, 0.01ml Yytrium standard for 6020/WW method only, 10ml 2:5:3(6020/WW) or 1ml HNO3(200.8DW), and bring up to 100ml with DI H2O.

4.2.19 Rinse Solution:

4.2.19.1 6020a/200.8WW: Partially fill with container with DI H20. Add 25 mL reagent 4.2.2 and 10 mL 4.2.3. Bring to 500 mL with DI water.

4.2.19.2 200.8DW: A 100x dilution of reagent 4.2.3 to the desired volume with DI water (1%).

4.2.20 Solid Metals Standard: A certified reference material (CRM), traceable to NIST, and purchased from a reputable supplier, such as RT-Corp cat. No. CRM020-050.

4.2.21 Solid Metals Blank: A certified reference material (SiO_2), traceable to NIST, and purchased from a reputable supplier, such as RT-Cop. Cat. No.: MB060

4.2.22 Pb in Paint chip standard: A certified reference material (CRM), traceable to NIST, and purchased from a reputable supplier, such as RT-Corp cat. No. SQC074-50G

5.0 Method Summary

5.1 This method is used to determine the concentration of metals in aqueous, solid, and filter samples by ICPMS. The samples are combined with an internal standard then nebulized and the resulting aerosol is carried to the plasma torch. The method measures ions produced by the plasma and extracted from the plasma gas through a differentially pumped vacuum interface and separated based on their mass-to-charge ratio by quadrupoles. Depending on the element and method implemented, a collision cell may be used to separate interfering non-target ions from target ions. The response of these separated ions is detected and amplified by a mass spectrometer and then analyzed in Syngistix software by the analyst. The final result are compiled and formatted using reduction software and then results at the instrument are calculated in the Laboratory Information Management System (LIMS). For reporting limits refer to Table 2.0.

5.1.1 Sample Collection, Preservation and Storage: Aqueous samples are collected

in previously cleaned polyethylene bottles which are preserved with HNO_3 (see SOP 402 for preservation requirements and see SOP 400 for sample receiving procedure and requirements). If a sample is to be analyzed directly for dissolved analytes, it must be field filtered prior to preservation with HNO_3. The filter pore size must be 0.45µm. Anytime metals are preserved after collection, the sample must then be held for a minimum of 16 hours. Shipment of samples must follow the preservation requirements of the metals. Solid samples must be stored at 4°C. Air filters, dust wipes, and paint chips as received in the laboratory have no preservation requirements. These solids may be collected in glass or plastic, volume appropriate containers. The laboratory holding time for metals is 180 days from the date of collection.

5.2 **Calibration Procedure**

5.2.1 Calibration is typically performed daily. Calibration must be performed daily for all MCP, DoD, and NLLAP samples prior to analysis.

5.2.2 The Calibration curve includes all metals of interest. Typical reporting limits are listed in Table 2. The lower reporting limit of each metal must be verified by the LLCS. The upper limit of quantitation is established by the LDR. The upper LDR may be verified using the ICS A, typically at 100ppm, if samples analyzed require analysis of elements with naturally high abundance such as minerals. The calibration standard is made from stock standards at the highest level of the calibration curve (typically 0.1ppm, 1.1ppm and 5.1ppm) and then further diluted to create the lower concentrations. This calibration standard is documented in the metals standard prep log. The calibration standard is given an expiration date of one week from date of prep.

5.2.3 Prepare a Calibration blank that matches samples analyzed by the addition of reagent 4.2.4 or nitric acid to DI water at appropriate ratios.

5.2.4 Suggested calibration levels for method 6020A 200.8 WW and 200.8 DW are listed in Table 4. The dilutions for the levels are made as follows:

5.2.4.1 **0.1ppm**: Made from stock standards and given a QA#. (Ag=0.05ppm, Al, Na, K, Ca, Fe, Zn, and Mg = 10.1ppm for 6020/200.8WW and 5.1ppm for 200.8DW, Pb=1.1ppm for 6020/200.8WW and 0.1 for 200.8DW): Fill 100mL volumetric flask partially with DI water, add 10mL of 2:5:3 solution(sec 4.2.4), 0.1mL of 100ppm calibration standard concentrates, 1mL of 1000ppm mineral calibration standard concentrates, and 0.1mL of 1000ppm Pb calibration standard and bring to volume with DI water.

5.2.4.2 **0.05ppm**: 20mL 0.1ppm calibration standard + 20ml CB

5.2.4.3 **0.01ppm or 0.02ppm**: 5mL 0.05ppm calibration standard + 25mL

CCB for 0.01ppm calibration standard. 5mL of 0.1ppm calibration standard + 25 mL CCB for the 0.02ppm calibration standard (200.8DW method only).

5.2.4.4 **0.001ppm**: 2mL 0.01ppm calibration standard + 18mL CB

5.2.4.5 Record the calibration standard in the Metals Batch Checklist.

5.2.5 See Table 2 for a typical element list with reporting limits and linear dynamic range.

5.2.6 Calibration Verification Requirements

5.2.6.1 The calibration curve must have an R^2 value (calibration coefficient) of 0.998 or greater.

5.2.6.2 For 6020A: A mid-level ICV, ICB, LLCS, and ICSs (at LDR concentration for minerals) must be run immediately following the calibration curve. See 7.1.4 for ICV QC requirements and 7.1.5 for ICB requirements.

5.2.6.3 For 200.8 WW and DW: At least one initial calibration standard and calibration blank must be run immediately following the calibration curve as well as a LLCS that checks acceptance at the limit of quantitation. An ICV (called a QCS in 200.8) at a midpoint in the calibration curve is run after each new calibration curve followed by an optional HLCS to check a higher point of the linear range. If this HLCS standard does not pass, then the highest level standard validated by the calibration curve dictates the upper reporting limit for the analytical sequence.

5.2.6.4 These requirements must be met before using a new calibration curve. If any fail, then the standard can be run again to check the accuracy of the first reading. If these standards cannot be verified within the acceptable range, then the curve must be re-analyzed and re-verified. If any are still unacceptable, the problem must be investigated and corrective actions taken, prior to recalibration.

5.3 **Interferences**

5.3.1 Isobaric Elemental Interferences: This is caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio. Elements were chosen to reduce this interference.

5.3.2 Abundance Sensitivity: The degree to which the wings of a mass peak contribute to adjacent masses. May result when a small ion peak is being measured next to a large one. Spectrometer resolution is adjusted to minimize this.

5.3.3 Isobaric Polyatomic Ion Interferences: This is cause by ions consisting of more than one atom that have the same nominal mass-to-charge ratio as the

isotope of interest. This type of interference cannot be accounted for by the spectrometer. Equations are used to correct for this type of interference as well as using the KED cell to analyze the samples (only for 6020A).

5.3.4 Physical Interferences: These are associated with the sample nebulization and transport process as well as with ion-transmission efficiencies. Nebulization and transport processes can be affected if a matrix component causes a change to the surface tension or viscosity of a sample. Samples should not be run if they are >0.2% total solids, this helps reduce the matrix component interferences. An internal standard is also used to help identify physical interferences. The internal standard is monitored and if a suppression of the signal is observed, a dilution is required to properly analyze the sample.

5.3.5 Memory Interferences: Also known as carry-over can occur when there are large concentration differences between samples or standards analyzed sequentially. Sample deposition on the sampler and skimmer cones, spray chamber, and the nebulizer affect the extent of memory interferences. A sufficient rinse between samples reduces this type of interference.

5.3.6 Stability: Samples should be analyzed as soon as possible. Precipitation may occur and affect sample analysis particularly for silver. Samples with concentrations of 0.05 mg/L or higher require HCl preservative and should be digested according to Table 1.4. If inconsistent results for silver are observed upon reanalysis, it is suggested that samples be re-warmed to at least 50°C, cooled and analyzed immediately.

5.3.7 Contamination: If analyzing for very low levels of Na or Ca, acid wash filters using 5 mL CCB, followed by 10 mL DI Water and repeat using 5 mL more CCB and 10 mL DI Water, and discard the washing filtrate.

6.0 Procedure

6.1 Sample Preparation

6.1.1 Drinking Water Samples (200.8)

6.1.1.1 Turbidity must first be analyzed by the turbidity method in SOP5862.

6.1.1.2 If the turbidity is < 1 NTU, it is run as a dissolved sample. The collision cell must not be used for drinking water analyses (if not permitted current Federal Register guidelines).

6.1.1.3 If the turbidity is >1 NTU, proceed to the digestion procedure in section 6.1.3 and qualify any drinking water samples where analytes require the collision cell and it is not allowed. (When a sample is digested, certain metals REQUIRE the collision cell to be used – see Table 2 for a list of affected metals).

6.1.1.4 Ensure that pH has been verified to be pH<2. If pH>2, it is acidified with 1:1 HNO_3 and held for at least 16 hours. Any actions are

recorded on the Sample Receipt and Condition Report.

6.1.1.5 The QC for dissolved drinking water samples is based on the analytical batch. The ICV is typically used as the LCS, and a second reading is used as the LCSD. The ICB is typically used as the Prep Blank.

6.1.1.6 Sample duplicates are selected based on section 7.2.6.

6.1.1.7 Matrix spikes are selected and prepared based on section 7.2.7.

6.1.1.8 Samples requiring the analysis of Ag must be digested using the digestion procedure in section 6.1.3 and analyzed with other digested samples.

6.1.2 Dissolved Water Samples (6020A)

6.1.2.1 Prepare digestion tubes for samples and QC: Label with sample numbers, MS, MSD or Duplicate sample, LCS, LCSD, Prep Blank.

6.1.2.2 In a fume hood, add approximately 40ml of DI H2O and 0.5mL HNO_3 to PB, LCS, and LCSD. Spike any control samples as needed (MS, MSD, LCS, LCSD) with second source standards.

6.1.2.3 Samples are now ready to be analyzed.

6.1.3 Digestion by method SW3005A for 6020A, 200.8 Waste Water and Other Aqueous Samples

6.1.3.1 Ensure that pH has been verified to be pH<2. If pH>2, it is acidified with 1:1 HNO_3 and held for at least 16 hours. Any actions are recorded on the Sample Receipt and Condition Report.

6.1.3.2 Transfer 50mL of well-mixed sample into a pre-cleaned digestion tube. Repeat procedure for each sample.

6.1.3.3 Prepare digestion tubes/sample for QC: MS, MSD or Duplicate sample, LCS, LCSD, Prep Blank.

6.1.3.4 In a fume hood, add 1mL HNO_3 and 2.5mL HCl. Spike any control samples as need (MS, MSD, LCS, LCSD) with 0.25mL second source standard.

6.1.3.5 Digest samples, covered with disposable watch glasses, for at least 2 hours in the block digester. The digester should be set to achieve an internal temperature of 95ºC +/- 3 ºC.

6.1.3.5.1 Monitor samples during digestion to make sure that there is always volume in the digestion tubes. Typically 15-20mL should be left. If sample goes dry, discard and re-prepare the sample.

6.1.3.5.2 The samples should never be allowed to boil.

6.1.3.6 Let samples cool, bring final volume to 50mL with DI Water, and

filter using FilterMate or Whatman filters. Cover samples if they will not be immediately analyzed.

6.1.3.6.1 Depending on the suspended matter in the samples, they can be centrifuged and/or decanted in place of filtration.

Solid Samples by Method SW3051A "Microwave Assisted Digestion"

6.1.3.7 Refer to TS SOP for dry weight determination procedure.

6.1.3.8 Place the 0.1g calibration weight on the balance and confirm that the value is within 10% of the true value. Record this check in the metals prep log.

6.1.3.9 Into a clean microwave vessel, add ~0.50g well mixed solid sample, ~0.25g oil, ~0.25g paint chip, or 1 wipe. The PB is prepared using 0.50g SiO_2 blank (for wipe PB only use a blank wipe, no SiO_2), and the LCS/LCSD is prepared using 0.50g of solid Certified Reference Material or 0.15mL of paint based Certified Reference Material. Record all amounts in the log book.

6.1.3.9.1 For DUP/MS/MSD, mix the necessary volume for the sample and all QC in a separate container to homogenize the sample, then weigh each aliquot out into a separate vessel.

6.1.3.9.2 If provided by customer, use extra, unused wipes for PB and LCS/LCSD, if not provided by customer use stock "ghost" wipes for QC. If enough samples are provided, prepare a sample DUP and a sample MS. If the customer requests to have wipes composited into one sample, the total count of wipes allowable is limited by the volume of reagent used in digestion. The wipes must be completely submerged for adequate digestion to occur.

6.1.3.9.3 Note: If the sample has standing water, pour off the water before mixing the soil. If the sample contains large rocks or pieces of debris, attempt to get a representative aliquot of sample without the large pieces.

6.1.3.10 In a hood to reduce exposure to harmful fumes, add 9mL HNO_3 then 3mL HCl to each microwave vessel.

6.1.3.10.1 For wipes, add the HNO_3 in two 4.5mL increments to avoid spillover caused by reaction with wipes.

6.1.3.11 The cap should be finger tight and then rotate an additional 30 degrees to achieve the proper seal and prevent any leaking.

6.1.3.12 Place each vessel in a composite sleeve evenly throughout the microwave turntable. (Note: no less than 8 vessels in the microwave).

Place samples in the turntable on the turntable drive lug and close the door. Record start time in prep log. Select the appropriate method. (Method is programmed to have the sample temperature rise to 175 ±5ºC in 5.5 ±0.25minutes and remain at that temperature for the remaining digestion period.)

6.1.3.12.1 8 samples: EPA3051_8Xpress

6.1.3.12.2 9-16 samples: EPA3051_16Xpress

6.1.3.12.3 17-24 Samples: EPA3051_24Xpress

6.1.3.13 Once the program has concluded, remove the turntable from the microwave and remove each vessel from their sleeve. Allow vessels to cool to room temperature before proceeding.

6.1.3.14 As caps are removed, confirm that each vessel has maintained its seal throughout the digestion process (no liquid visible on the outside of or directly under the cap).

6.1.3.14.1 Re-prep any sample with a seal that was not maintained. This time weigh sealed vessel before and after the digestion. Weights differing by more than 1% indicate sample has been lost. Flag the report as "Estimated."

6.1.3.15 Pour digestate into sample tubes, rinse the vessel three times with DI water, adding the solution from each rinse to the same tube. Bring to a final volume of 50mL and filter using FilterMate or Whatman filters. Dilute appropriately with CB to account for dissolved solids in the digested samples (Typically x5). QC samples must undergo the same dilution factor as samples. For analysis of paint chips, samples should be diluted x20 before analyzing.

6.1.3.15.1 Depending on the suspended matter in the samples, they can be centrifuged and/or decanted in place of or in addition to filtration.

6.1.3.16 Clean microwave tubes while in the hood, placing tubes in a container of warm soapy water. Refer to Glassware Cleaning SOP 802 for further instructions.

6.1.4 <u>Air Filter Samples by NIOSH Method 7303</u>

6.1.4.1 Remove filter from cassette holder with forceps, fold into quarters, and transfer to a clean 50mL disposable digestion tube

6.1.4.1.1 If provided, use blank filters for PB and LCS/LCSD, otherwise add all reagents to empty digestion tube.

6.1.4.2 Add 1.25mL HCl, cover with cap, and heat for 15 minutes to an internal temperature of 95ºC ±5ºC.

6.1.4.3 After cooling, add 1.25mL HNO_3, spike appropriate tubes for

LCS/LCSD and MS, if provided, with 0.125mL secondary source standard, and heat for another 15 minutes to an internal temperature of 95ºC ±5ºC.

6.1.4.4 Cool and bring final volume up to 25mL with DIH2O, filter if needed.

6.2 **Instrument Setup (see manual for additional instructions and details)**

6.2.1 Quality Control

6.2.1.1 Prior to analysis the MDL, IDL and LDR quality control parameters must be determined and updated or verified at a specified frequency. See section 7.1 for requirements.

6.2.2 Initial Setup

6.2.2.1 Inspect the sample and skimmer cones and the torch. Clean the cones as needed, typically once a week when the instrument is in regular use.

6.2.2.2 Inspect the nebulizer daily. Use compressed gas to push air through the nebulizer at the nebulizer tip. This will dislodge built up salts as well as larger suspended solids.

6.2.2.3 Replace internal standard (IS), waste, and sample tubing on the peristaltic pump as needed, typically once each week.

6.2.2.4 Clamp all tubes on the peristaltic pump, move (IS) sipper and sample line sipper to rinse blank containing 1% nitric solution. Start the pump and set to -20 rpm so that lines can be inspected for proper flow.

6.2.2.5 Ignite the plasma.

6.2.2.6 Allow ICPMS to warm up for 30 minutes or more.

6.2.2.7 Place both sippers into the NexION Setup Solution and allow it to stabilize

6.2.2.8 If the ICPMS was opened, perform torch alignment.

6.2.2.9 Optimize the ICPMS using the SmartTune manual wizard. Run the STD Performance check at the start to check the general working condition of the instrument.

6.2.2.10 Optimize the Nebulizer Gas Flow, QID STD/DRC, and KED Mode QID. This is typically performed daily or every couple of days.

6.2.2.11 The Detector Voltages and Dual Detector Calibration should be run daily or at least every couple of days.

6.2.2.12 After passing the STD performance check to check the

above stated changes in performance, perform the Mass Calibration and Resolution. See Section 7.2.13 for acceptance criteria.

6.2.2.13 If running 200.8 proceed to next step, if running 6020A, run the KED Performance Check

6.2.2.14 Save the default.tun and default.dac files to save tuning and dual detector calibration.

6.2.2.15 After successful optimization, create a dataset and sample list for the day's run saved as that date (i.e.: 010115).

6.2.2.16 Move IS sipper to the IS bottle and allow it to stabilize. ICPMS is now ready for calibration and sample analysis.

6.2.2.17 Standard Operating Conditions

6.2.2.17.1 Rinse time between samples: 60 seconds
6.2.2.17.2 Sample Flush Time: 60 seconds
6.2.2.17.3 Repeats per sample: 3
6.2.2.17.4 Delay Time: 25 Seconds
6.2.2.17.5 Flush Pump Rate: 48 rpm
6.2.2.17.6 Analysis Pump Rate: 20ppm

6.2.3 Calibration Setup

6.2.3.1 Prepare standards according to 5.2

6.2.3.2 Typically run two blanks and then the calibration for analytical method being used (200.8 DW or 6020A/ 200.8WW) and save calibration as day's date (i.e.: 010115)

6.2.3.3 Verify calibration according to 5.2.6

6.2.3.4 Recalibrate and verify new calibration if needed.

6.2.4 ICS

6.2.4.1 Verify the magnitude of elemental and molecular-ion isobaric interferences by analyzing the interference check solutions. See Table 1 and section 7.2 for frequency and requirements.

6.2.5 Sample Setup

6.2.5.1 After successful calibration, samples can be analyzed manually by running an unknown and moving the sipper into the sample solution or samples can be run on the autosampler (preferable method).

6.2.5.1.1 If running samples by hand, run a LLCS(optional), CCV and CCB every 10 samples. For 200.8, calibration standards required by the method need to be run every 10 samples as a CCV.

6.2.5.2 Save sample list as today's date with a letter in alphabetical order,

if needed.

6.2.5.3 Verify the correct method (200.8 or 6020A) is selected in the sample list for all samples.

6.2.5.4 Place all samples tubes in their assigned locations.

6.2.5.5 Run sequence by highlighting samples to be run and select build run list.

6.2.5.5.1 Do not clear the calibration data after successful calibration.

6.2.5.5.2 If needed, pause sequence for errors or to run dilutions, and resume sequence.

6.3 **Analysis**

6.3.1 For new or unusual matrices, run a screen the sample for elements at high concentration. This is done to prevent damage being inflicted on the detector during continuing sample analysis and/or to minimize interference effects to analytes and matrix effects to internal standards. The sample will also be screened for background levels of elements used as internal standards.

6.3.2 Evaluate the data using the Syngistix software. The RSDs of the three readings for each analyte must be monitored. High RSD values for hits over the reporting limit may be a result of carryover. Reportable results with RSDs greater than 5% will be evaluated for potential carry over from previously run samples. Blanks run after the ICSB, ICV, or high calibration point can provide a proper comparison of carry over effects in clean samples. Matrix interference and decline of accurate quantitation on the low end of the linear dynamic range can also cause high RSDs.

6.3.3 If an element has more than one monitored isotope, the concentration calculated for each isotope should be examined, to help detect possible spectral interferences.

6.3.4 In each sample, evaluate the Internal Standard ratios and recoveries. See section 7.2 for acceptance criteria.

6.3.5 Verify that all QC is acceptable (Rinse Blanks, CCB, PB, LCS/D, MS, DUPs, CCVs). Assess any drift at the low or high end of the dual detector range of the instrument that would limit the reportable range for sample results. Also, review any LLCS check and LDR verification standards run at the end of the analytical sequence to further verify that the linear range of instrument is acceptable. This is typically done on an element by element basis when reviewing reported data for each sample in an import file.

6.3.6 Determine which samples need to be diluted (>90%LDR)

6.3.6.1 Samples should be diluted with CCB in a separate digestion tube

and then reanalyzed.

6.3.7 Based on the results of matrix spikes, dilution tests or post digest spikes can be prepared an analyzed as needed. See QC section 7.2 for requirements and criteria.

6.3.8 Care should also be taken when reporting samples with low or high percent recoveries for a given internal standard. Low recoveries are typically due to high levels of minerals which causes suppression of instrument response. High recoveries may be due to native concentrations of the internal standard or interference causing a false internal standard reading. Monitor the IS and it's percent recovery in relation to continuing QC and dilute samples with unjustifiably high or low IS readings. See section 7.2.5 for QC requirements.

6.3.9 Data should be exported to Excel and then imported to the LIM system.

6.4 **Sample Calculations**

6.4.1 Results are obtained from the Syngistix Software. The result given is the instrument result. If there is a dilution factor, multiply the result and the reporting limit by that factor.

6.4.1.1 All masses which might affect data quality are monitored and accounted for by the software.

6.4.2 For solid samples, the concentration is calculated based on the dry fraction of the sample. The following equation is used to calculate the concentration of an analyte in a solid:

$$\text{Concentration } (\mu g/g) = \frac{C \times V}{W \times S}$$

Where, C = concentration of analyte in extract (µg/ml)
V = volume used in extraction (ml)
W = weight of material (g, as received)
S = percent solids in sample

6.4.3 For wipes, the concentration is reported as µg/wipe. The weight = 1.

6.4.4 For filters, the concentration is report as µg/filter. The weight = 1.

6.4.5 Hardness: Calcium and Magnesium Hardness is calculated using to Method 2340B (1997 and 2011) according to the following equation:

$$\text{mg } CaCO_3/L = 2.497*(\text{Ca, mg/L}) + 4.118*(\text{Mg, mg/L})$$

Title: Trace Metals By ICPMS EPA 200.8/6020A

7.0 Quality Control Requirements

7.1 Method Performance

7.1.1 Initial Demonstration of Performance: This is used to characterize instrument performance (linear calibration ranges and analysis of the ICV), analyst performance and laboratory performance (MDLs) prior to use of this method. The Demonstration of Capability as described in the QA Manual is followed prior to use of this method by an analyst.

7.1.1.1 Instrument Detection Limits: This is used to determine the level of noise on the instrument. The low level reporting limits and method detection limits should be above the IDL. The IDL should be established initially or whenever, in the judgment of the analyst, a change in analytical performance caused either by a change in instrument hardware or operating conditions would dictate they be re-determined. IDLs are determined by calculating the average of the standard deviations of three runs with seven consecutive measurements (analyses) on three non-consecutive days using a reagent blank solution. The IDL is verified quarterly using the LOD verification process.

7.1.1.2 Linear Calibration Range: This is the criteria of the calibration curve. Must be a minimum of three standards and a blank for 6020A and one standard and a blank for 200.8. All NLLAP data must be within this range to be reported.

7.1.1.3 Linear Dynamic Range (LDR): The linear dynamic range (LDR) determines the highest concentration of each analyte that can be reported. The LDR for each element is verified daily (ICSA), every six months (or quarterly as required by USACE work) or whenever, in the judgment of the analyst, a change in analytical performance caused either by a change in instrument hardware or operating conditions would dictate they be re-determined. The LDRs are established by analyzing successively higher concentrations of the analyte of concern, typically three to five standards are used. The upper range limit is an observed signal no more than 10% below the level extrapolated from the lower standards. Samples with results higher than 90% of the LDR are serially diluted with CCB and reanalyzed to assure final results are within the established LDR. The data, calculations and rationale for the choice of range are kept on file in the LDR file folder at the ICPMS.

7.1.1.4 Linear Dynamic Range Verification: After the LDR is established it must be verified initially and every six months. A standard at the

upper limit is prepared, analyzed and quantitated against the normal calibration curve. The calculated value should be within 10% of the true value. If this criteria is not met, the LDRs should be re-determined.

7.1.1.5 Initial Calibration Verification:

7.1.1.5.1 6020A: Two types of calibration verifications are required for 6020A.

7.1.1.5.1.1 The ICV must be from a second source and must be within ±10% of the true value. The concentration is typically near the middle of the curve.

7.1.1.5.1.2 The method also requires a "low level ICV", however since this is made from the primary calibration standard, it is referred to here as an LLCS. The concentration is at or near the reporting limit. The acceptance criteria is ±30% of the expected value.

7.1.1.5.2 200.8: Two types of calibration verifications are required for 200.8.

7.1.1.5.2.1 A calibration standard or multiple calibration standards must be analyzed after the curve. The recovery of the standard or standards must be ±10%.

7.1.1.5.2.2 A QCS (Quality Control Sample) must be analyzed at least once per quarter. This is an un-digested second source standard with a concentration </= .1mg/L, also known as an ICV. The acceptance criteria is ±10% of the expected value.

7.1.1.5.3 For NLLAP, the concentrations of the ICV and LLCS should be near the level of concern or actions level and whenever possible shall not require extensive pretreatment dilution or concentration prior to analysis.

7.1.1.6 Initial Calibration Blank: This un-digested blank is analyzed after the calibration curve to check for carryover/contamination. This must be matched correctly with the calibration standards and samples. The ICB must be lower than the reporting limit for each analyte. For NLLAP work, the absolute value of the ICB must not be more than 50 % of the lowest regulatory limit for the sample

matrix analyzed or minimum level of concern.

7.1.2 Method Detection Limit: The MDLs must be established for all analytes. Reagent water is fortified at a concentration of two to three times the estimated instrument detection limit. To Determine the MDL values, take seven replicates of the fortified reagent water and process through entire analytical system (the same preparation process as the LCS). Calculate the values of the MDL for each analyte. Calculate the MDL as follows:

$$MDL = (t)*(S)$$

t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. For seven replicates t = 3.14.

S = Standard deviation of the replicate analysis.

MDLs should be determined initially and whenever there is a significant change in the method or instrument. MDL data are stored electronically, in the QA/MDL file folder.

7.1.3 LLQC: Low Level QC is a requirement of method 6020A. It is made with a second source standard at a concentration of one to two times the reporting limit, and carried through the sample prep procedures. It is analyzed after establishing reporting limits and on an "as needed" basis. Recovery of ±30% verifies that the reporting limits are acceptable. This QC is run once per quarter and also referred to as an "LOQ". See LOQ verification spreadsheets.

7.1.4 LOD/LOQ: Refer to QAM *Analytical Procedures* section, for specific LOD/LOQ requirements. LOD/LOQ data are stored electronically in the QA/LOD&LOQ file folder.

7.1.5 IDC/CDC: Proficiency of the analyst is ensured by documentation of an Initial or Continuing Demonstration of Capability (IDC or CDC). Typically, the analyst analyzes four LCSs with acceptable precision and accuracy or shows acceptable performance of a blind sample analysis (usually a PT). See QAM for additional information.

7.1.6 Control Charts: Control Charts are quality control tools which graphically display QC data over time. The data required for generation of accuracy and precision control charts is maintained by the laboratory. See QAM for

additional information.

7.1.7 Inter-laboratory performance: Proficiency Test studies, conducted by approved providers, constitute the majority of inter-laboratory performance comparisons. Proficiency tests are performed at a frequency as required by method, regulation, or accrediting body. See QAM for additional information.

7.2 **Laboratory Performance Quality Control and Corrective Actions**

7.2.1 Continuing Calibration Verification: A CCV is performed after a curve is analyzed (as re-analysis of cal standards) for 200.8. For both 200.8 and 6020A a CCV is analyzed after every 10 samples, and at the end of each batch. The concentrations of CCVs should be varied throughout the run. The CCV must be within ±10% of the expected value. If the CCV is out of control, a second CCV can be analyzed, and if it passes the batch can be continued. If it is also outside of acceptance criteria, any problems should be investigated and corrected, and then a new curve analyzed if necessary. All samples analyzed after a failing CCV must be re-analyzed or flagged. Samples must be bracketed by acceptable CCVs (within ±10%), except for samples analyzed under method 200.8: if the ending CCV recovery is beyond +/- 15%, then the preceding samples must be reanalyzed.

7.2.1.1 The HLCS is an optional QC sample analyzed at a higher level in the linear range that is typically run at the end of the sequence. The acceptance limit of the HLCS is ±10% of the expected value.

7.2.1.2 A Low Level CCV (LLCCV or LLCS) needs to be run at the end of the sequence for 6020A with a concentration at or near the lower limit of quantitation. The recovery must be ±30% of the expected value. The LLCCV can be run more frequently if similar concentrations are expected in samples and to verify acceptable quantitation at the reporting limit throughout the run.

7.2.2 Continuing Calibration Blank: The matrix of this undigested blank should match the samples being analyzed. The CCB is analyzed at the same frequency as the CCV. The CCB must below the reporting limit for the analyte of interest. If the CCB is not below the reporting limit steps must be taken to determine the source of the problem. Corrective actions may include re-analysis of the CCB with fresh reagent water to trace the contamination. If there is suspected data impact (discuss with the QAO), the samples may be reanalyzed or the data qualified to disclose the suspected impact to the customer. If the concentration of the sample is greater than ten times the contamination level, three is no need to raise the

reporting limit or reanalyze. For NLLAP work, the absolute value of the CCB must not be more than 50 % of the lowest regulatory limit for the sample matrix analyzed or minimum level of concern.

7.2.3 Prep Blank: The PB is prepared and/or digested along with samples, to assess any contamination in the sample preparation procedure. The PB is analyzed once every batch or 24 hours, whichever is reached first. The PB must be below the reporting limit of the analyte. If the PB is above the reporting limit, the PB and samples are re-digested and re-analyzed or the sample is qualified at the discretion of the analyst, referring to the QAO and QA manual.

7.2.4 Laboratory Control Sample (LCS): The LCS is prepared and/or digested along with samples, to assess any bias in the sample preparation procedure and is analyzed once every batch or 24 hours, whichever is reached first. In each batch, duplicate precision is shown with a LCS Duplicate; For all methods, water and solids, the relative percent difference must be ≤20%. If the LCS is out of control, the source of the problem must be identified and solved or the data should be flagged. The entire batch should be re-digested if needed.

7.2.4.1 200.8WW&DW: It is also known as a LFB (Lab Fortified Blank) in method 200.8. The percent recovery is calculated and must be within ±15% of the expected value.

7.2.4.2 6020AWater: The percent recovery is calculated and must be within ±20% of the expected value for 6020A/NLLAP.

7.2.4.3 6020ASolid: CRM must meet the vendor control limits (95% confidence limits).

7.2.5 Internal Standard Monitoring: The internal standard response is monitored to identify issues with instrument drift and physical interferences. The ratios of the internal standard responses should be monitored by the analyst routinely. The ICPMS software will flag any response that is outside of the established acceptance range. If the IS is outside of the acceptance criteria, the sample must be diluted and re-analyzed. Flushing the instrument is recommended. If the problem persists, the run should be terminated and issue investigated. Possible causes include a partially blocked sampling cone, unfavorable mixing in the spray chamber or mixing T, or change in tuning conditions of the instrument.

7.2.5.1 6020A: The method establishes a minimum acceptance limit of 30%. Additionally, MCP analysis requires an acceptance range of 70% to 130% while DOD acceptance requirements are from 30% to 120%. All requirements are taken into account based on the

regulatory program of the sample.

7.2.5.2 200.8 WW and DW: Acceptance criteria is 60-125% of the original response in the calibration blank.

7.2.6 InterElement Correction Standard (ICS): Run at the beginning of the sequence or every 12 hours, whichever is more frequent. Acceptance criteria is 80-120%. Used to confirm the interference equations built into the method are accurate and check instrument performance (Ex. KED performance).

7.2.7 Matrix Spike: A known amount of analyte is added to an aliquot of a randomly chosen sample and carried through all prep and/or digestion procedures to access the effects of the sample matrix on analyte recovery.

7.2.7.1 6020A: One spike must be analyzed per 20 samples. For aqueous matrices, the spike must have a percent recovery of 75-125%. For solid matrices, the percent recovery is 70-130%.

7.2.7.2 200.8: One spike must be analyzed every 10 samples. The acceptance criteria is 70-130%.

7.2.7.3 NLLAP: acceptance criteria is 75-125% recovery.

7.2.7.4 The calculation of the percent recovery is as follows:

$$R = (Cs - C)/s * 100$$

R = percent recovery
Cs = Fortified sample concentration
C = Sample background concentration
s = Concentration equivalent of analyte added to the sample

If the recovery of any analyte falls outside the designated matrix spike recovery range and all the other QC for that analyte are shown to be in control, the recovery problem encountered with the spike is considered matrix or solution related, not system related. Make a note in the QC database for spikes. Qualify the out of control recovery as matrix interference in the database and on the final report. Post digestion spikes or serial dilutions may also be used as investigative tools. For MADEP MCP solid samples, as long as the % recovery is greater than 30% and the LCS in the batch is acceptable, no further action is necessary.

7.2.8 Matrix Duplicate: One matrix duplicate is run per 20 samples. The relative percent difference must be <20%. Matrix Spike Duplicates are run per customer request and can be substituted for a Duplicate Sample. For water sample results that are <5 times the reporting limit, the RPD control limit

is +/- the reporting limit. For solid sample results that are >5 times the reporting limit, the control limit is 20%. For solid sample results that are <5 times the reporting limit, the control limit is 2 times the reporting limit. The RPD is calculated as follows:

$$RPD = (D1-D2)/((D1+D2)/2) *100$$

D1 = The initial result of the analyte
D2 = The duplicate result of the analyte

7.2.9 Dilution Test: Re-analysis of a sample with a high concentration (typically >50 the RL) at a dilution to help identify matrix interference issues. A dilution test can be prepared when an MS or MSD has failed. See the current DoD QSM for current requirements of this standard. A 1:5 dilution is prepared and analyzed. If the difference between the diluted and undiluted result is greater than 10%, matrix effects are suspected and further evaluation with the use of a Post Digestion Spike (PDS) or MSA is required.

7.2.10 Post Digestion Spike (PDS): Analysis of a Matrix Spike prepared by adding the spike after the digestion process, to help identify matrix interference issues. This is typically performed when a Matrix Spike has failed on a sample with a native concentration < 50X the RL. When requested or required by regulatory program, one sample per batch is spiked post digestion. Whenever possible, the PDS should be performed on the sample that was used for the matrix spiking. The original sample digestate must be spiked, not the matrix spiked digestate. The sample is spiked at generally the same concentration as the pre digestion spike or up to two times the sample concentration. Recovery Acceptance Criteria: 80-120%. If both the MS and PDS indicate matrix interference, MSA can be done upon request to determine sample concentration.

7.2.11 Quarterly Lead Wipes: Per NLLAP, once a quarter preparation surfaces will be sampled with a wipe and analyzed for lead. Amount detected must be <50% the lowest regulatory limit for lead dust wipe samples.

7.2.12 NLLAP Reporting requirements: The lead concentration range for NLLAP samples is the Reporting Limit through 50,000 ug/sample. The reporting or quantitation limit must be equal to or less than 20 % of the lowest relevant action level or regulatory limit of interest for paint, and soil and 50 % of the lowest action level for dust wipe samples. The reporting limit is set **at a value at least 2 times,** but no greater than 10 times the method detection

limit.

7.2.13 Mass Calibration and Resolution: The mass calibration and resolution method reads Li, Mg, In, and U and measures peak qualities. Mass calibration criteria is determined by finding the middle of each peak. This point must be ±0.1AMU of the atomic mass set for this analyte within the method. For mass resolution criteria, six measurements of peak width at 5% peak height are attained for each analyte. These widths are then averaged. The average width must measure 0.75±0.03AMU for all 4 elements. The mass calibration and resolution is valid for 12 hours and must be rerun if the analytical batch runs beyond this 12 hour period.

7.3 See the QA Manual Section 15 for additional corrective action guidance.

8.0 Responsibilities

8.1 The analyst uses the Quality Control Criteria outlined in this SOP to evaluate and validate the data. The analyst or technicians responsibility is to follow this SOP and record all the data in the lab notebook. Any irregularities within the test or samples should be recorded in the lab notebook and reported to the lab manager who is responsible for reviewing the data.

9.0 Health and Safety

9.1 Normal, accepted laboratory safety practices should be followed during reagent preparation and instrument operation. However, every sample in the lab should be handled as if it is hazardous waste. Analysts should take extra care with concentrated acids. All digestions must be performed in a fume hood with proper ventilation. All technicians shall be familiar with the Chemical Hygiene Plan. Refer to SOP QA-604 and the Chemical Hygiene Plan for additional information.

10.0 Pollution Prevention and Waste Management

10.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. All standards are bought in 100 or 125mL quantities, except ICV (second source) std. and Yttrium (internal standard) in 250mL and IEC std. in 500mL. Refer to EPA 200.8 section 14.0 for contacts and information on pollution prevention.

10.2 The EPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Waste management in this laboratory is regulated by the Safety director. Waste management in this laboratory is regulated by the Safety director. (Refer to SOP QA-5001, Laboratory and Sample Waste Characterization, and Disposal.)

Title: Trace Metals By ICPMS EPA 200.8/6020A

Table 1: Recommended ICS Values

Analyte	Solution A Concentration (mg/L)	Solution B Concentration (mg/L)
Aluminum	100	100
Calcium	100	100
Iron	100	100
Magnesium	100	100
Sodium	100	100
Phosphorus	100	100
Potassium	100	100
Sulfur	100	100
Carbon	200	200
Chlorine	720	720
Molybdenum	2	2
Titanium	2	2
Arsenic	0	0.02
Cadmium	0	0.01
Chromium	0	0.02
Cobalt	0	0.04
Copper	0	0.02
Manganese	0	0.02
Nickel	0	0.04
Selenium	0	0.02
Silver	0	0.02
Vanadium	0	0.04
Zinc	0	0.02

Title: Trace Metals By ICPMS EPA 200.8/6020A

Table 2: Typical Element List and Reporting Limits #

Analyte	Chemical Symbol	Reporting Limits for Aqueous Samples* (mg/L)	Reporting Limits for Solid Samples* (ug/g)	Linear Dynamic Range (mg/L)
Aluminum	Al	0.05	25	20
Antimony	Sb	0.001	0.5	10
**Arsenic	As	0.005	2.5	10
Barium	Ba	0.01	5	10
Beryllium	Be	0.001	0.5	10
Boron	B	0.01	5	10
Cadmium	Cd	0.001	0.5	10
Calcium	Ca	0.5	250	20
**Chromium	Cr	0.01	5	10
**Cobalt	Co	0.01	5	10
**Copper	Cu	0.01	5	10
**Iron	Fe	0.05	25	20
Lead	Pb	0.005	2.5	10
Magnesium	Mg	0.1	50	20
**Manganese	Mn	0.01	5	10
Molybdenum	Mo	0.01	5	10
**Nickel	Ni	0.01	5	10
Potassium	K	0.5	50	20
**Selenium	Se	0.01	5	10
Silicon	Si	0.01	5	10
Silver	Ag	0.005	2.5	5
Sodium	Na	0.1	50	20
**Strontium	Sr	0.01	5	10
Thallium	Tl	0.001	0.5	Calibration Range
Tin	Sn	0.01	5	10
Titanium	Ti	0.01	5	10
**Vanadium	V	0.01	5	10
**Zinc	Zn	0.01	5	10
Hardness (by calc.)	CaCO3	1.7	NA	132

Current MDLs and LDRs are on file
*Note: RLs are typically 2-5(x) MDL
** Elements are run in KED Mode for digested samples.

Title: Trace Metals By ICPMS EPA 200.8/6020A

Table 3: Typical Analytical Sequence with Acceptance Criteria

Sample Number	Instrument Run Log	Acceptance Limit
	Analysis of Calibration Curve	
	LLCS ("LLICV" 6020) 0.001ppm	± 30%
	LLCS (200.8 reqd cal pt and "LLICV") 0.01ppm	± 10% for 200.8, ± 30% for 6020
	ICV low level ("QCS" in 200.8) 0.05ppm	± 10%
	ICV mid level ("QCS" in 200.8) 0.5ppm	± 10%
	ICB	<RL
	Inter-element Correction Standards (ICSA serves as LDR Check for minerals)	
	Test Blank	Check for carry over effects
1	PB-A	< RL
2	LCS-A	± 15% 200.8 or ± 20% 6020A
3	Sample 1	
4	Sample 2	
5	Sample 3	
6	Sample 4	
7	Sample 5	
8	Sample 6	
9	Sample 7	
10	Sample 8	
11	Sample 9	
12	Sample 10	
13	Sample 10 MS	±75-125 % Aq 6020A,70-130% Solid&200.8
14	Sample 10 MSD or dup Sample	RPD ≤20%
	LCSD-A	± 15% 200.8 or ± 20% 6020A,RPD ≤20%
	LLCS-CCV (0.01ppm)	+/-10% for CCV criteria, +/-30% for LLCS criteria
	CCV (0.05ppm)	+/-10% (+/-15% for closing 200.8)
	CCB	< RL
15	Sample 11	
16	Sample 12	
17	Sample 13	
18	Sample 14	
19	Sample 15	
20	Sample 16	
21	Sample 17	
22	Sample 18	
23	Sample 19	
24	Sample 20	
25	Sample 20 MS (for 200.8 only)	±75-125 % water
	LLCS-CCV (0.01ppm)	+/-10% for CCV criteria, +/-30% for LLCS criteria
	CCV (0.05ppm)	+/-10% (+/-15% for closing 200.8)
	CCB	
	LLCS (0.001ppm)	+/-30%
	HLCS (5ppm)	

Note: This is an example of how a sequence may be run. The sequence order may be slightly different as long as the SOP requirements are met.

Title: Trace Metals By ICPMS EPA 200.8/6020A

Table 4: Typical Calibration Parameters 6020A, 200.8 WW and 200.8 DW

	6020A and 200.8 WW						200.8 DW		
Level	**0**	**1**	**2**	**3**	**4**	**0**	**1**	**2**	**3**
	Conc (ppm)	**Conc (ppm**	**Conc (ppm)**	**Conc (ppm)**	**Conc (ppm)**	**Conc (ppm)**	**Conc (ppm)**	**Conc (ppm)**	**Conc (ppm)**
Ag	0.000	0.0005	0.005	0.025	0.05	0.000	0.0005	0.01	0.05
Al	0.000	0.051	0.51	2.55	5.1	0.000	0.051	1.02	5.1
As	0.000	0.001	0.01	0.05	0.1	0.000	0.001	0.02	0.1
B	0.000	0.001	0.01	0.05	0.1	0.000	0.001	0.02	0.1
Ba	0.000	0.001	0.01	0.05	0.1	0.000	0.001	0.02	0.1
Be	0.000	0.001	0.01	0.05	0.1	0.000	0.001	0.02	0.1
Ca	0.000	0.051	0.51	2.55	5.1	0.000	0.051	1.02	5.1
Cd	0.000	0.001	0.01	0.05	0.1	0.000	0.001	0.02	0.1
Co	0.000	0.001	0.01	0.05	0.1	0.000	0.001	0.02	0.1
Cr	0.000	0.001	0.01	0.05	0.1	0.000	0.001	0.02	0.1
Cu	0.000	0.001	0.01	0.05	0.1	0.000	0.001	0.02	0.1
Fe	0.000	0.051	0.51	2.55	5.1	0.000	0.051	1.02	5.1
K	0.000	0.01	0.1	0.5	1.0	0.000	0.01	0.2	1
Mg	0.000	0.051	0.51	2.55	5.1	0.000	0.051	1.02	5.1
Mn	0.000	0.001	0.01	0.05	0.1	0.000	0.001	0.02	0.1
Mo	0.000	0.001	0.01	0.05	0.1	0.000	0.001	0.02	0.1
Na	0.000	0.051	0.51	2.55	5.1	0.000	0.051	1.02	5.1
Ni	0.000	0.001	0.01	0.05	0.1	0.000	0.001	0.02	0.1
Pb	0.000	0.011	0.11	0.55	1.1	0.000	0.001	0.02	0.1
Sb	0.000	0.001	0.01	0.05	0.1	0.000	0.001	0.02	0.1
Se	0.000	0.001	0.01	0.05	0.1	0.000	0.001	0.02	0.1
Sn	0.000	0.001	0.01	0.05	0.1	0.000	0.001	0.02	0.1
Sr	0.000	0.001	0.01	0.05	0.1	0.000	0.001	0.02	0.1
Ti	0.000	0.001	0.01	0.05	0.1	0.000	0.001	0.02	0.1
Tl	0.000	0.001	0.01	0.05	0.1	0.000	0.001	0.02	0.1
V	0.000	0.001	0.01	0.05	0.1	0.000	0.001	0.02	0.1
Zn	0.000	0.051	0.51	2.55	5.1	0.000	0.051	1.02	5.1

Title: Hexavalent Chromium

QA Officer: Jennifer Guerrette Date: 4/4/16

Laboratory Director: Susan Ashworth Date: 4/4/16

Author: [signature] Date: 4/4/16

Analyst: _____ Date: _____

By signing this SOP, the analyst has read and understood the latest version of the test method; has read and understood this SOP; acknowledges receipt of this SOP; and is responsible for the implementation of this SOP.

Revision History:

Revision	Changes	Date
1	Format Update	12/07
2	Updated Co. name	7/11
3	Addition of solid requirements	4/12
4	Table 1 addition	1/13
5	Solid QC clarifications	1/14
6	Table 1 edit (BDL to BRL), location of LOQ/MDL	4/14
7	Table 1 edit spike recovery +/-25%, Method Performance added	3/16

Title: **Hexavalent Chromium SM 3500 Cr-B and SW-846 7196A**

1.0 Purpose and Applicability

1.1 To determine the concentration of hexavalent chromium in groundwater, domestic and industrial wastewaters, and solids. The effective analytical range is from 0.5 to 50 mg/L Cr+6.

2.0 Definitions

2.1 Continuing Calibration Blank (CCB): A volume of reagent water fortified with the same matrix as the calibration standards. A CCB is run at the beginning of a run when the calibration curve in place is being verified at the start of a batch, after every ten samples, and as a closing bracket. No CCB is run at the start of a batch if a new calibration curve is built on that day.

2.2 Initial Calibration Verification (Instrument Performance Check) (ICV): A standard at a mid-point in the calibration curve to verify the integrity of the curve. The ICV is made from a second source standard. All analytes must have a different lot number than the one that made the calibration curve.

2.3 Continuing Calibration Verification (CCV): A CCV is analyzed every ten samples, at the start of a batch (if verifying a previous curve), and at the end of every batch. A CCV is a mid-point on the calibration curve. The concentration of the CCV must be varied during a run.

2.4 Calibration Standard (ICal): Standard solutions prepared from the stock standard at levels corresponding to the calibration curve. These are used to calibrate the instrument response with respect to analyte concentration.

2.5 Field Duplicates: A duplicate sample taken in the field. These are only done at the customer's request.

2.6 Laboratory Control Sample (Laboratory Fortified Blank) (LCS): An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

2.7 Prep Blank (Laboratory Reagent Blank) (PB): An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, and reagents that are used with other samples. The PB is used to determine if the method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. The PB must be below the reporting limit for the analyte.

2.8 Linear Calibration Range (LCR): The concentration range over which the instrument response is linear and calibrated for.

2.9 Quality Control Sample (QCS): See definition for ICV.

Title: **Hexavalent Chromium SM 3500 Cr-B and SW-846 7196A**

2.10 Stock Standard Solution (SSS): A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source, i.e Ricca Chemical or Ultra Scientific. Documentation of the purity of the SSS must be retained for traceability purposes in the inorganic standards binder.

2.11 Matrix Spike: An aliquot of a sample spiked with a known amount of secondary spiking solution.

3.0 Applicable Documents/References

3.1 Standard Methods for the Examination of Water and Wastewater, APHA et al, 20th edition 1998, Method 3500 Cr-B.
3.2 SW-846 Method 7196A Colorimetric Hexavalent Chromium
3.3 SW-846 Method 9045C Soil and Waste pH
3.4 ASTM D1498-08 Standard Test Method for Oxidation-Reduction Potential of Water
3.5 SW-846 Method 3060A Alkaline Digestion for Hexavalent Chromium
3.6 ARA SOP QA-800, Use, Calibration, and Maintenance of Laboratory Equipment
3.7 ARA SOP QA-400
3.8 ARA QA Manual

4.0 Materials and Apparatus

4.1 **Equipment:**

4.1.1 Turner model 390 Spectrophotometer for use at 540nm, 1cm path length
4.1.2 Various volumetric pipettes and pipette tips
4.1.3 Plastic dose cups
4.1.4 Glass culture tubes
4.1.5 Heated stir plate and stir bars capable of 90-95ºC
4.1.6 250mL digestion vessels
4.1.7 Various graduated cylinders
4.1.8 Various volumetric flasks
4.1.9 Vacuum filtration apparatus
4.1.10 0.45um filter membranes
4.1.11 Balance
4.1.12 Thermometer

4.2 **Reagents/Standards:**

4.2.1 ASTM Type II Water (DI water)
4.2.2 Potassium Dichromate Stock Solution - Dissolve 141.4 mg of dried K2Cr2O2 (Reagent Grade) in ASTM Type II Water. Dilute to 1 liter

(1000 ml). 1 ml = 50 µg Cr. Prepared in the lab or purchased from a reputable supplier such as Ricca Chemical or Ultra Scientific Standards.

4.2.3 Potassium Dichromate Standard Solution - dilute 10.0 ml of Potassium Dichromate Stock Solution to 100 ml using DI water. 1 ml = 5 µg Cr.

4.2.4 Sulfuric Acid (10%) - Dilute 10 ml of reagent grade sulfuric acid (H2SO4) to 100 ml with DI water.

4.2.5 Diphenylcarbohydrazide Solution - Dissolve 50 mg of 1,5-Diphenylcarbohydrazide in 10 ml of Acetone. Store in a brown bottle and discard when discolored (solution is yellow and non-turbid when fresh) or when recovery begins to deteriorate.

4.2.6 Acetone- Reagent Grade

4.2.7 Magnesium Chloride

4.2.8 Phosphate Buffer: Dissolve 87.09g K_2HPO_4 and 68.04g KH_2PO_4 into 700mLs of reagent water and bring up to 1L in a volumetric flask.

4.2.9 Digestion Solution: Dissolve 20.0 +/- 0.05g NaOH and 30.0 +/- 0.05g Na_2CO_3 in reagent water and bring up to 1L in a volumetric flask. Prepare fresh monthly. The pH of the solution must be checked before using and must be 11.5 or greater.

4.2.10 5.0M Nitric Acid: 155mLs conc HNO3 diluted to 500mLs reagent water.

5.0 Method Summary/Calibration/Interferences

5.1 **Method Summary**: The aqueous sample is measured directly into a plastic dose cup. The sample is acidified with sulfuric acid and colored with the diphenylcarbohydrazide solution. After 5-10 minutes the absorbance is analyzed on the spectrophotometer at 540nm. Solid samples are preliminarily evaluated for pH and ORP analysis. The solid is prepared by the alkaline digestion method and the resulting digestate is analyzed the same as an aqueous sample.

5.2 **Sample Collection, Preservation, and Storage**

5.2.1 Aqueous samples are collected in 125mL previously cleaned polyethylene bottles. Solid samples are collected in two 4oz glass jars. One of the jars is designated for pH and ORP determination while the other jar is to remain unopened until the digestion for hexavalent chromium is ready to begin.

5.2.2 Aqueous samples are preserved with an ammonium sulfate buffer solution, (NH4)2SO4, and cooled to 0-6 degrees C at the time of collection. Solid samples are cooled to 0-6 degrees C at the time of collection.

5.2.3 Preserved aqueous samples are to be stored between 0 and 6 degrees C and may be analyzed up to 28 days after sample collection. Solid samples are to be stored between 0 and 6 degrees C and must be digested within 30 days of sampling. The digestate must be analyzed within 7 days of

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digestion. However, for MCP compliance the pH and ORP determinations for solid samples must be done within 24 hours of sampling.

5.2.4 Unpreserved aqueous samples are to be stored between 0 and 6 degrees C and must be analyzed within 24 hours of collection.

5.2.5 Shipment of samples must follow the preservation requirements of the method.

5.3 **Calibration Procedure**

5.3.1 The stock standard is diluted to create five points for the calibration curve. Typical levels for the curve are: 0.01ug/mL, 0.05ug/mL, 0.1ug/mL, 0.5ug/mL, and 1ug/mL. The reporting limit is the lowest point in the curve.

5.3.2 Calibration Verification Requirements

5.3.2.1 The calibration curve must have an R^2 value (calibration coefficient) of 0.995 or greater. An ICV and an ICB must be run immediately following the calibration curve. The ICV must be recovered at ± 15% the expected value. The ICB must be below the reporting limit for each analyte. These requirements must be met before using a new calibration curve.

5.4 **Interferences**

5.4.1 Interferent 1: Vanadium interferes strongly, but concentrations up to 10 times the hexavalent chromium levels are tolerable.

5.4.2 Interferent 2: Turbidity can cause interference and is typically dealt with by analyzing a background sample and subtracting the background absorbance from the analytical absorbance. If turbidity it too great, the sample can also be diluted as long as the quantitation limit is adjusted for this dilution.

6.0 **Procedure**

6.1 **Aqueous Sample Preparation**

6.1.1 Preparation blanks are prepared from reagent water.

6.1.2 Laboratory Control Samples are prepared by spiking reagent water with an appropriate amount of spike.

6.1.3 Aqueous samples are analyzed as received and do not require any preparation. Skip to 6.4

6.2 **Solid Sample Preliminary Evaluation**

6.2.1 Preliminary evaluation of pH and ORP are performed for MCP samples, when requested by the customer, and for matrix spike samples.

6.2.1.1 Use the sample jar designated for pH and ORP determination.

6.2.1.2 Evaluate the sample pH by following the procedure outlined in ARA SOP 5851 pH by Method SM 4500 H+B and SW-846 9045C

6.2.1.3 Evaluate the sample ORP by following the procedure outlined in

ARA SOP 5852 ORP by Method ASTM D1498-08 Oxidation-Reduction Potential of Water.

6.2.1.4 These values are reported for MA MCP samples or when requested by the customer.

6.3 **Alkaline Digestion for Solid Samples**

6.3.1.1 Place 2.5g +/- 0.1g of sample into a clean and labeled 250mL digestion vessel. Record the exact amount of sample used in the extraction log.

6.3.1.2 Weight out 2.5g +/-0.1g of play sand for method blanks and a 1.0g +/-0.1g of a certified reference material for the LCS/D. For spiked samples, add the appropriate amount of spike directly to the sample digestion vessel at this time.

6.3.1.3 Add 50mL +/- 1mL of the digestion solution (4.2.9) to each sample.

6.3.1.4 Add 400mg of MgCl2 (4.2.7) to each sample.

6.3.1.5 Add 0.5mL of 1.0M phosphate buffer (4.2.8) to each sample.

6.3.1.6 Add a stir bar and cover all samples with a watch glass.

6.3.1.7 Stir the samples for five minutes without heat.

6.3.1.8 Heat the samples to 90-95ºC and maintain the temperature for 60 minutes while stirring continuously.

6.3.1.9 Turn off the heat and all the samples to gradually cool to room temperature while continuing to stir.

6.3.1.10 Filter the contents of the digestion vessel through a 0.45um filter membrane using vacuum filtration. Rinse the digestion vessel with three successive reagent water rinses pouring each rinse through the filtration membrane. Transfer the filtrate to a clean 250mL vessel.

6.3.1.11 Place the vessel on a stir plate and while maintaining constant stirring add 5.0M nitric acid solution (4.2.10) dropwise until the pH of the solution is 7.5 +/- 0.5. If the pH deviates from the desired range the sample will need to be re-digested.

6.3.1.12 Remove the stirring device and rinse, collecting the rinsate in the beaker. Quantitatively transfer the digestate to a 100mL volumetric flask and bring to volume with reagent water. Mix well.

6.3.1.13 Analyze the resulting digestate for hexavalent chromium.

6.4 **Instrument Setup** (see manual for additional instructions and details)

6.4.1 The spectrophotometer is allowed to warm up for at least 1 hour.

6.4.2 Using distilled water, the spectrophotometer is zeroed out before analysis.

6.5 **Sample Analysis**

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6.5.1 Transfer 9.5 mLs of the sample or digestate to be tested into a plastic dose cup.
6.5.2 Add 0.3 mL 10%H2SO4 (for solid digestates add 0.3mL conc. H2SO4) Test with pH paper (pH should read 2.0 ± 0.5).
6.5.3 Add 0.2 mLs Diphenylcarbohydrazide solution to the cup and swirl.
6.5.4 Wait 5-10 minutes for full color development (positive color= purple). Transfer a portion of the sample to a clean culture tube and measure its absorbance at 540 nm on the spectrophotometer.
6.5.5 If there is no purple color development and the sample gives a positive reading on the spectrophotometer, a background correction is needed. To do a background correction, obtain an acidified sub aliquot of the sample containing no reagent and read absorbance. The background is subtracted from the original reading.
6.5.6 If the sample is still positive due to turbidity, dilute until a satisfactory reading and matrix correction can be obtained.

6.6 **Sample Calculations**

6.6.1 Sample absorbances are compared to absorbances obtained from calibration standards and concentrations are calculated. These concentrations are saved in the Excel template and imported into Aspen for final reporting.
6.6.2 Results are obtained by entering the absorbance reading into the Excel template. The result given is the instrument result. If there is a dilution factor, multiply the result and the reporting limit by that factor.

7.0 Quality Control Requirements

7.1 **Method Performance**

7.1.1 Initial Demonstration of Performance: This is used to characterize instrument performance (linear calibration ranges and analysis of the ICV), analyst performance and laboratory performance (MDL's) prior to use of this method. The Demonstration of Capability as described in the QA Manual is followed prior to use of this method by an analyst.
7.1.1.1 Linear Calibration Range: This is the criteria of the calibration curve. Must be a minimum of three standards and a blank and verified at ±15% by a second source. The calibration coefficient must be 0.995 or better.
7.1.1.2 Initial Calibration Verification: The ICV must be from a second source and be within ±15% of the true value.
7.1.1.3 Method Detection Limit: The MDL's must be established for all

analytes. Reagent water is fortified at a concentration of two to three times the estimated instrument detection limit. To Determine the MDL values, take seven replicates of the fortified reagent water and process through entire analytical system (the same preparation process as the LCS). Calculate the values of the MDL for each analyte. Calculate the MDL as follows:

MDL = (t)*(S)

t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. For seven replicates t = 3.14.

S = Standard deviation of the replicate analysis.

MDLs should be determined initially, as per the method requirement, if any, and whenever there is a significant change in the method or instrument. MDLs are not required for the solid digestion as the determinative method is the same. The MDL data is stored electronically, in the QA/MDL file folder.

7.1.2 LOD/LOQ: Refer to QAM Analytical Procedures section, for specific LOD/LOQ requirements. LOD/LOQ data is stored electronically in the QA/LOD&LOQ file folder.

7.1.3 IDC/CDC: Proficiency of the analyst is ensured by documentation of an Initial or Continuing Demonstration of Capability (IDC or CDC). Typically, the analyst analyzes four LCS's with acceptable precision and accuracy or shows acceptable performance of a blind sample analysis (usually a PT). See QAM for additional information.

7.1.4 Control Charts: Control Charts are quality control tools which graphically display QC data over time. The data required for generation of accuracy and precision control charts is maintained by the laboratory. See QAM for additional information.

7.1.5 Inter-laboratory performance: Proficiency Test studies, conducted by approved providers, constitute the majority of inter-laboratory performance comparisons. Proficiency tests are performed at a frequency as required by method, regulation, or accrediting body. See QAM for additional information.

7.2 **Laboratory Performance Quality Control and Corrective Actions**

7.2.1 Continuing Calibration Verification: The CCV must be performed every

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10 samples and be recovered within ± 15% of the expected values. The concentration of the CCV must be varied. If the CCV performed at the beginning of a run (in order to verify a curve) is out of control, either remake and re-analyze the CCV, if it passes the batch can be continued, or build a new calibration curve. If the CCV performed after 10 samples is out of control stop running. Locate the source of the problem and re-analyze the CCV. If the CCV is within ±15% of the expected value, re-analyze all the samples run after the last passing CCV.

7.2.2 Continuing Calibration Blank: The CCB is analyzed at the same frequency as the CCV. The CCB must be below the lowest point of the calibration curve, the reporting limit for the analyte. If the CCB is not below the reporting limit steps must be taken to determine the source of the problem. Corrective actions may include re-analysis of the CCB with fresh reagent water to trace the contamination. If there is suspected data impact (discuss with the QAO), the samples may be reanalyzed or the data qualified to disclose the suspected impact to the customer.

7.2.3 Prep Blank: The PB is analyzed once every batch or 24hours, whichever is reached first. The PB undergoes the same sample preparation process including the addition of ammonium sulfate buffer solution. The PB must be below the reporting limit of the analyte. Follow the same corrective action steps as the CCB.

7.2.4 Laboratory Control Sample/Duplicate (LCS/D) Standard Reference Material/Duplicate (SRM/D): The LCS/D is analyzed once every batch or 24 hours, whichever is reached first. The LCS/D undergoes the same sample preparation process including the addition of ammonium sulfate buffer solution. The percent recovery is calculated and must be within ± 15% of the expected value, aqueous samples. For solid samples, a standard reference material (SRM) is utilized. The recovery of the SRM/D must be within the 95% confidence limits. If the LCS/D or SRM/D is out of control, the source of the problem must be identified and the LCS/D SRM/D re-analyzed before continuing onto samples.

7.2.5 Matrix Spike: One spike must be analyzed per 10 samples. A known amount of analyte is added to an aliquot of a randomly chosen sample. The acceptance criteria for a matrix spike is 75-125% The calculation of the percent recovery is as follows:

$$R = (Cs - C)/s * 100$$

R = percent recovery
Cs = Fortified sample concentration

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C = Sample background concentration
s = Concentration equivalent of analyte added to the sample

For Solids: if the matrix spike is below the acceptance criteria and the SRM/D are acceptable, determine the sample's reducing/oxidizing nature by plotting the pH and ORP results on Chart 1. If the sample is in the reducing range of the chart, the spike has been reduced and therefore is unable to be recovered. Qualify the results accordingly.

7.2.6 Matrix Duplicate: One matrix duplicate is run per 20 aqueous samples. Duplicates are not required for solid matrix. The relative percent difference must be ± 20%. The RPD is calculated as follows:

$$RPD = (D1-D2)/((D1+D2)/2) *100$$

D1 = The initial result of the analyte
D2 = The duplicate result of the analyte

8.0 Responsibilities

8.1 The analyst uses the Quality Control Criteria outlined in this SOP to evaluate and validate the data. It is the analyst's or technician's responsibility to follow this SOP and record all the data in the lab notebook. Any irregularities within the test or samples should be recorded in the lab notebook and reported to the lab manager who is responsible for reviewing the data.

9.0 **Health and Safety**

9.1 Normal, accepted laboratory safety practices should be followed during reagent preparation and instrument operation. Diphenylcarbohydrazide is a known carcinogen and should be handled in the hood. However, every sample in the lab should be handled as if it is hazardous waste. Refer to Chemical Hygiene Plan (SOP QA604), and SDS sheets.

10.0 **Pollution Prevention and Waste Management**

10.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation.

10.2 The EPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Waste management in this laboratory is regulated by the Safety director. All waste generated by this method is collected in a carboy and disposed of accordingly.

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Table 1
Analytical Sequence With Acceptance Criteria

Sample Number	Instrument Run Log	Acceptance Limit
1	ICV*	± 15%
2	ICB	BRL
3	LCS/D SRM/D	± 15%,95% conf. int.
4	PB	BRL
5	Sample 1	
6	Sample 2	
7	Sample 3	
8	Sample 4	
9	Sample 5	
10	Sample 6	
11	Sample 7	
12	Sample 8	
13	Sample 9	
14	Sample 10	
15	Sample 10 spike	± 25%
16	CCV	± 15%
17	CCB	BRL
18	Sample 11	
19	Sample 12	
20	Sample 13	
21	Sample 14	
22	Sample 15	
23	Sample 16	
24	Sample 17	
25	Sample 18	
26	Sample 19	
27	Sample 20	
28	Sample 20 dup	± 20%
29	Sample 20 spike	± 25%
30	CCV	± 15%
31	CCB	BRL
32	LCS/D SRM/D	± 15%,95% conf. int.
33	PB	BRL
34	Sample 1	

- Sample 1 above will be an ICV if run right after a curve otherwise it would be a CCV.

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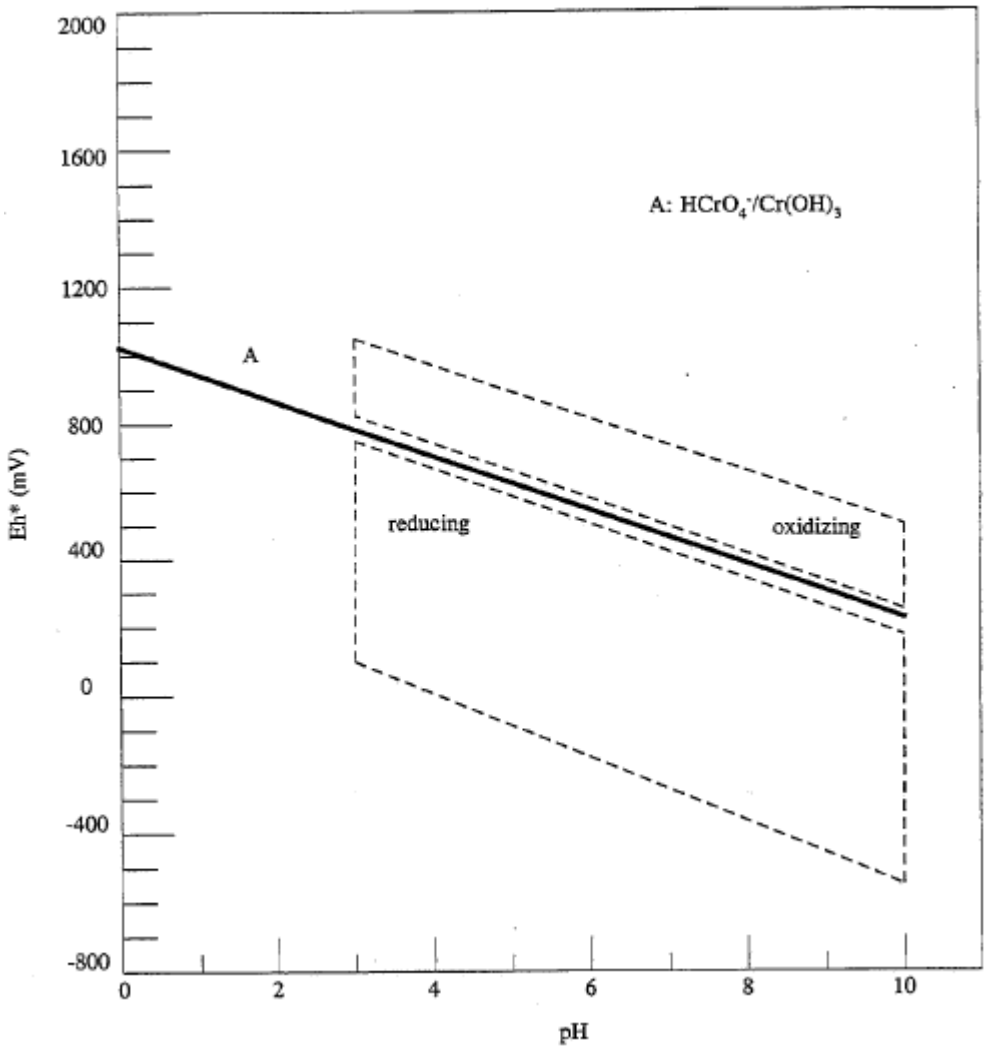
Chart 1



Title: pH by Method SM 4500 H+B and SW-846 9045C

QA Officer: Jennifer Guerrette Date: 2/28/17

Laboratory Director: [signature] Date: 2/28/17

Author: [signature] Date: 2/28/17

Analyst: _____ Date: _____

By signing this SOP, the analyst has read and understood the latest version of the test method; has read and understood this SOP; acknowledges receipt of this SOP; and is responsible for the implementation of this SOP.

Revision History:

Revision	Changes	Date
1	New format	
2	Added Method SM4500 CN-F; added calib. Procedure for portable	8/07
3	Clarified calibration procedure	8/09
4	Updated Co name, Solid holding time updated.	8/11
5	Added reference to 9045C for analysis of solids	4/12
6	Updated SM revision to 2000(DW),2011(WW)	01/13
7	Method Performance 7.1 clarified	04/16
8	Calibration stnds updated, ICV from different vendor, 7.2.4 CCV added.	02/17

Title: pH by Method SM 4500 H+B and SW-846 9045C

1.0 Purpose and Applicability

1.1 This Standard Operating Procedure is to describe the pH test for the analysis of liquid and solid samples. This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes and acid rain (atmospheric deposition). This method can also be used for testing pH of solid samples. This method is not applicable to measuring pH (corrosivity) of non aqueous solutions.

1.2 This SOP is applicable to both Drinking water and Wastewater approved SM4500H+B revisions and associated QC. The procedures are accurately represented in this SOP, and cover both versions. The QC from the most stringent version will be followed. See Section 3, Applicable References, for the method revision dates applicable to this SOP.

1.3 This method is recommended for use only by or under the supervision of analysts experienced in the use of a pH meter. Each analyst must demonstrate the ability to generate acceptable results with this method

2.0 Definitions

2.1 NA

3.0 Applicable Documents/References

3.1 Standard Methods for the Examination of Water and Wastewater, SM 4500H+B-2000, SM 4500H+B-2011

3.2 Method 9045C Revision 3, 1995

3.3 ARA SOP QA-400

3.4 ARA SOP QA-800, Use, Calibration, and Maintenance of Laboratory Equipment - Inorganic Laboratories

3.5 ARA QA Manual QA-003

4.0 Materials and Apparatus

4.1 Equipment:

- 4.1.1 Hanna pH21 Meter or Portable pH Meter (with temperature compensation)
- 4.1.2 Stir plate and tiny stir bars
- 4.1.3 Plastic dose cups
- 4.1.4 Balance

4.2 Reagents/Standards:

- 4.2.1 pH 4 Buffer
- 4.2.2 pH 7 Buffer
- 4.2.3 pH 10 Buffer
- 4.2.4 pH 6 Buffer-different manufacturer from pH 4, 7, and 10.
- 4.2.5 pH 8 Buffer-different manufacturer from pH 4, 7, and 10.

4.2.6 Electrode Filling solution

5.0 Method/Calibration/Interferences

5.1 Method Summary

5.1.1 Samples are poured into clean beakers or plastic dose cups. The pH meter is calibrated and then the electrode is immersed into the liquid sample while stirring. The meter will stabilize on a pH value. This is the result.

5.1.2 Sample Collection, Preservation and Storage

5.1.2.1 Aqueous samples are collected in previously cleaned polyethylene bottles and solid samples are collected in two or four ounce glass jars.

5.1.2.2 Sample preservation: Samples must be placed on ice immediately after collection and transported to the laboratory for analysis. Aqueous pH samples are tested upon receipt at the laboratory.

5.1.2.3 Sample holding time: Aqueous samples must be analyzed within 15 minutes of sampling, or reported with a qualifier. Solid samples must be analyzed within 7 days of sampling.

5.2 Calibration Procedure for Hanna pH21 Meter

5.2.1 Meter must be calibrated each day of use or if the time between sample readings is more than four hours. This two-point calibration is carried out with pH buffers 7 and 10 or 7 and 4. A calibration verification (ICV) is performed with a pH buffer that was purchased from a different vendor than the calibration standards (typically pH6 or pH8).

5.2.2 The concentrations of the two point calibration should bracket the sample results or be verified with the ICV or CCV as to bracket the sample results. For example, if the instrument is calibrated with 7 and 10 and the sample results are 6.2, the meter's calibration can be verified with pH 6 to confirm linearity. If the pH 6 is not with in the acceptance criteria (+/- 0.06 pH units), the meter must be recalibrated with 7 and 4 and the sample re-analyzed.

5.2.3 Pour approximately 20 mL of each buffer solution into plastic dose cups. Place a tiny stir bar in each new cup.

5.2.4 Uncover probe (electrode) and check filling solution. Make sure to keep the filling solution to within 1/4inch of the hole. (KCl filling solution is kept in pH equipment drawer.)

5.2.5 Rinse probes (i.e.: electrode and temperature compensator probe) with DIwater.

5.2.6 Immerse probe into the 7.00 buffer and stir. Turn stir plate on so that the stir bar spins without creating a vortex. Press the Cal key and wait for the hourglass to disappear.

5.2.7 Once the hourglass has disappeared push the CFM button to lock that

reading in as the calibration point. The reading should be within 0.06 units of the true value of the buffer. If it is not, try fresh buffer or refer to the manual for maintenance. Record readings in the logbook.

5.2.8 Rinse probe with DI water and place probe in either the pH10 or pH4 buffer while mixing. Once the hourglass has disappeared push the CFM button to lock that reading in as the second calibration point. The reading should be within 0.06 units of the true value of the buffer. If it is not, try fresh buffer or refer to the manual for maintenance. Record readings in the logbook.

5.2.9 Note: If buffer readings are not within 0.06 pH units of expected values, with temperature compensation, the electrode may need cleaning. Recalibrate with fresh solutions or seek assistance from the instrument manual and lab manager prior to performing pH analysis.

5.2.10 Be sure to remember that temperature compensation must be taken into effect. Refer to the instrument manual located in drawer near pH meter for guidance.

5.2.11 Once an acceptable calibration curve is established, it must be verified with an ICV pH standard. The reading must be within 0.06 pH units to validate the curve.

5.3 Calibration for portable pH meter

5.3.1 The pH meter must be calibrated each day of use or if the time between sample readings is more than four hours. This two-point calibration is carried out with pH buffers 7 and 10 or 7 and 4. A calibration verification (ICV) is performed with a pH buffer that was purchased from a different vendor than the calibration standards (typically pH6 or pH8).

5.3.2 The concentrations of the two point calibration should bracket the sample results or be verified with the ICV or CCV as to bracket the sample results. For example, if the instrument is calibrated with 7 and 10 and the sample results are 6.2, the meter's calibration can be verified with pH 6 to confirm linearity. If the pH 6 is not with in the acceptance criteria (+/- 0.06 pH units), the meter must be recalibrated with 7 and 4 and the sample re-analyzed.

5.3.3 Pour approximately 20 mL of buffer 7 solution into a plastic dose cup.

5.3.4 Place the portable pH probe in the standard and allow it to sit for 30 minutes. This conditions the electrode.

5.3.5 Pour approximately 20 mL of buffer 7 and 10 into two other plastic dose cups.

5.3.6 After conditioning for 30 minutes, remove the probe from the 7 and rinse it with a small amount of DI water.

5.3.7 Push the ON/OFF button to turn on the probe.

5.3.8 Once the display lights, push the "cal" button and place the probe in the

pH 7 standard. Wait two minutes, then push "hold/ent" button.

5.3.9 Record the value in the top part of the display in the field notebook.

5.3.10 Rinse the probe with a small amount of DI water and place the probe in the pH10 standard.

5.3.11 Wait two minutes, then push "hold/ent" button.

5.3.12 Record the value in the top part of the display in the field notebook.

5.3.13 Push the "cal" button. The pH meter is now calibrated. If the two recorded values are +/- 0.06 pH units from the true value the calibration is acceptable. If either value does not meet this criteria, return the probe to the pH 7 and allow to condition another 30 minutes before trying again.

5.3.14 Once an acceptable calibration curve is established, it must be verified with an ICV pH standard. The reading must be within 0.06 pH units to validate the curve.

5.3.15 To transport the pH meter: moisten the sponge in the probe cap with the remaining pH7 standard, and place the cap on the probe. The probe is now ready to bring to the sample location.

5.3.16 Once on site, the calibration needs to be verified prior to sample analysis, by analyzing pH 7 Buffer.

5.3.17 Push the ON/OFF to turn on the probe. Tear open the pH 7 pouch and place the probe into the solution. Wait two minutes (until the meter has stabilized) and record the calibration verification result in the field notebook. This result needs to be +/- 0.06 pH units from the true value to verify the calibration is still valid.

5.3.18 The calibration is good for four hours.

5.3.19 If this criterion is not met, recalibrate the probe in the field using the two other pouches and following the steps listed above. If this criterion is still not met consult the instrument manual or call the lab manager. Do not proceed with any sample analysis.

5.3.20 Rinse the probe with DI water before beginning sample analysis.

5.4 Interferences

5.4.1 Samples with pH <3

5.4.1.1 The probe may need to be rejuvenated after exposure to samples with pH <3. The following are steps that may be taken to rejuvenate refillable combination electrodes. Continue on to the next step in the following sequence only if the previous step has failed.

5.4.1.1.1 Replace filling solution and soak overnight in dilute KCL solution (electrode filling solution diluted 2- to 4-fold.)

5.4.1.1.2 Cycle the bulb of the electrode between 1N HCl and 1N

NaOH, immersing the probe one minute in each solution. Soak in pH 4 buffer for ten minutes.

5.4.1.1.3 Empty electrode and immerse in 3 to 4 molar NH4OH for ten minutes. Rinse thoroughly with water and add fresh filling solution. Soak in pH 4 buffer for ten minutes.

5.4.2 Probe Cleaning Procedure (taken from SW 846 9045)

5.4.2.1 If an electrode becomes coated with an oily material that will not rinse free, the electrode can either be cleaned with an ultrasonic bath, or be washed with detergent, rinsed several times with water, placed in 1:10 HCl so that the lower third of the electrode submerged, and then thoroughly rinsed with water.

6.0 Procedure

6.1 Calibrate the meter according to section 5.2 or 5.3. If the meter has already been calibrated for the day (within four hours), check the calibration with a new pH 7 buffer and record this reading in pH lab notebook. If the result is within the acceptance criteria, analysis of samples may proceed. If it has been more than four hours since the calibration, the meter must be re-calibrated.

6.2 Record date, time and initials for this analytical session.

6.3 Shake sample well. Pour about 25 ml into a clean dose cup. Place a tiny stir bar in cup. Place on stir plate, turn on stir plate and immerse probes.

6.4 For solid samples: add 20g of sample to 20mL of DI water in a plastic cup. Add a stir bar, cover with the appropriate lid and stir for 5 minutes. Allow the suspension to stand for about an hour or long enough to settle significantly. Filtration or centrifugation may also be utilized. Decant water from the suspension and analyze the pH of the water as described above.

6.5 Wait for meter to stabilize, record value displayed.

6.6 Rinse probes with DI water between samples.

6.7 Report results to two significant figures.

6.8 See section 7.2.3 and 7.2.4 for batch QC frequency requirements.

7.0 Quality Control Requirements

7.1 Method Performance

7.1.1 Initial Demonstration of Performance: Prior to analyzing pH samples the analyst must be trained by an approved analyst, must read the SOP, and must successfully analyze 4 LCS samples. The pH reading between LCS samples should be within 0.2 units.

7.1.2 The ICV must be within the acceptance criteria (+/- 0.06 pH units).

7.1.3 IDC/CDC: Proficiency of the analyst is ensured by documentation of an Initial or Continuing Demonstration of Capability (IDC or CDC).

Typically, the analyst analyzes four LCS's with acceptable precision and accuracy or shows acceptable performance of a blind sample analysis (usually a PT). See QAM for additional information.

7.1.4 Control Charts: Control Charts are quality control tools which graphically display QC data over time. The data required for generation of accuracy and precision control charts is maintained by the laboratory. See QAM for additional information.

7.1.5 Inter-laboratory performance: Proficiency Test studies, conducted by approved providers, constitute the majority of inter-laboratory performance comparisons. Proficiency tests are performed at a frequency as required by method, regulation, or accrediting body. See QAM for additional information.

7.2 Laboratory Performance Quality Control/ Corrective Actions

7.2.1 Calibration: See sections 5.2 or 5.3 for procedure and acceptance criteria. The pH meter must be calibrated each day of use or if the time between sample readings is more than four hours. This two point calibration is carried out with pH buffers 7 and 10 or 7 and 4.

7.2.2 Initial Calibration Verification: The calibration curve must be verified with an ICV to confirm linearity. A second source standard, (typically pH 6 or pH 8) purchased from a different vendor than the calibration standards, is analyzed to verify calibration curve. See sections 5.2.11 or 5.3.14 for acceptance criteria.

7.2.3 Duplicate Sample: One duplicate sample should be analyzed per 10 samples or each day of use, whichever is more frequent. Duplicates should be within 0.2 pH units.

7.2.4 Continuing Calibration Verification: A CCV or ICV/LCS is analyzed every ten samples and at the end of a batch. The acceptance criterion is +/- 0.06 units of the expected value.

7.2.5 Refer to the Quality Assurance Manual for guidance on corrective actions not covered in this SOP.

8.0 Responsibilities

8.1 The analyst or technicians responsibility is to follow this SOP and record all the data in the lab notebook. Any irregularities within the test or samples should be recorded in the lab notebook and reported to the lab manager who is responsible for reviewing the data.

9.0 Health and Safety

9.1 Normal, accepted laboratory safety practices should be followed during reagent

preparation and instrument operation. No known carcinogens are used in this analysis. All technicians shall be familiar with the Chemical Hygiene Plan.

10.0 Pollution Prevention and Waste Management

10.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. The waste generated in this analysis is non hazardous.

10.2 The EPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Waste management in this laboratory is regulated by the Hazardous Waste Coordinator.

ABSOLUTE RESOURCE ASSOCIATES
Standard Operating Procedure QA-801

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Revision History
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Title: Sample Readiness

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Analyst: Date:

By signing this SOP, the analyst has read and understood the latest version of the test method; has read and understood this SOP; acknowledges receipt of this SOP; and is responsible for the implementation of this SOP.

Revision History:

Revision	Changes	Date
1	Updated Co Name	7/11
2	Incremental sampling added-Table 8	9/15
3	Added additional info to SOP sections, IDOC added to table 8	3/16

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1.0 Purpose and Applicability

1.1 The purpose of this SOP is to outline various sample readiness procedures to ensure consistency and accuracy in the generation of the analytical data. These procedures "ready" the sample for the subsequent extraction/digestion/etc. associated with the method of analysis. See Tables 1-8 for applicable matrices.

2.0 Definitions

2.1 Refer to Tables 1-8 for any definitions that may be applicable to particular sample readiness procedures, otherwise, refer to determinative method SOP.

3.0 Applicable Documents/References

3.1 ABSOLUTE RESOURCE ASSOCIATES Standard Operating Procedures

3.2 ARA SOP800, "Use, Calibration, Cleaning and Maintenance of General Laboratory Equipment and Glassware"

3.3 ARA QA Manual

3.4 Manufacturer Instrument Manuals

4.0 Materials and Apparatus

4.1 Refer to Tables 1 through 8 for specific requirements.

5.0 Methods/Calibration/Interferences

5.1 Method Summary

5.1.1 Refer to Table 1 through 8 for sample readiness procedures.

5.2 Preservation and Holding time

5.2.1 As applicable, referenced in tables 1-8. Also, refer to the determinative method SOP for guidance regarding preservation and holding time requiremments.

5.3 Calibration Procedure

5.3.1 As applicable, referenced in tables 1-8. Also, refer to the determinative method SOP for guidance regarding calibration.

5.4 Interferences

5.4.1 As applicable, referenced in tables 1-8. Also, refer to the determinative method SOP for guidance regarding interferences.

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6.0 Procedure

6.1 The attached tables provide procedures to ready the samples prior to the preparation, digestion, or extraction steps associated with the designated analysis.

6.2 All equipment must be used only by trained persons. Refer to manufacturer's instructions for details of procedures not defined in Tables 1 through 8.

6.3 Accurate and complete records of all calibrations and procedures must be maintained in the appropriate logbooks.

6.4 Refer to Tables 1 through 8 for details of information that needs to be recorded.

7.0 Quality Control Requirements

7.1 Method Performance

7.1.1 Initial Demonstration of Performance

7.1.1.1 Linear Calibration Range: Not applicable to sample readiness procedures.

7.1.1.2 Initial Calibration Verification: Not applicable to sample readiness procedures.

7.1.1.3 Method Detection Limits: Not applicable to sample readiness procedures, refer to determinative method SOPs.

7.1.2 Initial Demonstration of Capability (IDOC): proficiency of the analyst is ensured by documentation of an IDOC as applicable for sample readiness procedures. At a minimum, new analysts are provided a copy of this SOP and are trained on the sample readiness procedures by an experienced analyst. New analysts are observed by experienced analysts performing the sample readiness procedures before performing them on their own. Refer to specific Tables below for additional IDOC requirements.

7.1.3 Inter-laboratory performance: Not applicable to sample readiness procedures, refer to determinative method SOPs.

7.2 Laboratory Performance Quality Control and Corrective Actions

7.2.1 Refer to the determinative method SOP for guidance regarding Quality Control and Corrective actions. A supervisor or QAO must be consulted anytime the procedures outlined in tables 1-8 cannot be followed, to determine an appropriate action.

7.2.2 Each procedure listed in Tables 1 through 8 must be recorded in the Sample Readiness logbook.

7.2.3 All equipment and instrumentation used must be clean, well maintained and accurately calibrated. Refer to SOP 800 for information on the use, calibration, cleaning and maintenance of laboratory equipment and glassware.

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7.2.4 Equipment must be assigned an ID number, which must be recorded in the Sample Readiness Logbook.

7.2.5 Standards and reagents must be assigned an ID number, as assigned when recorded in the appropriate "Standards Prep Log". The ID must be recorded in the Sample Readiness Logbook

7.2.6 The highest level of care should be taken to prevent contamination of samples when performing sample readiness procedures.

8.0 Responsibilities

8.1 It is the analyst or technician's responsibility to follow this SOP and record all the data in the lab notebook. Any irregularities within the test or samples should be recorded in the lab notebook and reported to the lab manager who is responsible for reviewing the data.

8.2 The Lab Director shall be responsible for assuring that the analyst and technicians are adequately trained in these procedures and shall periodically review the log books.

8.3 The Quality Assurance Officer shall be responsible for conducting periodic audits and inspections of these procedures and for reporting the findings to the Lab Director.

9.0 Health and Safety

9.1 Normal, accepted laboratory safety practices should be followed during reagent preparation and instrument operation. However, every sample in the lab should be handled with caution. Refer to Chemical Hygiene Plan (SOP QA604), and SDS sheets.

9.2 The use of equipment and instrumentation in the laboratory involves the handling of dangerous and hazardous chemicals. Any persons involved in the operation of this equipment must be trained in the handling of those materials. Before performing any procedure the analyst must be familiar with the Safety Data Sheet (SDS) for all compounds which are used with this equipment.

9.3 Appropriate safety equipment, including gloves, lab coats, face shields, safety glasses, etc. should be used when appropriate to prevent injury or contamination.

9.4 All technicians shall be familiar with the Chemical Hygiene Plan.

10.0 Pollution Prevention and Waste Management

10.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. All waste generated as a result of this method is disposed of in labeled drums that are stored until full and picked up by a company dealing in disposal of hazardous material. If you are unsure

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which drum the waste generated belongs in, check with the Hazardous Waste Manager/Coordinator.

10.2 The EPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Waste management in this laboratory is regulated by the Hazardous Waste Manager/Coordinator.

10.3 See SOP 5001 for laboratory waste handling procedures.

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TABLE 1
Compositing Samples (Excluding VOCs)

A) Equipment/Reagents/Standards

1) Calibrated Balance
2) Wooden scoopula
3) Appropriate sample jars/bottles

B) Procedure

I. Solid Matrix

1) Each individual sample to be included in the composite is mixed well inside the original container to ensure that the sub-sample taken for the composite is representative of the sample. Miscellaneous debris such as roots, twigs, glass, etc. should be avoided if possible.
2) Using a balance, equal aliquots of each sample for the composite are collected in a clean sample container appropriate to the analysis being performed. The amount of each aliquot should be sufficient to yield a volume appropriate for the subsequent analyses. This amount can be discussed with the Lab Director. Do not selectively choose particles to achieve a particular weight. (ie. 1.00g) Each aliquot weight should be within 10% of the target weight. Record the weight of the representative subsample.
3) The sample aliquots in the composite container are then mixed well prior to analysis being performed. Dry weight determination is performed on the composited sample.

II. Aqueous Matrix

1) Each individual sample to be included in the composite is shaken well to ensure homogeneity.
2) A graduated cylinder is used to measure the aliquots. Equal aliquots of each sample for the composite are collected in a clean sample container appropriate to the analysis being performed. The amount of each aliquot should be sufficient to yield a volume appropriate for the subsequent analyses. This amount can be discussed with the Lab Director.

C) Recordkeeping

1) Record the pre-composite Sample IDs
2) Record the sample amounts used for each of the pre-composites
3) Record the post-composite Sample ID
4) Record any problems or irregularities noted during this procedure.

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TABLE 2
Compositing VOC Samples

A) Equipment/ Reagents/Standards

1) Calibrated Balance
2) Wooden scoopula
3) Gas tight syringes (verified upon receipt)
4) Pipette

C) Procedure

I. Solid Matrix

1) Each individual sample to be included in the composite is collected in a methanol preserved container in the field.
2) At the laboratory, each individual sample is prepared for analysis as per SOP 5125, including the addition of surrogates to the sample.
3) The net weight for each container is calculated, the average weight will be used in calculations. Dry weight determination is performed on each individual sample and the average is used in the calculation.
4) At the time of analysis, equal aliquots of methanol are taken from each individual sample with a gas tight syringe and combined following the specified method procedure for analysis.
5) The composite sample is then analyzed as per the designated analysis.

II. Aqueous Matrix

ONLY PERFORMED WHEN REQUESTED BY CUSTOMER AND QUALIFIED ON FINAL REPORT

1) Each individual sample to be included in the composite is collected in a pre-preserved VOC vial with zero headspace.
2) At the time of analysis, equal aliquots of each sample are taken using a pipette or gas tight syringe and combined in a 50mL volumetric flask.
3) The final volume of the sample is 50mLs. Aliquot volumes will depend on the number of samples contributing to the composite. For instance, if there are five samples, 10mLs will be withdrawn from each sample to make the composite.

C) Recordkeeping

5) Record the pre-composite Sample IDs
6) Record the sample amounts used for each of the pre-composites
7) Record the post-composite Sample ID
8) Record any problems or irregularities noted during this procedure

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TABLE 3
Filtering Metals

A) Equipment/ Reagents/Standards

1) 60cc B-D syringe with Luer lock fitting
2) 13mm or 25mm diameter, 0.45um nylon Luer lock syringe filters
3) DIH2O to rinse the syringe before and after use
4) 50mL disposable digestion tubes with screw caps
5) 1**:**1 (HNO3 **:** DIH2O) for sample preservation after filtering

B) Procedure

1) Verify that the aqueous sample has not been previously preserved with HNO3 by testing with pH paper on inside of sample lid. (If it has been preserved, notify the customer that the sample cannot be filtered to analyze for dissolved metals; total metals may still be determined after digestion.)
2) Rinse the syringe 3 times with DIH2O.
3) Fill the syringe with ~25-50mL of sample. Attach the 0.45um filter to the Luer fitting.
4) Filter 1mL of sample into a waste container to flush the filter, filter the rest of the sample into a digestion tube labeled with the lab number and "Lab Filtered", date and time of preservation (~25mL is sufficient unless DUP/MS is required, for which 25mL more is required for each QC.) If filter becomes clogged, replace with a new filter and continue from the start of step 4.
5) If the sample is extremely difficult to filter, it may be pre-filtered with a 0.7um or 1.2um filter, and then filtered with a 0.45um filter. A minimum of 10mL is required for ICP analysis.
6) Discard used filter and rinse syringe 3X with DIH2O.
7) A Prep Blank, consisting of DIH2O, should be filtered through all filter types utilized.
8) Add 5 drops 1:1 HNO3 to each 50mL filtered sample to bring to <pH2; cap sample.
9) The sample is now ready for analysis (no digestion is needed) as a "dissolved" metals sample.

C) Recordkeeping

1) Note on the SRCR that the sample has been lab filtered with 0.45um filter and preserved with HNO3 to <pH2; include date and time of filtering/preservation.
2) In the Sample Readiness logbook, record the date and time of filtering/preservation, any problems or irregularities encountered during the procedure and the lot number of the filters used.

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TABLE 4

Decanting/Separating Aqueous and Solid samples

A) Equipment/ Reagents/Standards

1) Scoopula

B) Procedure

I. Solid Matrix with water on surface

1) Allow sample to settle thoroughly.
2) Slowly and gently pour water off the surface into a container until just before the solid starts to flow.
3) Discard water in trash or sink, depending on volume.
4) Thoroughly mix the remaining solid for dry weight determination and analysis.

II. Aqueous Matrix with solid in bottle

1) Allow sample to settle thoroughly.
2) Slowly and gently pour water off the surface until just before the solid starts to flow. Collect the water in a method appropriate, labeled container for analysis or storage.
3) Analyze aqueous portion and allow solid portion to dry in a fume hood.
4) Discard dried solid portion in solid waste.

C) Recordkeeping

1) Record the Sample ID
2) Any problems or irregularities noted during the procedure.

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TABLE 5
Particle Size Reduction

A) Equipment/ Reagents/Standards

1) 9.5mm sieve
2) As required by physical characteristics of sample: mortar and pestle, hammer, heavy mallet, scissors, tin snips, etc.

B) Procedure

1) In consultation with the lab director, determine the best tool(s) and methods to reduce the particle size of the appropriate sample volume to sizes that will pass through a 9.5mm sieve.
2) If the sample is very heterogeneous (i.e. assorted building materials, assorted dyed leather scraps), make sure to prep it in the same relative proportions as the entire sample and discuss the heterogeneity with the customer.
3) Using the appropriate tools, as noted above, reduce the particle size sufficient for it to pass through the sieve.
4) Deposit the particle-size-reduced sample into an appropriate, labeled container for storage or analysis. Be sure to wash the tools in between each sample.

C) Recordkeeping

1) Record the Sample ID and all pertinent prep data in the sample readiness logbook
2) Note on the SRCR: particle size reduction was performed, the date, and initials.
3) Record all tools used
4) Note any problems or irregularities encountered during the reduction process.

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TABLE 6
Subsampling

A) Equipment/ Reagents/Standards

1) Disposable wooden scoopula
2) Appropriate sampling bottles

B) Procedure

I. Solid Matrix

1) Mix the sample well in the original jar to ensure that the sub-sample taken is representative of the sample. Miscellaneous debris such as roots, twigs, glass, etc. should be avoided if possible.
2) Scoop an appropriate volume of representative sample into the method specific jar labeled with lab number.
3) Discard used scoopula and take care to prevent contamination between samples.

II. Aqueous Matrix

1) Shake sample thoroughly.
2) Pour the appropriate volume of sample into the method specified bottle labeled with the lab number.
3) Preserve according to the method of analysis.

C) Recordkeeping

1) Record the Sample ID
2) Record how many bottles/jars the sample was distributed into
3) Any problems or irregularities noted during the procedure.

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Table 7
Multiphasic Samples

A. **Equipment/ Reagents/Standards**
1) Calibrated Balance
2) Disposable glass pipette
3) Appropriate sample bottles
4) Various solvents

B. **Procedure**
1) The customer must specify which phase of the sample they wish to have analyzed.
2) If free product is on the surface of a water sample and the customer wants to analyze the free product, a glass pipette is used to withdraw a portion of the product.
3) A vial or jar of pre-weighed solvent appropriate for the analysis is placed on the balance. The balance is then tared.
4) The product is added to the solvent, using the balance to determine the amount of sample added.
5) The dissolved product can then be analyzed by the requested method.

C. **Recordkeeping**
1) Record the sample ID
2) Record the volume of sample withdrawn and the volume and ID of solvent.

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Table 8
Incremental Sampling Procedure

A. Equipment/ Reagents/Standards

1) Calibrated Balance
2) Clean Tray or contained flat surface
3) Scoopula
4) Sample jar for subsample

B. Reference

1) SW846 8330B, Rev2,Appendix A, Section A.5.0

C. Procedure

1) The entire sample must be mixed and spread out evenly 1-2 cm thick, on a clean surface, preferably in a fume hood. The sample should be air dried.
2) A minimum of thirty randomly located increments must be scooped from the spread out sample and must cover the entire sample profile area and depth, to form a subsample of adequate size to perform the determinative method and meet applicable reporting limits. For instance, a determinative method that requires a 10g sample size would need 30 randomly chosen aliquots of approximately 0.33g to generate the subsample.
3) To assess the heterogeneity of the subsample, triplicate subsamples should be removed and analyzed for every 5 to 20 samples processed.
4) The subsample may now be extracted or digested by the prep method and/or analyzed by the determinative method indicated.
5) Depending on the customer request and sample matrix, the sample may receive particle size reduction by sieving to exclude solid pieces > 2mm. The entire sample could then also be ground, in small batches, to reduce the particle size to less than 75 microns.

D. Recordkeeping

1) Record the sample ID and ID's for equipment used.
2) Record the number of increments taken to form the subsample and the final weight of the subsample.
3) Record additional quality control samples taken from the sample.

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E. Initial Demonstration of Capability (IDOC)

1) Prior to performing the procedure outlined in Table 8, new analysts must be observed performing the procedure by the QA officer or an analyst previously trained in incremental sampling techniques. Documentation of this observation must be filed in the employees training file.

Title: Analysis of Samples for Polychlorinated Dioxins and Furans by HRGC/HRMS

[Methods 8290, 8290A & TO-9A]

Approvals (Signature/Date):

Robert Hrabak — 9/22/16 — Date
Operations Manager

Joe Schairer — 9/22/16 — Date
Health & Safety Manager / Coordinator

Lisa Stafford — 10/20/16 — Date
Quality Assurance Manager

Crystal Pollock — 10/31/16 — Date
Laboratory Director

1. SCOPE AND APPLICATION

1.1. This method provides procedures for the detection and quantitative measurement of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD), polychlorinated dibenzo-p-dioxins (tetra- through octachlorinated homologs; PCDDs), and polychlorinated dibenzofurans (tetra- through octachlorinated homologs; PCDFs) in a variety of environmental matrices at part-per-trillion (ppt) concentrations by SW 846 Method 8290 and 8290A. The analytical method calls for the use of high-resolution gas chromatography and high-resolution mass spectrometry (HRGC/HRMS) on purified sample extracts. An optional method for reporting the analytical results using a 2,3,7,8-TCDD toxicity equivalency factor (TEF) is also described. Table 1 lists the various sample types covered by this analytical protocol, the 2,3,7,8-TCDD-based method calibration limits and other pertinent information.

1.2. The sensitivity of this method is dependent upon the level of interferences within a given matrix.

1.3. This method is designed for use by analysts who are experienced with residue analysis and skilled in high-resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS).

1.4. Samples containing concentrations of specific congeners (PCDDs and PCDFs) that are greater than the calibration limit should be analyzed by a protocol designed for such concentrations, such as 8280A/B.

1.5. When undertaking projects for Department of Defense (DoD) or the Department of Energy (DOE), the relevant criteria in QA Policy WS-PQA-021 must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. This procedure uses matrix-specific extraction, analyte-specific cleanup, and high-resolution capillary column gas chromatography/high resolution mass spectrometry (HRGC/HRMS) techniques. Sample preparation is addressed in WS-IDP-0005.

2.2. One to two µL of the concentrated extract are injected into an HRGC/HRMS system capable of performing selected ion monitoring at resolving powers of at least 10,000 (10 percent valley definition).

2.3. The identification of ten of the 2,3,7,8-substituted congeners (Table 3), for which a ^{13}C-labeled standard is included as a spiked compound, is based on their elution at their exact retention time (-1 to +3 seconds from the respective isotope dilution analyte or internal standard signal) and simultaneous detection of the two most abundant ions in the molecular ion region. All other identified PCDD/PCDF congeners are identified by

their RRTs based on the daily CCV standard, and the simultaneous detection of the two most abundant ions in the molecular ion region. Confirmation is based on a comparison of the ratio of the integrated ion abundance of the molecular ion species to their theoretical abundance ratio.

2.4. Quantification of the individual congeners, total PCDDs and total PCDFs is achieved in conjunction with the establishment of a multipoint (five points) calibration curve for each homolog, during which each calibration solution is analyzed once.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

3.3. Polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs): compounds (Figure 1) that contain from one to eight chlorine atoms. The seventeen 2,3,7,8-substituted PCDDs and PCDFs are shown in Table 3. The number of isomers at different chlorination levels is shown in Table 4.

3.4. Homologous series: Defined as a group of chlorinated dibenzodioxins or dibenzofurans having a specific number of chlorine atoms.

3.5. Isomer: Chemical compounds that contain the same number of atoms of the same elements, but differ in structural arrangement and properties. For example, 1,2,3,4-TCDD and 2,3,7,8-TCDD are different structural isomers.

3.6. Congener: Any isomer of any homologous series.

3.7. Isotope Dilution Analyte: An isotope dilution analyte is a ^{13}C-labeled analog of a congener chosen from the compounds listed in Table 3. Isotope dilution analytes are added to all samples including method blanks and quality control samples before extraction, and they are used to quantitate the concentration of the analytes. Nine isotope dilution analytes are used in this method. There is one for each of the dioxin and furan homologs (except for OCDF) with the degree of chlorination ranging from four to eight. Additional isotope dilution analytes may be added to act as retention time references, but they are not used for quantitation.

3.8. Internal Standard: Two internal standards are used to determine the percent recoveries for the isotope dilution analytes. The ^{13}C-1,2,3,4-TCDD is used to measure the percent recoveries of the tetra- and pentachlorinated isotope dilution analytes while ^{13}C-1,2,3,7,8,9-HxCDD is used to determine the recovery of the hexa-, hepta- and octachlorinated isotope dilution analytes. ^{13}C-1,2,3,7,8,9-HxCDD also acts as a

retention time reference for the unlabeled analog present in sample extracts. They are added to the final sample extract before HRGC/HRMS instrument analysis.

3.9. Estimated Detection Limit (EDL)/ Estimated Quantitation Limit (EQL): The sample specific estimated detection limit (EDL/EQL) is the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background noise level.

3.10. Estimated Maximum Possible Concentration (EMPC): The calculated concentration of a signal having the same retention time as a PCDD/PCDF congener, but which does not meet the other qualitative identification criteria defined in the method.

4. INTERFERENCES

4.1. Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts or elevated baselines that may cause misinterpretation of the chromatographic data. All of these materials must be demonstrated to be free from interferents under the conditions of analysis by running laboratory method blanks. Analysts shall not use PVC gloves.

4.2. The use of high-purity reagents and solvents helps minimize interference problems. Purification of solvents by distillation in all-glass systems may be necessary.

4.3. Re-use of glassware is to be minimized to avoid the risk of contamination.

4.4. Interferents co-extracted from the sample will vary considerably from matrix to matrix. PCDDs and PCDFs are often associated with other interfering chlorinated substances such as polychlorinated biphenyls (PCBs), polychlorinated diphenyl ethers (PCDPEs), polychlorinated naphthalenes, and polychlorinated xanthenes that may be found at concentrations several orders of magnitude higher than the analytes of interest. Retention times of target analytes must be verified using reference standards. These values must correspond to the retention time windows established. While certain clean-up techniques are provided as part of this method, unique samples may require additional cleanup steps to achieve lower detection limits.

4.5. A high-resolution capillary column (60m DB-5) is used to resolve as many PCDD and PCDF isomers as possible. However, no single column is known to resolve all isomers. The DB-225 column is used for the quantitation of 2,3,7,8-TCDF when 2,3,7,8-TCDF on the DB-5 column is detected.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) 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-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. The effluents of sample splitters for the gas chromatograph and roughing pumps on the HRGC/HRMS system should pass through either a column of activated charcoal or be bubbled through a trap containing oil or high-boiling alcohols.

5.1.2. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex and vinyl gloves provide no protection against most of the organic solvents used in this method. Nitrile or similar gloves must be used. Latex gloves may be used for methanol.

5.1.3. Exposure to chemicals must be maintained as low as reasonably achievable, therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.4. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Iso-octane	Flammable Irritant	None established	Inhalation of vapors may cause nausea, headache, dizziness, loss of consciousness, irritation to upper respiratory tract, pain in throat and nose, coughing, wheezing, shortness of breath.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Tetradecane	Irritant	None established	Inhalation of vapors may cause difficulty breathing, headache, intoxication and central nervous system damage.
Toluene	Flammable Poison Irritant	200 ppm-TWA 300 ppm-Ceiling	Inhalation may cause irritation of the upper respiratory tract. Symptoms of overexposure may include fatigue, confusion, headache, dizziness and drowsiness. Peculiar skin sensations (e. g. pins and needles) or numbness may be produced. Causes severe eye and skin irritation with redness and pain. May be absorbed through the skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. High-Resolution Gas Chromatograph/High-Resolution Mass Spectrometer/Data System (HRGC/HRMS/DS).

6.1.1. Capable of collecting, recording and storing MS data. The VG70 and Autospec Ultima systems utilize Opus version 3.6 software and the Autospec Premiere system utilizes MassLynx version 4.1 software.

6.1.2. The GC must be equipped for temperature programming. All required accessories must be available, such as syringes, gases, and capillary columns. The GC injection port must be designed for capillary columns. The use of splitless injection techniques is recommended. The use of a moving needle injection port is also acceptable. When using the method described in this protocol, a 2-µL injection volume is used consistently (i.e., the injection volumes for all extracts, blanks, calibration solutions and the performance

check samples are 2 µL). 1 µL injections are allowed; however, laboratories are encouraged to remain consistent throughout the analyses by using the same injection volume at all times on a given HRGC/HRMS/DS.

6.1.3. Gas Chromatograph/Mass Spectrometer (GC/MS) Interface - The GC/MS interface components should withstand 350° C. The interface must be designed so that the separation of 2,3,7,8-TCDD from the other TCDD isomers achieved in the gas chromatographic column is not appreciably degraded. Cold spots or active surfaces (adsorption sites) in the GC/MS interface can cause peak tailing and peak broadening. It is recommended that the GC column be fitted directly into the mass spectrometer ion source without being exposed to the ionizing electron beam. Graphite ferrules should be avoided in the injection port because they may adsorb the PCDDs and PCDFs. Vespel® or equivalent ferrules are recommended.

6.1.4. Mass Spectrometer - The static resolving power of the instrument must be maintained at a minimum of 10,000 (10 percent valley). The mass spectrometer must be operated in a selected ion monitoring (SIM) mode with a total cycle time (including the voltage reset time) of one second or less.

6.1.5. Data System - A dedicated data system is employed to control the rapid multiple ion monitoring process and to acquire the data. Quantification data (peak areas or peak heights) and SIM traces (displays of intensities of each ion signal being monitored including the lock-mass ion as a function of time) must be acquired during the analyses and stored. Quantifications may be reported based upon computer-generated peak areas or upon measured peak heights (chart recording). The data system must be capable of acquiring data for a minimum of 10 ions in a single scan. It is also recommended to have a data system capable of switching to different sets of ions (descriptors) at specified times during an HRGC/HRMS acquisition. The data system should be able to provide hard copies of individual ion chromatograms for selected gas chromatographic time intervals. It should also be able to acquire mass-spectral peak profiles and provide hard copies of peak profiles to demonstrate the required resolving power. The data system should also permit the measurement of noise on the base line.

6.2. GC Column

6.2.1. Due to poor separation of 2,3,7,8-TCDF from other TCDF isomers on the 60 m DB-5 column, a 30m DB-225 is used to quantitate 2,3,7,8-TCDF. This column is used when 2,3,7,8-TCDF is detected.

6.2.2. In order to have an isomer-specific determination for 2,3,7,8-TCDD and to allow the detection of OCDD/OCDF within a reasonable time interval in one HRGC/HRMS analysis, the 60m DB-5 fused-silica capillary column is

recommended. At the beginning of each 12-hour period during which samples are analyzed and after tuning, acceptable compound separation on the GC column must be demonstrated through the analysis of a column performance check solution. Operating conditions known to produce acceptable results with the recommended column are shown in Table 7.

6.3. Recommend Maintenance

On an As-Needed Basis:
Full Bake-Out.
Change oil in rotary pump.
Change oil in diffusion pump. Replace o-rings.
Solvent rinse the flight tube.
Clean the first field free region.
Check detector voltages.
Clean and dust connectors, etc on the outside of the instrument.
Check the vacuum: ~5 x. 10^{-7} MBAR on both analyzer ion gauges, and ~5 x 10^{-6} MBAR on the source, with no helium flowing.
Check isolation valve for leaks, correct if needed.
Check for thermal trip by taking the magnet to maximum current, and verify that the coolant flow is acceptable.
Replace septum.
Clean injector port.
Cut off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required.
Replace injection port liner when front portion of capillary column is removed.
Clean Source, including all ceramics and lenses - the source cleaning is indicated by a variety of symptoms including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination.
Replace filaments when performance indicates need for replacement.
On a Daily (when in use) basis:
Check resolution sensitivity.
Check stability.
Check for sufficient gas supply. Check for correct column flow and/or inlet pressure.
Check temperatures of injector, detector.
Verify temperature programs.
Check inlets, septa.
Check baseline level.
Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds.
Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.

7. REAGENTS AND STANDARDS

7.1. Solvents

7.1.1. High-purity, distilled-in-glass or highest available purity: methylene chloride, hexane, methanol, tetradecane, isooctane, toluene, and acetone.

7.2. All calibration, daily isotope dilution analyte, daily clean up internal standards, and daily spiking solutions are stable for one year from preparation. After 1 year, solutions may be re-verified. The re-verified solution may be used for an additional year, or until there is evidence of compound degradation or concentration. The re-verification must be performed using an unexpired, not previously re-verified solution from a second lot or second vendor.

7.2.1. Sealed ampules may be used until the manufacturer's expiration date is exceeded. If no expiration date is provided, then the expiration date will be 10 years from the date the ampule is opened. The solvent level should be monitored prior to each use to assure there has been no concentration of the standard over time.

7.3. Calibration Solutions

7.3.1. High-Resolution Concentration Calibration Solutions (Table 5) - Five tetradecane solutions containing unlabeled (totaling 17) and carbon-labeled (totaling 16) PCDDs and PCDFs at known concentrations are used to calibrate the instrument. The concentration ranges are homolog dependent, with the lowest values associated with the tetra chlorinated dioxins and furans (0.5 pg/µL) and the highest for the octachlorinated congeners (2000 pg/µL).

7.3.2. Individual isomers that make up the high-resolution concentration calibration solutions are obtained from commercial sources and prepared in the laboratory. These standards are traceable back to EPA-supplied standard solutions.

7.3.3. Store the calibration solutions in appropriate containers and at room temperature in the dark.

7.3.4. Standards for method 8290A require storage at $\leq 6^{o}$C.

7.4. GC Column Performance Check Solution

7.4.1. This solution contains the first and last eluting isomers for each homologous series from tetra- through hepta-chlorinated congeners. The solution also contains a series of other TCDD isomers for the purpose of documenting the chromatographic resolution. The ^{13}C-2,3,7,8-TCDD is also present. The laboratory is required to use tetradecane as the solvent and adjust the volume so that the final concentration does not exceed 100 pg/µL per congener. Table 8 summarizes the qualitative composition (minimum requirement) of this performance evaluation solution for the DB-5 column.

7.4.2. For the DB-225 column, the column performance check solution contains a series of TCDF isomers in addition to the 2,3,7,8-TCDF. The solution is injected and evaluated at the start of each analytical sequence on the DB-225 column to ensure that 2,3,7,8-TCDF is resolved from its closest eluting isomers with a baseline-to-valley ratio of ≤ 25%. Table 8 summarizes the qualitative composition (minimum requirement) of this performance evaluation solution on for the DB-225 column.

7.5. Field Surrogate Solution (air matrices)

7.5.1. This solution contains one ^{37}Cl labeled analog (for Method TO-9/TO-9A) or one ^{37}Cl and four ^{13}C labeled analogs (for Methods 23 and/or 0023A) at the nominal concentration indicated in Table 2. It is used to assess sample collection and recovery procedures.

7.6. Sample Fortification Solution (Isotope dilution analyte)

7.6.1. This isooctane (or toluene) solution contains the nine isotope dilution analytes at the nominal concentrations that are listed in Table 2. The solution contains at least one carbon-labeled standard for each homologous series, and it is used to measure the concentrations of the native substances. (Note that ^{13}C-OCDF is not present in the solution.)

7.7. Internal Standard Solution

7.7.1. This tetradecane solution contains two internal standards (^{13}C-1,2,3,4-TCDD and ^{13}C-1,2,3,7,8,9-HxCDD). An appropriate volume of this solution will be spiked into each sample extract before the final concentration step and HRGC/HRMS analysis.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. The sample collection, shipping, handling, and chain-of-custody procedures are not described in this document. Sample collection personnel will, to the extent possible, homogenize samples in the field before filling the sample containers. This should minimize or eliminate the necessity for sample homogenization in the laboratory. The analyst should make a judgment, based on the appearance of the sample, regarding the necessity for additional mixing. If the sample is clearly non-homogeneous, the entire contents should be transferred to a glass or stainless steel pan for mixing with a stainless steel spoon or spatula before removal of a sample portion for analysis.

8.2. Grab and composite samples must be collected in glass containers.

8.3. Ambient air samples are collected on a Quartz Fiber Filter followed by a glass sleeve containing a polyurethane foam plug.

8.4. Samples from stationary sources are collected on glass or quartz fiber filters and XAD-2 Resin. (See WS-ID-0009 for sample preparation procedures).

8.5. Conventional sampling practices must be followed. Do not rinse the bottle with sample before collection. Sampling equipment must be free of potential sources of contamination.

8.6. With the exception of the fish tissues, which must be stored at - 20°C, all samples should be stored at 4°C ± 2, extracted within 30 days and completely analyzed within 45 days of extraction. Fish tissue is extracted within 30 days and completely analyzes withn 45 days of collection. The 30 day hold time is recommended. PCDDs and PCDFs have demonstrated stability for greater than one year.

8.7. Air samples by MethodTO-9A must be extracted within 40 days and completely analyzed within 45 days of extraction.

8.8. All extracts must be stored capped, in the dark, at room temperature (approximately 21°C to 28°C). All extracts for method 8290A must be stored capped at ≤ 6ºC.

9. QUALITY CONTROL

9.1. One method blank (MB) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The method blank is an aliquot of laboratory matrix (reagent water, Ottawa sand, sodium sulfate, PUF, XAD, filter, etc.) processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when target analytes are detected in the method blank above the reporting limit or when surrogate recoveries are outside control limits. Re-extraction of the blank, other batch QC, and the affected samples are required when the method blank is deemed unacceptable. The method blank contains a PUF plug, XAD, or filter prepared from the same batch as the field samples whenever possible for air samples.

Certain programs, such as the DOD/DOE Quality Systems Manual (QSM) may require a more stringent evaluation of the method blank, for instance, that the blank not contain any analytes of interest at a concentration greater than ½ the lower calibration limit.

Note: Re-extraction of the blank, QC and affected samples for the air matrices (PUF, XAD, and filter) is not generally possible because the entire sample is consumed in the initial extraction.

9.1.1. The method blank must be spiked prior to extraction with the same amount of ^{13}C-labeled isotope dilution analytes as added to samples.

9.1.2. If method blank contamination is present, check solvents, reagents, fortification solutions, apparatus and glassware to locate and eliminate the source of contamination before any further samples are extracted and

analyzed.

9.1.2.1. OCDD is a ubiquitous laboratory contaminant. A method blank and the associated samples are deemed acceptable if the OCDD concentration is <5x the specified reporting limit. Flag data appropriately. The analyst is expected to investigate and eliminate potential sources of systematic contamination.

9.1.2.2. If a target analyte is detected in the blank but the associated samples are ND (not detected), then the data may be reported, unless otherwise directed by the client. Note the action in the narrative.

9.1.2.3. If a target analyte is detected in the blank, but the concentration of the contaminant in the samples >10x the blank concentration, then the data may be reported, unless otherwise directed by the client. Note the action in the narrative.

9.1.3. If new batches of reagents or solvents contain interfering contaminants, purify or discard them.

9.2. A Laboratory Control Sample (LCS) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The LCS is an aliquot of laboratory matrix (e.g. water, Ottawa sand, sodium sulfate, PUF, XAD, etc.) spiked with analytes of known identity and concentration. The LCS must be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, other batch QC and all associated samples are required if the LCS is deemed unacceptable. See policy WS-PQA-003 for specific acceptance criteria. When associated with PUF samples, the LCS should contain a PUF plug prepared from the same batch as the field samples whenever possible.

Note: Re-extraction of the blank, QC and affected samples for the air matrices (PUF, XAD, and filter) is not generally possible because the entire sample is consumed in the initial extraction.

9.2.1. A LCS is deemed acceptable if control analytes are above upper control limits and the associated samples are ND, unless otherwise specified by the client. Note any actions in the narrative.

9.3. The assessment of matrix effects on method performance, as required by NELAP, is met in Method 8290 and 8290A, as in all isotope dilution techniques, with the use of isotopically labeled compounds. These isotopically labeled compounds are analogs of target analytes and are spiked into each sample. Therefore, matrix effects on method performance may be judged by the recovery of these analogs. Sample analysis acceptance is controlled by the performance of these analogs in each sample. A Matrix

Spike/Matrix Spike Duplicate (MS/MSD or MS/SD) pair are extracted at the client's request only. Method 8290A does not address analysis of MS/MSD. An exception to this rule is a batch containing South Carolina samples for Method 8290. These batches must have an MS/MSD prepared. However, South Carolina requires Method 8290A after December 31, 2008. An MS/MSD pair are aliquots of a selected field sample spiked with analytes of known identity and concentration. When requested by the client, the MS/MSD pair shall be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spike analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, the LCS, the selected field sample, and the MS/MSD may be required after evaluation and review. Matrix Spike/ Matrix Spike Duplicates are not generally applicable for air samples due to the difficulty in collecting identical or representative samples. An LCS/LCSD may be extracted to show precision of the extraction and analysis process.

9.3.1. Matrix Spike (MS): A sample, which is spiked with a known amount of the matrix spike fortification solution prior to the extraction step. The recoveries of the matrix spike compounds are determined; they are used to estimate the effect of the sample matrix upon the analytical methodology.

9.3.2. Matrix Spike Duplicate (MSD): A second portion of the same sample as used in the matrix spike analysis and which is treated like the matrix spike sample.

9.3.3. Locate the sample for the MS and MSD analyses (the sample may be labeled "double volume").

9.3.4. Add an appropriate volume of the matrix spike fortification solution, adjusting the fortification level as specified in Table 1, under IS Spiking Levels.

9.3.5. Analyze the MS and MSD samples as described in Section 11.

9.3.6. The results obtained from the MS and MSD samples (percent recovery and concentrations of 2,3,7,8-substituted PCDDs/PCDFs) should agree within 20 percent relative difference. Report all results and flag outliers.

9.3.7. Isotope dilution analyte recoveries are flagged if they are outside the recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any isotope dilution analyte is less than 10:1.

9.4. Duplicates

9.4.1. Upon client request, duplicates may be processed. Locate the sample specified for duplicate analysis, and prepare and analyze a second 10-g soil or sediment sample portion or 1-L water sample, or an appropriate amount of the type of matrix under consideration. Duplicate samples are not generally

applicable for air samples due to the difficulty in collecting identical or representative samples. A duplicate injection of a sample extract may be performed to display instrument precision.

9.4.1.1. The results of the laboratory duplicates (percent recovery and concentrations of 2,3,7,8-substituted PCDD/PCDF compounds) should agree within 25 percent relative difference. Report all results and flag outliers.

9.4.2. Isotope dilution analyte recoveries are flagged if they are outside the recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any isotope dilution analyte is less than 10:1.

9.5. Surrogate/Clean Up Internal Standard

A surrogate compound may be spiked into all air media samples prior to collection. For all other matrices, a clean up internal standard is spiked following extraction and just prior to cleanup, in order to monitor relative loss of isotope dilution analyte during both extraction and cleanup.

9.6. An Instrument Blank must be evaluated after calibration standards are injected and before sample analysis may begin. This may be accomplished by analysis of a method blank. If a method blank is not available, an instrument blank must be analyzed.

9.6.1. An instrument blanks consists of solvent (isooctane, toluene, or tetradecane). It is evaluated by inspection for contamination that may affect sample analysis.

9.7. Isotope Dilution Analytes

9.7.1. Isotope dilution analytes must be spiked into all samples, QC samples, and included in all calibrations.

9.7.2. For each sample and QC aliquot, calculate the percent recovery. The percent recovery should be between 40 percent and 135 percent for all nine isotope dilution analytes.

9.7.3. A low or high percent recovery for a blank does not require discarding the analytical data but it may indicate a potential problem with future analytical data. Isotope dilution analyte recoveries are flagged if they are outside the recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any isotope dilution analyte is less than 10:1.

9.8. Recommended Corrective Actions and Troubleshooting Steps

- Verify satisfactory instrument performance.
- If possible, verify that no error was made while weighing the sample portions.

- Review the analytical procedures with the performing laboratory personnel.

10. CALIBRATION

Calibration and Standardization requires a check of mass resolution (tuning), a check of chromatographic resolution, a verification of switching times (i.e. descriptors), and a calibration curve verification.

10.1. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to Policy CA-Q-P-003, "Calibration Curves and Selection of Calibration Points".

10.2. Tuning (Mass Resolution Check)

10.2.1. The mass spectrometer must be operated in the electron ionization mode. A static resolving power of at least 10,000 (10 percent valley definition) must be demonstrated at appropriate masses before any analysis is performed. Corrective actions must be implemented whenever the resolving power does not meet the requirement.

10.2.2. Chromatography time for PCDDs and PCDFs exceeds the long-term mass stability of the mass spectrometer. Because the instrument is operated in the high-resolution mode, mass drifts of a few ppm (e.g., 5 ppm in mass) can have serious adverse effects on instrument performance. Therefore, a mass-drift correction is mandatory. To that effect, it is recommended to select a lock-mass ion from the reference compound (PFK is recommended) used for tuning the mass spectrometer. The selection of the lock-mass ion is dependent on the masses of the ions monitored within each descriptor. Table 6 offers some suggestions for the lock-mass ions. However, an acceptable lock-mass ion at any mass between the lightest and heaviest ion in each descriptor can be used to monitor and correct mass drifts. The level of the reference compound (PFK) metered into the ion chamber during HRGC/HRMS analyses should be adjusted so that the amplitude of the most intense selected lock-mass ion signal (regardless of the descriptor number) does not exceed 10 percent of the full-scale deflection for a given set of detector parameters. Under those conditions, sensitivity changes that might occur during the analysis can be more effectively monitored.

NOTE: Excessive PFK (or any other reference substance) may cause noise problems and contamination of the ion source resulting in downtime for source cleaning.

10.2.3. By using a PFK molecular leak, tune the instrument to meet minimum required resolving power of 10,000 (10 percent valley) at m/z 292.9825 (PFK) or any other reference signal close to m/z 303.9016 (from TCDF).

10.2.4. Documentation of the instrument resolving power must then be accomplished

by recording the peak profile for all the descriptors. The minimum resolving power of 10,000 must be demonstrated on the high-mass ion while it is transmitted at a lower accelerating voltage than the low-mass reference ion, which is transmitted at full sensitivity. The format of the peak profile representation (Figure 3) must allow manual determination of the resolution, i.e., the horizontal axis must be a calibrated mass scale (amu or ppm per division). The result of the peak width measurement (performed at 5 percent of the maximum, which corresponds to the 10-percent valley definition) must appear on the hard copy and cannot exceed 100 ppm at m/z 380.9760 (or 0.038 amu at that particular mass).

10.3. Performance Checks

10.3.1. At the beginning of each 12-hour period during which samples are to be analyzed, aliquots of the 1) GC column performance check solution and 2) high-resolution concentration calibration solution No. 4 (HRCC-4) shall be analyzed to demonstrate adequate GC resolution and sensitivity, response factor reproducibility, and mass range calibration, and to establish the PCDD/PCDF retention time windows. (Note: A HRCC-3 or HRCC-5 may be acquired to meet the requirement of #2 above. This is to provide documentation of consistency for varying concentration levels, and to meet NELAC requirements). A mass resolution check shall also be performed to demonstrate adequate mass resolution using an appropriate reference compound (PFK is recommended). If the required criteria are not met, remedial action must be taken before any samples are analyzed. The mass resolution check will be taken at the beginning and completion of an analytical sequence. An analytical sequence may contain one or more 12 hour periods.

10.3.1.1. Method blanks or solvent blanks are used to demonstrate that the analytical system is free of contamination after the analysis of calibration standards or high level samples. The blank must demonstrate that the system has returned to appropriate background levels prior to continued analysis.

10.3.2. At a minimum, the ions listed in Table 6 for each of the five SIM descriptors must be monitored. Note that the PeCDF masses (M+2 & M+4) are also monitored in the first descriptor. This is because the first PeCDF isomer elutes closely to the final tetra isomer. The selection (Table 6) of the molecular ions M and M+2 for ^{13}C-HxCDF and ^{13}C-HpCDF rather than M+2 and M+4 (for consistency) is to eliminate, even under high-resolution mass spectrometric conditions, interferences occurring in these two ion channels for samples containing high levels of native HxCDDs and HpCDDs. It is important to maintain the same set of ions for both calibration and sample extract analyses. The recommended mass spectrometer tuning conditions are

based on the groups of monitored ions shown in Table 6.

10.3.2.1. The GC column performance check mixture, high-resolution concentration calibration solutions, and the sample fortification solutions may be obtained from the EMSL-CIN. However, if not available from the EMSL-CIN, standards can be obtained from other sources, and solutions can be prepared in the laboratory. Concentrations of all solutions containing 2,3,7,8-substituted native PCDDs/PCDFs, must be verified by comparison with second-source standard solutions.

10.4. Initial Calibration

Initial calibration is required before any samples are analyzed for PCDDs and PCDFs. Initial calibration is also required if any routine calibration (Section 10.5) does not meet the required criteria listed in Section 10.6.

10.4.1. Five high-resolution concentration calibration solutions, listed in Table 5, must be used for the initial calibration.

10.4.2. Tune the instrument with PFK.

10.4.3. Inject 1 or 2 µL of the GC column performance check solution and acquire SIM mass spectral data as described earlier in Section 6.1.4. The total cycle time must be ≤ 1 second. This is analyzed prior to a calibration curve to set descriptor windows only and may not otherwise be documented.

10.4.3.1. Select the injection volume based upon the expected target analyte concentration, or expected matrix interferences.

10.4.3.2. The same injection volume must be used for all samples, QC, and standards.

10.4.4. By using the same GC and mass spectrometer conditions that produced acceptable results with the column performance check solution, analyze a 1 or 2-µL portion of each of the five concentration calibration solutions once with the following mass spectrometer operating parameter.

10.4.4.1. The total cycle time for data acquisition must be < 1 second. The total cycle time includes the sum of all dwell times and voltage reset times.

10.4.4.2. Acquire SIM data for all the ions listed in the five descriptors of Table 6.

10.4.4.3. The ratio of integrated ion current for the ions appearing in Table 9 (homologous series quantification ions) must be within the indicated control limits (set for each homologous series).

10.4.4.4. The ratio of integrated ion current for the ions belonging to the ^{13}C labeled isotope dilution analytes and internal standards must be within the control limits stipulated in Table 9.

NOTE: Section 10.4.3 requires that ion ratios be within the specified control limits simultaneously in one run. It is the laboratory's responsibility to take corrective action if the ion abundance ratios are outside the limits.

10.4.5. For each SICP and for each GC signal corresponding to the elution of a target analyte and of its labeled standards, the signal-to-noise ratio (S/N) must be better than or equal to 10. This measurement is suggested for any GC peak that has an apparent S/N of less than 5:1. The result of the calculation must appear on the SICP above the GC peak in question.

10.4.5.1. Referring to Table 5, calculate the 17 relative response factors (RRF) for unlabeled target analytes [RRF(n); n=1 to 17] relative to their appropriate isotope dilution analytes (Table 5) and the nine RRFs for the labeled ^{13}C isotope dilution analytes [RRF(m); m=18 to 26] relative to the two internal standards according to the following formulae:

$$RRF(n) = \frac{A_x \times Q_{IDA}}{Q_x \times A_{IDA}} \qquad RRF(m) = \frac{A_{IDA} \times Q_{IS}}{Q_{IDA} \times A_{IS}}$$

Where:

A_x = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 5) for unlabeled PCDDs/PCDFs,

A_{IDA} = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 5) for the labeled isotope dilution analytes,

A_{IS} = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for the labeled internal standards,

Q_{IDA} = quantity of the isotope dilution analyte injected (pg),

Q_{IS} = quantity of the internal standard injected (pg), and

Q_x = quantity of the unlabeled PCDD/PCDF analyte injected (pg).

The RRF (n) and RRF (m) are dimensionless quantities; the units used to express Q_{IDA}, Q_{IS}, and Qx must be the same.

10.4.5.2. Calculate the RRF(n)s and their respective percent relative standard deviations (%RSD) for the five calibration solutions:

$$\overline{RRF}(n) = (1/5)\sum_{j=1}^{5} RRF_j(n)$$

Where n represents a particular PCDD/PCDF (2,3,7,8-substituted) congener (n = 1 to 17; Table 5), and j is the injection number (or calibration solution number; j = 1 to 5).

10.4.5.3. The relative response factors to be used for the determination of the concentration of total isomers in a homologous series are calculated as follows:

10.4.5.3.1. For congeners that belong to a homologous series containing only one isomer (e.g., OCDD and OCDF) or only one 2,3,7,8-substituted isomer (Table 4; TCDD, PeCDD, HpCDD, and TCDF), the mean RRF used will be the same as the mean RRF determined in Section 10.3.5.2.

NOTE: The calibration solutions do not contain ^{13}C-OCDF as an isotope dilution analyte. This is because a minimum resolving power of 12,000 is required to resolve the [M+6]+ ion of ^{13}C-OCDF from the [M+2]+ ion of OCDD (and [M+4]+ from ^{13}C-OCDF with [M]+ of OCDD). Therefore, the RRF for OCDF is calculated relative to ^{13}C-OCDD.

10.4.5.3.2. For congeners that belong to a homologous series containing more than one 2,3,7,8-substituted isomer (Table 4), the mean RRF used for those homologous series will be the mean of the RRFs calculated for all individual 2,3,7,8-substituted congeners using the equation below:

$$\overline{RRF}(k) = (1/t)\sum_{n=1}^{t} RRF_n$$

Where:

k = 27 to 30, with 27 = PeCDF;

28 = HxCDF; 29 = HxCDD; and 30 = HpCDF,

t = total number of 2,3,7,8-substituted isomers present in the calibration solutions (Table 5) for each homologous series (e.g., two for PeCDF, four for HxCDF, three for HxCDD, two for HpCDF).

NOTE: Presumably, the HRGC/HRMS response factors of different isomers within a homologous series are different. However, this analytical protocol will make the assumption that the HRGC/HRMS responses of all isomers in a homologous series that

do not have the 2,3,7,8-substitution patterns are the same as the responses of one or more of the 2,3,7,8-substituted isomer(s) in that homologous series.

10.4.5.4. Relative response factors [RRF(m)] to be used for the determination of the percent recoveries for the nine isotope dilution analytes are calculated as follows:

$$RRF(m) = \frac{A_{IDA}^{m} \times Q_{IS}}{Q_{IDA}^{m} \times A_{IS}}$$

$$\overline{RRF}(m) = (1/5) \sum_{j=1}^{5} RRF_j(m)$$

Where:

m =	18 to 26 (congener type)
j =	1 to 5 (injection number),
A_{IDA}^{m} =	sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for a given isotope dilution analyte (m = 18 to 26),
A_{IDA} =	sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for a given isotope dilution analyte (m = 18 to 26),
Q_{IDA} & Q_{IDA}^{m} =	quantities of, respectively, the internal standard (rs) and a particular isotope dilution analyte (m) injected (pg),
RRF(m) =	relative response factor of a particular isotope dilution analyte (m) relative to an appropriate internal standard, as determined from one injection, and
$\overline{RRF(m)}$ =	calculated mean relative response factor of a particular isotope dilution analyte, as determined from the five initial calibration injections (j).

10.5. Criteria for acceptable calibration

The criteria listed below for acceptable calibration must be met before sample analysis is performed.

10.5.1. The percent relative standard deviations for the mean response factors [RRF(n) and RRF(m)] from the 17 unlabeled standards must be ≤ 20 percent, and those for the nine labeled reference compounds must be ≤ 30 percent.

Note: If Method 8290A criteria are required for the project then both the percent standard relative standard deviation for the mean response factors for the 17 unlabeled standards and the nine labeled reference compounds must be ≤ 20 percent.

10.5.2. The signal/noise ratio (S/N) for the GC signals present in every SICP (including the ones for the labeled standards) must be ≥ 10.

10.5.3. The isotopic ratios (Table 9) must be within the specified control limits.

NOTE: If the criterion for acceptable calibration listed in Section 10.4.1 is met, the analyte-specific RRF can then be considered independent of the analyte quantity for the calibration concentration range. The mean RRFs will be used for all calculations until the routine calibration criteria (Section 10.6) are no longer met. At such time, new mean RRFs will be calculated from a new set of injections of the calibration solutions.

10.6. Initial calibration verification standard (ICV): A second source calibration standard is analyzed following the initial calibration curve, prior to samples. Criteria are as follows: All native (unlabelled) compounds must be within ± 20% of expected value. IDA (labeled) compounds must be within ± 30% of expected value.

10.7. Routine Calibration (continuing calibration check)

Routine calibrations must be performed at the beginning of (following a successful tune and GC column performance check) and after a 12 hour period. The routine calibration initiates the 12 hour clock during which samples may be subsequently analyzed. The last sample in the sequence must be injected within 12 hours of the routine calibration, followed by the analysis of a closing calibration check. An acceptable closing calibration check standard may be used to initiate the next 12 hour analysis sequence when consecutive acquisition sequences occur. The ending mass resolution check shall be performed after the closing calibration check of an analysis acquisition sequence or after the final bracketing standard when consecutive 12 hour acquisition sequences are run.

10.7.1. Inject 1 or 2 µL of the concentration calibration solution HRCC-4 containing 10 pg/µL of tetrachlorinated congeners, 50 pg/µL of penta-, hexa-, and heptachlorinated congeners, 100 pg/µL of octachlorinated congeners, and the respective isotope dilution analyte and internal standards (Table 5). By using the same HRGC/HRMS conditions as used in Sections 6.1.4 through 6.2, determine and document an acceptable calibration as provided in Section 10.6.

10.8. Criteria for Acceptable Routine Calibration

The following criteria must be met before further analysis is performed. If these criteria are not met, corrective action must be taken, including recalibration if needed.

10.8.1. The measured RRFs [RRF(n)] for the unlabeled standards obtained during the

opening continuing calibration must be ± 20 percent of the mean values established during the initial calibration (Section 10.3.5.)

10.8.1.1. The bracketing continuing calibration must be ± 20% of the average RRF calculated from the initial calibration.

10.8.1.1.1. If the target compounds in the ending standard are less than or equal to ± 20% of the average RRF from the initial calibration, the RRFs of the initial calibration shall be used to quantitate the unlabeled isomers.

10.8.1.1.2. If the target analytes are greater than ± 20% but less or equal to ±25% and the samples are non-detect, the data is acceptable and this anomaly is documented. If these isomers are greater than ± 20% but less or equal to ±25% and are positive, an average RRF of the initial and ending daily standard is calculated and used to quantitate the concentration of the affected congener, and the anomaly is documented.

10.8.1.1.3. If the percent deviation of unlabeled compounds exceeds ± 25%, a new initial calibration is initiated within 2 hours following the analysis of the samples. Otherwise, reanalyze all sample extracts with positives for the failed target compounds.

10.8.2. The measured RRFs [RRF(m)] for the labeled standards obtained during the opening continuing calibration must be less than or equal to ± 30 percent of the mean values established during the initial calibration (Section 10.1.5).

10.8.2.1. The bracketing continuing calibration must be ± 30% of the average RRF calculated from the initial calibration.

10.8.2.1.1. If the labelled compounds in the ending standard are less than or equal to ±30% of the average RRF from the initial calibration, the RRFs of the initial calibration shall be used to quantitate the labeled isomers.

10.8.2.1.2. If the isotope dilution analyte analytes are greater than ± 30% but less or equal to ±35%, an average RRF of the initial and ending daily standards is calculated and used to quantitate the concentration of the affected congener.

10.8.2.1.3. If the percent deviation of labeled compounds exceeds ± 35%, reanalyze samples if adversely impacted.

10.8.3. The ion-abundance ratios (Table 9) must be within the allowed control limits.

10.8.4. If either criteria in Sections 10.7.1 or 10.7.2 are not met, additional samples may not be analyzed. Sample data collected must be evaluated for usability. Narrate any reported data from the analytical sequence. If the ion-abundance ratio criterion is not satisfied, refer to the note in Section 10.4.3 for resolution.

10.8.5. If the above criteria (Section 10.7) cannot be satisfied, the entire initial calibration process (Section 10.4) must be repeated.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Sample Dilution Procedure – Simple Dilutions

Dilutions from 2X to 20X can be achieved without respiking the final extract. The calculation to determine the final extract concentration is as follows:

(Concentration of the original extract) x (amount of aliquot taken) x (volume of diluted extract) = final concentration of dilution.

Ex: 20X dilution of original 10 g/20 µL sample

(10 g/20 µL) x (2 µL aliquot + 38 µL keeper) = 1 g/40 µL FV

Record the final sample concentration on the extract label.

11.3. Sample Dilution Procedure – Complex Dilutions

Complex dilution requiring respiking of IDA and IS: Dilutions greater than 20x must be done by diluting and respiking the extract with IDA and IS. This procedure may require serial dilution to be performed. If this procedure is done, then the sample size must be adjusted to reflect the aliquot taken.

Ex. 100X dilution (original sample with 10 g/20 µL final volume)

Take a 2 µL aliquot (1/10 of original sample) and add 18 µL of solvent keeper. Take a 2 µL aliquot of the dilution (1/100 of the original sample), respike with 1 mL IDA and 20 µL IS, reduced to 20 µL FV.

Record the final sample concentration of the extract label.

11.4. Analytical Procedures

11.4.1. Inject a 1 or 2 µL aliquot of the extract into the GC, operated under the conditions previously used (Section 6.2) to produce acceptable results with the performance check solution.

11.4.2. Acquire SIM data according to Section 6.1.4. Use the same acquisition and mass spectrometer operating conditions previously used to determine the relative response factors (Section 10). Ions characteristic for polychlorinated diphenyl ethers are included in the descriptors listed in Table 6. Their presence is used to monitor their interference during the characterization of PCDFs.

12. CALCULATIONS/DATA REDUCTION

12.1. Identification Criteria

For a gas chromatographic peak to be identified as a PCDD or PCDF, it must meet all of the following criteria:

12.1.1. Retention Times

12.1.1.1. For 2,3,7,8-substituted congeners, which have an isotopically labeled isotope dilution analyte or internal standard present in the sample extract, the retention time (at maximum peak height) of the sample components (i.e., the two ions used for quantitation purposes listed in Table 6) must be within -1 and +3 seconds of the retention time of the peak for the isotopically labeled isotope dilution analyte or internal standard at m/z corresponding to the first characteristic ion (of the set of two; Table 6) to obtain a positive identification of these nine 2,3,7,8-substituted PCDDs/PCDFs and OCDD.

12.1.1.2. For 2,3,7,8-substituted compounds that do not have an isotopically labeled isotope dilution analyte present in the sample extract, the relative retention time (relative to the appropriate isotope dilution analyte) must fall within 0.005 relative retention time units of the relative retention times measured in the daily routine calibration. Identification of OCDF is based on its retention time relative to ^{13}C-OCDD as determined from the daily routine calibration results.

12.1.1.3. For non-2,3,7,8-substituted compounds (tetra through octa; totaling 193 congeners), the retention time must be within the corresponding homologous retention time windows established by analyzing the column performance check solution.

12.1.1.4. The ion current responses for both ions used for quantitative purposes (e.g., for TCDDs: m/z 319.8965 and 321.8936) must reach a maximum simultaneously (± 2 seconds).

12.1.1.5. The ion current responses for both ions used for the labeled standards (e.g., for ^{13}C-TCDD: m/z 331.9368 and m/z 333.9339) must reach a maximum simultaneously (± 2 seconds).

12.1.2. Ion Abundance Ratios

The integrated ion current for the two ions used for quantitation purposes must have a ratio between the lower and upper limits established for the homologous series to which the peak is assigned. See Table 9.

12.1.3. Signal-To-Noise Ratio

All ion current intensities must be >2.5 times noise level for positive identification of the PCDD/PCDF compound or a group of coeluting isomers. Figure 4 describes the procedure to be followed for the determination of the S/N.

12.1.4. Polychlorinated Diphenyl Ether Interferences

In addition to the above criteria, the identification of a GC peak as a PCDF can only be made if no signal having a S/N >2.5 is detected, at the same retention time (± 2 seconds), in the corresponding polychlorinated diphenyl ether (PCDPE, Table 6) channel.

12.2. For gas chromatographic peaks that have met the criteria outlined above, calculate the concentration of the PCDD or PCDF compounds using the formula:

$$C_x = \frac{A_x \times Q_{IDA}}{A_{IDA} \times W \times RRF(n)}$$

Where:

C_x = concentration of unlabeled PCDD/PCDF congeners (or group of coeluting isomers within an homologous series) usually in pg/g or pg/L,

Ax = sum of the integrated ion abundances of the quantitation ions (Table 6) for the unlabeled PCDD/PCDFs,

A_{IDA} = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled isotope dilution analytes,

Q_{IDA} = quantity, in pg, of the isotope dilution analyte added to the sample before extraction,

W = sample size in g (if solid) or L (if liquid).

$RRF(n)$ = Calculated mean relative response factor for the analyte [RRF(n) with n = 1 to 17; Section 10.3.5].

If the analyte is identified as one of the 2,3,7,8-substituted PCDDs or PCDFs, RRF(n) is the value calculated using the equation in Section 10.3.5.1. However, if it is a non-2,3,7,8-substituted congener, the RRF(k) value is the one calculated using the equation in Section 10.3.5.3.2 [RRF(k) with k = 27 to 30].

12.3. Calculate the percent recovery of the nine isotope dilution analytes measured in the sample extract, using the formula:

$$\text{Isotope Dilution Analytes Percent Recovery} = \frac{A_{IDA} \times Q_{IS}}{Q_{IDA} \times A_{IS} \times RRF(m)} \times 100$$

Where:

A_{IDA} = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled isotope dilution analytes,

A_{IS} = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled internal standard; the selection of the internal standard depends on the type of congeners (see Table 5, footnotes),

Q_{IDA} = Quantity, in pg, of the isotope dilution analyte added to the sample before extraction,

Q_{IS} = Quantity, in pg, of the internal standard added to the cleaned-up sample residue before HRGC/HRMS analysis, and

$RRF(m)$ = calculated mean relative response factor for the labeled isotope dilution analyte relative to the appropriate (see Table 5, footnotes) internal standard. This represents the mean obtained in Section 10.3.5.4 [RRF(m) with m = 18 to 26].

12.4. If the concentration in the final extract of any of the fifteen 2,3,7,8-substituted PCDD/PCDF compounds (Table 3) exceeds the upper method calibration limit (MCL) for that compound listed in Table 1, the linear range of response versus concentration may have been exceeded. In such cases, the following corrective actions will be undertaken:

12.4.1. If the signal for the analyte has saturated the detector, a single dilution and reanalysis of the extract will be made in an attempt to bring the signal within the range of the detector. If the measured concentration of the analyte is still above the MCL, the reported concentration for the analyte will be qualified appropriately. Some programs, such as DOD/QSM QSM, require all compounds to be within the linear calibration range in which a serial dilution must be performed to achieve acceptable quantitation.

12.4.2. If the signal for the analyte is above the MCL but does not saturate the detector, the concentration will be reported and qualified appropriately. Some programs, such as DOD/DOE QSM, require all compounds to be within the linear calibration range in which a serial dilution must be performed to achieve acceptable quantitation.

12.5. In either case, **with the approval of the client**, the sample may be re-extracted and/or re-analyzed with one or more of the following adjustments made to the analytical procedure in order to provide a concentration which meets client-specific data quality objectives.

12.5.1. Extraction and analysis of a one tenth aliquot. This is appropriate if it will provide analyte concentration within the MCL and a representative sample aliquot.

12.5.2. Extraction of an aliquot large enough to be representative with an increased concentration of isotope dilution analyte and surrogate spike components added prior to the extraction. The extract is then diluted either prior to or after the cleanup procedures.

12.5.3. Dilution of the original extract. Isotope dilution analyte components are re-spiked at an appropriate level prior to analysis. In this case, the isotope dilution analyte recoveries are taken from the original analysis.

12.6. For the other congeners (including OCDD and OCDF), however, report the measured concentration and indicate that the value exceeds the upper calibration standard.

12.7. The total concentration for each homologous series of PCDD and PCDF is calculated by summing up the concentrations of all positively identified isomers of each homologous series. Therefore, the total should also include the 2,3,7,8-substituted congeners. The total number of GC signals included in the homologous total concentration value may be specified in the report.

12.8. Sample-Specific Estimated Detection Limit

The sample-specific estimated detection limit (EDL) or estimated quantitation limit (EQL, 8290A) is the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background signal level. An EDL/EQL is calculated for each 2,3,7,8-substituted congener that is not identified, regardless of whether or not other non-2,3,7,8-substituted isomers are present. Two methods of calculation can be used, as follows, depending on the type of response produced during the analysis of a particular sample.

12.8.1. Samples giving a response for both quantitation ions (Tables 6 and 9) that is less than 2.5 times the background level.

Use the expression for EDL/EQL (specific 2,3,7,8-substituted PCDD/PCDF) below to calculate an EDL/EQL for each absent 2,3,7,8-substituted PCDD/PCDF (i.e., S/N <2.5). The background level is determined by measuring the range of the noise (peak to peak) for the two quantitation ions (Table 6) of a particular 2,3,7,8-substituted isomer within an homologous series, in the region of the SICP trace corresponding to the elution of the isotope dilution analyte (if the congener possesses an isotope dilution analyte) or in the region of the SICP where the congener is expected to elute by comparison with the routine calibration data (for those congeners that do not have a ^{13}C-labeled standard), multiplying that noise height by 2.5, and relating the product to an estimated concentration that would produce that product height.

NOTE: The quantitation ions for both the unlabeled PCDDs/PCDFs and their isotope dilution analyte must be consistently paired (using either both lighter mass ions or both heavier mass ions).

Use the formula:

$$EDL_{Specific\,2,3,7,8-subst.\,PCDD/PCDF} = \frac{2.5 \times H_x \times Q_{IDA}}{H_{IDA} \times W \times RRF(n)}$$

Where:

EDL = estimated detection limit for homologous 2,3,7,8-substituted PCDDs/PCDFs. (also EQL for Method 8290A)

H_x = height of the average noise for one of the quantitation ions (Table 6) for the unlabeled PCDDs/PCDFs.

H_{IDA} = height of one of the quantitation ions (Table 6) for the labeled isotope dilution analytes.

W, RRF (n), and Q_{IDA} retain the same meanings as defined in Section 12.2

12.8.2. Samples characterized by a response above the background level with a S/N of at least 2.5 for at least one of the quantitation ions (Tables 6 and 9).

When the response of a signal having the same retention times as a 2,3,7,8-substituted congener has a S/N in excess of 2.5 and does not meet any of the other qualitative identification criteria listed in Section 12.1, calculate the "Estimated Maximum Possible Concentration" (EMPC) according to the expression shown in Section 12.1, except that Ax in Section 12.1 should represent the sum of the area under the smaller peak and of the other peak area calculated using the theoretical chlorine isotope ratio. Alternatively, an EDLEQL can be calculated using the above formula and the height of one of the ions as appropriate.

12.9. The relative percent difference (RPD) is calculated as follows:

$$RPD = \frac{|S_1 - S_2|}{(S_1 + S_2)/2} \times 100$$

S_1 and S_2 represent sample and duplicate sample results.

12.10. The 2,3,7,8-TCDD toxic equivalents (TEQ) of PCDDs and PCDFs present in the sample are calculated at the data user's request. This method assigns a 2,3,7,8-TCDD toxicity equivalency factor (TEF) to each of the seventeen 2,3,7,8-substituted PCDDs and PCDFs (Table 10). The 2,3,7,8-TCDD equivalent of the PCDDs and PCDFs present in the sample is calculated by summing the TEF times their concentration for each of the compounds or groups of compounds listed in Table 10.

12.11. Two-GC Column TEF Determination

12.11.1. The concentration of 2,3,7,8-TCDD (see note below), is calculated from the analysis of the sample extract on the 60m DB-5 fused silica capillary column. The chromatographic separation of this isomer must be $\leq$ 25% valley.

12.11.2. For samples that have a positive result for 2,3,7,8-TCDF on the DB-5 column, the extract is reanalyzed on a 30m DB-225 fused silica column. The GC/MS conditions are altered so that only the first descriptor (Table 6) is used. The reported concentration for 2,3,7,8-TCDF is then the result above the lower calibration limit is calculated from the DB-225 analysis. The chromatographic separation between 2,3,7,8-TCDF and any other unlabeled TCDF isomers must be < 25% valley using the column performance check solution for the DB-225 column. Concentration calculations are performed as in Section 12.1 through 12.6.

12.11.3. A DB-225 column can be used in the quantitative analysis of 2,3,7,8-TCDF and 2,3,7,8-TCDD analytes. Since the DB-225 cannot resolve 2,3,7,8-TCDD any positively identified 2,3,7,8-TCDD which exceeds the reporting limit shall be confirmed on a DB-5 column.

12.11.4. For a gas chromatographic peak to be identified as a 2,3,7,8-substituted PCDD/PCDF congener, it must meet the ion abundance (Section 11.5.4) and signal-to-noise ratio criteria. In addition, the retention time identification criterion described in Section 11.5.4 applies here for congeners for which a carbon-labeled analog is available in the sample extract. However, the relative retention time (RRT) of the 2,3,7,8-substituted congeners for which no carbon-labeled analogs are available must fall within 0.005 units of the carbon-labeled standard RRT. Experimentally, this is accomplished by using the attributions described in Table 11 and the results from the routine

calibration run on the DB-5 column.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

It must be documented that all applicable system performance criteria specified were met before analysis of any sample is performed. Table 7 provides recommended GC conditions that can be used to satisfy the required criteria. A GC column performance check is only required at the beginning of each 12-hour period during which samples are analyzed.

13.5. GC Column Performance

13.5.1. Inject 1 or 2 µL of the column performance check solution and acquire selected ion monitoring (SIM) data as described in Section 6.1.4 within a total cycle time of < 1 second.

13.5.2. The chromatographic separation between 2,3,7,8-TCDD and the peaks representing any other TCDD isomers must be resolved with a valley of ≤ 25

percent (Figure 2),

Where:

$$\text{Valley Percent} = (x/y) \times 100$$

x = measured as in Figure 2 from the 2,3,7,8-closest TCDD eluting isomer,

y = the peak height of 2,3,7,8-TCDD

13.5.3. It is the responsibility of the laboratory to verify the conditions suitable for the appropriate resolution of 2,3,7,8-TCDD from all other TCDD isomers. The GC column performance check solution also contains the known first and last PCDD/PCDF eluters under the conditions specified in this protocol. Their retention times are used for qualitative and quantitative purposes. The peak for 2,3,7,8-TCDD must be labeled on the chromatograms. The chromatograms showing the first and last eluters of a homologous series must be included.

13.5.4. The retention times for the switching of SIM ions characteristic of one homologous series to the next higher homologous series must be indicated in the SICP. Accurate switching at the appropriate times is absolutely necessary for accurate monitoring of these compounds.

14. 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 Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. 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 SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Autovials containing assorted solvents and extracts. As the autovials are removed from the instrument after analysis, they are collected in archive boxes and retained pending additional instructions. When no longer needed, the archive boxes are moved to the waste disposal area for disposal as PCB waste.

16. REFERENCES/CROSS REFERENCES

16.1. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update III. Method 8290 Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by high-Resolution Mass Spectrometry September 1994.

16.2. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update IV. Method 8290A Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by high-Resolution Mass Spectrometry February 2007.

16.3. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update III. Method 0023A, Sampling Method for Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans Emissions from Stationary Sources. December 1996.

16.4. Compendium Method TO-9A “Determination of Polychlorinated, Polybrominated, and Brominated, Chlorinated Dibenzo-p-dioxins and Dibenzofurans in Ambient Air”, EPA compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, second edition, January 1997.

16.5. Protocol for the Analysis of 2,3,7,8-TCDD by HRGC/HRMS". J. S. Stanley and T. M. Sack, EPA 600/4-86-004.

16.6. “Safety in Academic Chemistry Laboratories", American Chemical Society Publication, Committee on Chemical Safety (3rd Edition, 1979.)

16.7. “Carcinogens - Working with Carcinogens". Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control. National Institute for Occupational Safety and Health. Publication No. 77-206, August 1977.

16.8. "OSHA Safety and Health Standards, General Industry", (29 CFR 1910) Occupational Safety and Health Administration, OSHA 2206 (revised January 1976).

17. METHOD MODIFICATIONS

17.1. Modifications from EPA 8290 and EPA 8290A

17.1.1. The methods specify that 2 µL injections are used throughout the analysis. If an instrument demonstrates adequate sensitivity and chromatographic resolution, then the analyst may use 1 µL injections for all performance checks, standards, QC samples, and samples.

17.1.2. In Section 2.7 of Method 8290 and 8290A, a retention time window of 0.005 RT units is used to tentatively identify unlabeled PCDD/PCDFs for which there are no corresponding labeled isotope dilution analytes. All available labeled isotope dilution analytes are used; therefore, a retention time window

of -1 to +3 seconds is used to identify all compounds. See Section 7.8.4.1 of Method 8290 and 7.9 of Method 8290A.

17.1.3. Tetradecane instead of nonane is used as the final solvent to increase the stability of extracts and standards. Tetradecane is less volatile than nonane. Loss of analyte as a result of solvent incompatibility is monitored through recovery checks and calibration acceptance criteria.

17.2. Modifications from TO-9A method

17.2.1. The ^{37}Cl-2,3,7,8-TCDD surrogate is present at varying levels in the calibration curve (0.5-200 pg/ µL).

17.2.2. The laboratory uses 2 labeled internal standards for the quantitation of labeled isotope dilution analytes.

17.2.3. The final volume is adjusted to 20 µL in tetradecane.

17.2.4. Calibration and quantitation are performed in accordance to this SOP.

18. ATTACHMENTS

18.1. Table 1 - Types of Matrices

18.2. Table 2 - Composition of the Sample Fortification and Internal Standard Solutions.

18.3. Table 3 - The Fifteen 2,3,7,8-Substituted PCDD and PCDF Congeners

18.4. Table 4 - Isomers of Chlorinated Dioxins and Furans

18.5. Table 5 - Concentrations of Calibration Solutions

18.6. Table 6 - Ions Monitored for PCDDs/PCDFs

18.7. Table 7 - Recommended GC Operating Conditions

18.8. Table 8 - Congeners in the GC Performance Evaluation Solution (DB-5)

18.9. Table 9 - Theoretical Ion Abundance Ratios and Control Limits

18.10. Table 10 - 2,3,7,8-TCDD Equivalent Factors

18.11. Table 11 - TEF: Analyte Relative Retention Time Reference Attributes

18.12. Figure 1 - Compound Structure

18.13. Figure 2 - GC Performance Check Chromatogram on the DB-5 Column

18.14. Figure 3 - PFK Peak Profile

18.15. Figure 4 - Manual Determination of Signal-to-Noise

18.16. Appendix A - Periodic Wipe Test Performance

19. REVISION HISTORY

19.1. WS-ID-0005, Revision 7.9, Effective 10/07/2016

19.1.1. Added Section 9.6, "An Instrument Blank must be evaluated after calibration standards are injected and before sample analysis may begin. This may be accomplished by analysis of a method blank. If a method blank is not available, an instrument blank must be analyzed. "

19.1.2. Editorial changes.

19.2. WS-ID-0005 Revision 7.8, Effective 12/31/2015

19.2.1. Removed the revision history prior to 2011; it can be found in previous versions of this SOP.

19.2.2. Changed all references to DOD to be DOE inclusive – Sections 1.5, 9.1, 12.4.1, and 12.4.2.

19.2.3. Removed Section 6.1 (Table of routine and preventive maintenance may be found in the QAM)

19.2.4. Added Section 6.3 (Recommended Maintenance)

19.2.5. Inserted Section 10.6, "Initial calibration verification standard (ICV): A second source calibration standard is analyzed following the initial calibration curve, prior to samples. Criteria are as follows: All native (unlabelled) compounds must be within ± 20% of expected value. IDA (labeled) compounds must be within ± 30% of expected value."

19.3. WS-ID-0005 Revision 7.7, Effective 07/22/2015

19.3.1. Updated Copyright information on Title Page.

19.3.2. Changed Section 10.2.3 from "By using a PFK molecular leak, tune the instrument to meet minimum required resolving power of 10,000 (10 percent valley) at m/z 304.9824 (PFK)…", to "By using a PFK molecular leak, tune the instrument to meet minimum required resolving power of 10,000 (10

percent valley) at m/z 292.098285 (PFK)…". Deleted the last sentence of this Section.

19.3.3. Changed Section 10.2.4 from "Documentation of the instrument resolving power must then be accomplished by recording the peak profile of the high-mass reference signal (m/z 380.9760).." to "Documentation of the instrument resolving power must then be accomplished by recording the peak profile for all the descriptors."

19.3.4. Sections 11.2 and 11.3 – Changed all 50x dilutions to 20x and changed example in Section 11.2 .to:

Ex. (10 g/20 µL) x (2 µL aliquot + 38 µL keeper) = 1 g/40 µL FV

19.3.5. Editorial changes.

19.4. WS-ID-0005, Revision 7.6, Effective 06/06/2014

19.4.1. Changed Section 12.11.5 from "…carbon-labeled analogs are available must fall within 0.006 units…" to "…carbon-labeled analogs are available must fall within 0.005 units....".

19.4.2. Editorial changes.

19.5. WS-ID-0005, Revision 7.5, Effective 04/19/2013

19.5.1. Replaced all instances of 'internal standard' with isotope dilution analyte' and all instances of 'recovery standard' with 'internal standard' to conform with TALS naming guidelines.

19.5.2. Editorial revisions.

19.6. WS-ID-0005, Revision 7.4, Effective 01/14/2011.

19.6.1. Editorial revisions.

TABLE 1

Types of Matrices, Sample Sizes and 2,3,7,8-TCDD-Based Method Calibration Limits (Parts per Trillion)

	Water	Soil Sediment Paper Pulp	Fly Ash	Human/ Fish Tissue	Adipose Tissue	Sludges, Fuel Oil	Still-Bottom	Ambient or Source Samples
Lower MCL(a)	0.01	1.0	2.0	1.0	2.0	10	20	40
Upper MCL(a)	4.0	400	400	400	400	2000	4000	8000
Weight (g)	1000	10	10	10	10	2.0	1.0	1 sample
IDA Spiking Levels (ng)	2.0	2.0	2.0	2.0	2.0	2.0	2.0	4.0
Final Extract Volume (µL)	20	20	20	20	20	20	20	20

(a) For other congeners, multiply the values by 1 for TCDF, by 5 for PeCDD/PeCDF/HxCDD/HxCDF/HpCDD/HpCDF, and by 10 for OCDD/OCDF.

TABLE 2

Composition of the Sample Fortification and Internal Standard Solutions

Analyte	Sample Fortification Solution Concentration pg/µL; Solvent: Isooctane	Internal Standard Solution Concentration pg/µL; Solvent: Tetradecane
^{13}C-2,3,7,8-TCDD	2[a], 100[c]	--
^{13}C -2,3,7,8-TCDF	2[a], 100[c]	--
^{13}C -1,2,3,4-TCDD	--	100
^{13}C -1,2,3,7,8-PeCDD	2[a], 100[c]	--
^{13}C -1,2,3,7,8-PeCDF	2[a], 100[c]	--
^{13}C -1,2,3,6,7,8-HxCDD	2[a], 100[c]	--
^{13}C -1,2,3,4,7,8-HxCDF[d]	2[a], 100[c]	--
^{13}C -1,2,3,7,8,9-HxCDD	--	100
^{37}Cl-2,3,7,8-TCDD[b][c]	0.8[b], 100[c]	
	100[c]	
^{13}C -2,3,4,7,8-PeCDF[c]	100[c]	
^{13}C -1,2,3,6,7,8-HxCDF[c][d]	100[c]	
^{13}C -1,2,3,4,7,8-HxCDD[c]	100[c]	
^{13}C -1,2,3,4,7,8,9-HpCDD[c]	100[c]	
^{13}C -1,2,3,4,6,7,8-HpCDD	2[a], 100[c]	--
^{13}C -1,2,3,4,6,7,8-HpCDF	2[a], 100[c]	--
^{13}C -OCDD	4[a], 200[c]	--

(a) Standard 8290, 8290A, Method 23, Method 0023A, TO9 and TO9A Sample Fortification Solution concentrations
(b) Method TO9 and TO9A surrogate concentrations
(c) Method 23 and Method 0023A surrogate concentrations
(d) ^{13}C-1,2,3,6,7,8-HxCDF is used as a Sample Fortification Solution and ^{13}C -1,2,3,4,7,8-HxCDF is used as a surrogate solution in Method 0023A

TABLE 3

The Seventeen 2,3,7,8-Substituted PCDD and PCDF Congeners

PCDD	PCDF
2,3,7,8-TCDD(*)	2,3,7,8-TCDF(*)
1,2,3,7,8-PeCDD(*)	1,2,3,7,8-PeCDF(*)
1,2,3,6,7,8-HxCDD(*)	2,3,4,7,8-PeCDF
1,2,3,4,7,8-HxCDD	1,2,3,6,7,8-HxCDF
1,2,3,7,8,9-HxCDD(+)	1,2,3,7,8,9-HxCDF
1,2,3,4,6,7,8-HpCDD(*)	1,2,3,4,7,8-HxCDF(*)
1,2,3,4,5,6,7,8-OCDD(*)	2,3,4,6,7,8-HxCDF
	1,2,3,4,6,7,8-HpCDF(*)
	1,2,3,4,7,8,9-HpCDF
	1,2,3,4,5,6,7,8-OCDF

(*)The ^{13}C -labeled analog is used as an isotope dilution analyte.
(+)The ^{13}C -labeled analog is used as a internal standard.

TABLE 4

Isomers of Chlorinated Dioxins and Furans as a Function of the Number of Chlorine Atoms

# of Chlorine Atoms	# of Dioxin Isomers	# of 2,3,7,8 Isomers	# of Furan Isomers	# of 2,3,7,8 Isomers
1	2	---	4	---
2	10	---	16	---
3	14	---	28	---
4	22	1	38	1
5	14	1	28	2
6	10	3	16	4
7	2	1	4	2
8	1	1	1	1
Total	**75**	**7**	**135**	**10**

TABLE 5

High Resolution Concentration Calibration Solutions

	Compound	Concentration (ng/mL)				
RRF (n)(m)		**CS2**	**CS3**	**CS4 (ICV(6))**	**CS5**	**CS6**
	Native CDDs and CDFs					
1	2,3,7,8-TCDD	0.5	2	10	40	200
2	2,3,7,8-TCDF	0.5	2	10	40	200
3	1,2,3,7,8-PeCDD	2.5	10	50	200	1000
4	1,2,3,7,8-PeCDF	2.5	10	50	200	1000
5	2,3,4,7,8-PeCDF	2.5	10	50	200	1000
6	1,2,3,4,7,8-HxCDD	2.5	10	50	200	1000
7	1,2,3,6,7,8-HxCDD	2.5	10	50	200	1000
8	1,2,3,7,8,9-HxCDD	2.5	10	50	200	1000
9	1,2,3,4,7,8-HxCDF	2.5	10	50	200	1000
10	1,2,3,6,7,8-HxCDF	2.5	10	50	200	1000
11	1,2,3,7,8,9-HxCDF	2.5	10	50	200	1000
12	2,3,4,6,7,8-HxCDF	2.5	10	50	200	1000
13	1,2,3,4,6,7,8-HpCDD	2.5	10	50	200	1000
14	1,2,3,4,6,7,8-HpCDF	2.5	10	50	200	1000
15	1,2,3,4,7,8,9-HpCDF	2.5	10	50	200	1000
16	OCDD	5.0	20	100	400	2000
17	OCDF	5.0	20	100	400	2000
	Labeled CDDs and CDFs					
18	$^{13}C_{12}$-2,3,7,8-TCDD	100	100	100	100	100
19	$^{13}C_{12}$-2,3,7,8-TCDF	100	100	100	100	100
20	$^{13}C_{12}$-1,2,3,7,8-PeCDD	100	100	100	100	100
21	$^{13}C_{12}$-1,2,3,7,8-PeCDF	100	100	100	100	100
	$^{13}C_{12}$-2,3,4,7,8-PeCDF	100	100	100	100	100
	$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100	100	100	100	100
22	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	100	100	100	100	100
23	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100	100	100	100	100
	$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100	100	100	100	100
	$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	100	100	100	100	100
	$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	100	100	100	100	100
24	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100	100	100	100	100
25	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100	100	100	100	100
	$^{13}C_{12}$-1,2,3,4,7,8,9-	100	100	100	100	100

	Compound	Concentration (ng/mL)				
RRF (n)(m)		**CS2**	**CS3**	**CS4 (ICV(6))**	**CS5**	**CS6**
	HpCDF					
26	$^{13}C_{12}$-OCDD	200	200	200	200	200
	Cleanup Standard/ FS					
	$^{37}Cl_4$-2,3,7,8-TCDD	0.5	2	10	40	200
	Internal Standards					
	$^{13}C_{12}$-1,2,3,4-TCDD	100	100	100	100	100
	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	100	100	100	100	100

TABLE 6*
Elemental Compositions and Exact Masses of the Ions
Monitored by HR/MS for PCDDs and PCDFs

Descriptor	Exact m/z [1]	m/z Type	Elemental Composition	Substance [2]
1	292.9825	QC	C_7F_{11}	PFK
	303.9016	M	$C_{12}H_4{}^{35}Cl_4O$	TCDF
	305.8987	M+2	$C_{12}H_4{}^{35}Cl_3{}^{37}ClO$	TCDF
	315.9419	M	$^{13}C_{12}H_4{}^{35}Cl_4O$	TCDF [3]
	317.9389	M+2	$^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO$	TCDF [3]
	319.8965	M	$C_{12}H_4{}^{35}Cl_4O_2$	TCDD
	321.8936	M+2	$C_{12}H_4{}^{35}Cl_3{}^{37}ClO_2$	TCDD
	327.8847	M	$C_{12}H_4{}^{37}Cl_4O_2$	TCDD [4]
	330.9792	Lock	C_7F_{13}	PFK
	331.9368	M	$^{13}C_{12}H_4{}^{35}Cl_4O_2$	TCDD [3]
	333.9339	M+2	$^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO_2$	TCDD [3]
	339.8597	M+2	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF
	341.8567	M+4	$C_{12}H_3{}^{35}Cl_3{}^{37}ClO$	PeCDF
	375.8364	M+2	$C_{12}H_4{}^{35}Cl_5{}^{37}ClO$	HxCDPE
	409.7974	M+2	$C_{12}H_3{}^{35}Cl_6{}^{37}ClO$	HpCDPE
2	330.9792	QC	C_7F_{13}	PFK
	339.8597	M+2	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF
	341.8567	M+4	$C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$	PeCDF
	342.9792	Lock	C_8F_{12}	PFK
	351.9000	M+2	$^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF
	353.8970	M+4	$^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF [3]
	354.9792	Lock	C_9F_{13}	PFK
	355.8546	M+2	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$	PeCDD
	357.8516	M+4	$C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$	PeCDD
	366.9793	QC	C_9F_{13}	PFK
	367.8949	M+2	$^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$	PeCDD [3]
	369.8919	M+4	$^{13}C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$	PeCDD [3]
	409.7974	M+2	$C_{12}H_3{}^{35}Cl_6{}^{37}ClO$	HpCDPE
3	373.8208	M+2	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO$	HxCDF
	375.8178	M+4	$C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O$	HxCDF
	380.9760	Lock	C_8F_{15}	PFK
	383.8639	M	$^{13}C_{12}H_2{}^{35}Cl_6O$	HxCDF [3]
	385.8610	M+2	$^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO$	HxCDF [3]
	389.8157	M+2	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$	HxCDD
	391.8127	M+4	$C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$	HxCDD
	392.9760	Lock	C_9F_{15}	PFK
	401.8559	M+2	$^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$	HxCDD [3]
	403.8529	M+4	$^{13}C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$	HxCDD [3]
	430.9728	QC	C_9F_{17}	PFK
	445.7550	M+4	$C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2O$	OCDPE
4	392.9760	QC	C_9F_{15}	PFK
	407.7818	M+2	$C_{12}H^{35}Cl_6{}^{37}ClO$	HpCDF
	409.7789	M+4	$C_{12}H^{35}Cl_5{}^{37}Cl_2O$	HpCDF

Descriptor	Exact m/z [1]	m/z Type	Elemental Composition	Substance [2]
	417.8253	M	$^{13}C_{12}H^{35}Cl_7O$	HpCDF [3]
	419.8220	M+2	$^{13}C_{12}H^{35}Cl_6{}^{37}ClO$	HpCDF [3]
	423.7766	M+2	$C_{12}H^{35}Cl_6{}^{37}ClO_2$	HpCDD
	425.7737	M+4	$C_{12}H^{35}Cl_5{}^{37}Cl_2O_2$	HpCDD
	430.9729	Lock	C_9F_{17}	PFK
	435.8169	M+2	$^{13}C_{12}H^{35}Cl_6{}^{37}ClO_2$	HpCDD [3]
	437.8140	M+4	$^{13}C_{12}H^{35}Cl_5{}^{37}CL_2O_2$	HpCDD [3]
	479.7165	M+4	$C_{12}H^{35}Cl_7{}^{37}Cl_2O$	NCDPE
5	392.9760	QC	C_9F_{15}	PFK
	441.7428	M+2	$C_{12}{}^{35}Cl_7{}^{37}ClO$	OCDF
	442.9728	Lock	$C_{10}F_{17}$	PFK
	443.7399	M+4	$C_{12}{}^{35}Cl_6{}^{37}Cl_2O$	OCDF
	457.7377	M+2	$C_{12}{}^{35}Cl_7{}^{37}ClO_2$	OCDD
	459.7348	M+4	$C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$	OCDD
	469.7779	M+2	$^{13}C_{12}{}^{35}Cl_7{}^{37}ClO_2$	OCDD [3]
	471.7750	M+4	$^{13}C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$	OCDD [3]
	479.7165	M+4	$C_{12}Cl_8{}^{37}Cl_2O$	NCDPE
	513.6775	M+4	$^{13}C_{12}{}^{35}Cl_8{}^{37}Cl_2O$	DCDPE

---

[a] The following nuclidic masses were used:

H =	1.007825	O =	15.994915
C =	12.000000	^{35}Cl =	34.968853
^{13}C =	13.003355	^{37}Cl =	36.965903
F =	18.9984		

S = Isotope dilution analyte/internal standard

*The homologous groups for functions 1-3 do not use the same lockmass as described in Table 6. They use masses 316.9824, 366.9792, and 380.9760, respectively.

TABLE 7

Recommended GC Operating Conditions

The GC Operating Conditions (Temperatures (°C), and Times (minutes)) Are as Follows:

Injector Temperature: 280°C
Interface Temperature: 280°C
Initial Temperature and Time: 190°C / 1 Minute

Temperature Program: 190°C, increasing at a rate of 4°C per minute up to 240°C, and maintaining at this temperature until the last tetra of the tetra- group has eluted from the column. (The total time required for this is approximately 25 minutes, depending on the length of the column). The maintained temperature of 240°C is then increased to 320°C at the rate of 20°C per minute and held at this level until the last compound (octa-group) has eluted from the column.

TABLE 8

PCDD and PCDF Congeners Present in the GC Performance Evaluation Solution and Used for Defining the Homologous GC Retention Time Windows on a 60-M DB-5 Column[(b)]

# of Chlorine Atoms	PCDD Positional Isomer		PCDF Positional Isomer	
	Early Eluter	Late Eluter	Early Eluter	Late Eluter
4[(a)]	1,3,6,8	1,2,8,9	1,3,6,8	1,2,8,9
5	1,2,4,6,8/1,2,4,7,9	1,2,3,8,9	1,3,4,6,8	1,2,3,8,9
6	1,2,4,6,7.9	1,2,3,4,6,7	1,2,3,4,6,8	1,2,3,4,8,9
7	1,2,3,4,6,7,9	1,2,3,4,6,7,8	1,2,3,4,6,7,8	1,2,3,4,6,7,8,9
8	1,2,3,4,6,7,8,9		1,2,3,4,6,7,8,9	

[(a)] In addition to these two PCDD isomers, the 1,2,3,4-, 1,2,3,7-, 1,2,3,8-, 2,3,7,8-, $^{13}C_{12}$-2,3,7,8-, and 1,2,3,9-TCDD isomers must also be present.

(b) The PCDF Congeners present in GC the Performance Evaluation Solution for the 30 m DB-225 column include:

- 1,2,3,9-TCDF
- 2,3,7,8-TCDF
- 2,3,4,7-TCDF
- $^{13}C_{12}$-2,3,7,8-TCDF

Column performance criteria is met when the percent valleys between the 2,3,7,8-TCDF analyte and the closest eluting isomers are $\leq$ 25%.

TABLE 9

Theoretical Ion Abundance Ratios and Their Control Limits for PCDDs and PCDFs

# of Chlorine Atoms	Ion Type	Theoretical Ratio	Control Limits	
			Lower	Upper
4	M / M+2	0.77	0.65	0.89
5	M+2 / M+4	1.55	1.32	1.78
6	M+2 / M+4	1.24	1.05	1.43
6[(a)]	M / M+2	0.51	0.43	0.59
7[(b)]	M / M+2	0.44	0.37	0.51
7	M+2 / M+4	1.04	0.88	1.20
8	M+2 / M+4	0.89	0.76	1.02

(a) Used only for ^{13}C-HxCDF (IS) (b) Used only for ^{13}C-HpCDF (IS)

TABLE 10

2,3,7,8-TCDD Equivalent Factors (TEFs) for the Polychlorinated Dibenzodioxins and Dibenzofurans

Number	Compound(s)	TEF
1	2,3,7,8-TCDD	1.00
2	1,2,3,7,8-PeCDD	0.50
3	1,2,3,6,7,8-HxCDD	0.10
4	1,2,3,7,8,9-HxCDD	0.10
5	1,2,3,4,7,8-HxCDD	0.10
6	1,2,3,4,6,7,8-HpCDD	0.01
7	OCDD	0.001
8	2,3,6,7-TCDF	0.1
9	1,2,3,7,8-PeCDF	0.05
10	2,3,4,7,8PeCDF	0.5
11	1,2,3,6,7,8-HxCDF	0.1
12	1,2,3,7,8,9-HxCDF	0.1
13	1,2,3,4,7,8-HxCDF	0.1
14	2,3,4,6,7,8-HxCDF	0.1
15	1,2,3,4,6,7,8-HpCDF	0.01
16	1,2,3,4,7,8,9-HpCDF	0.01
17	OCDF	0.001

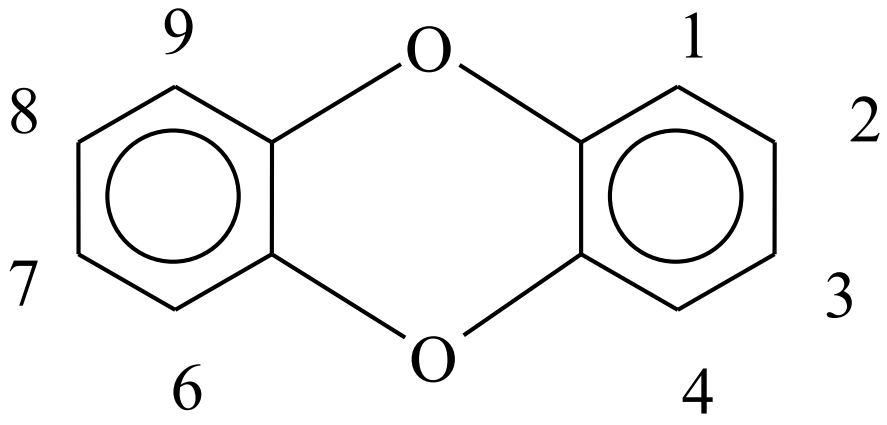
TABLE 11

Toxicity Equivalency Factor:
Analyte Relative Retention Time Reference Attributes

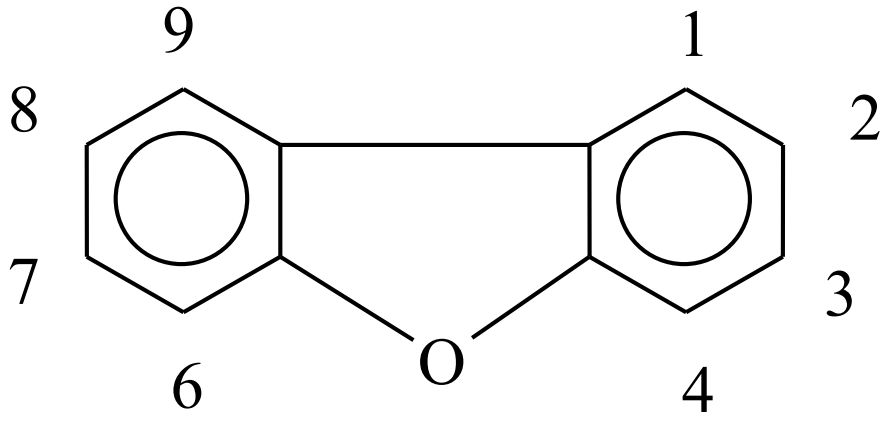
Analyte	Analyte RRT Reference (a)
1,2,3,4,7,8-HxCDD	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD
1,2,3,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF
1,2,3,7,8,9-HxCDF	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF
2,3,4,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF

(a) The retention time of 2,3,4,7,8-PeCDF on the DB-5 column is measured relative to $^{13}C_{12}$-1,3,7,8-PeCDF and the retention time of 1,2,3,4,7,8,9-HpCDF relative to $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF

FIGURE 1
Structure of Dibenzodioxin and Dibenzofuran

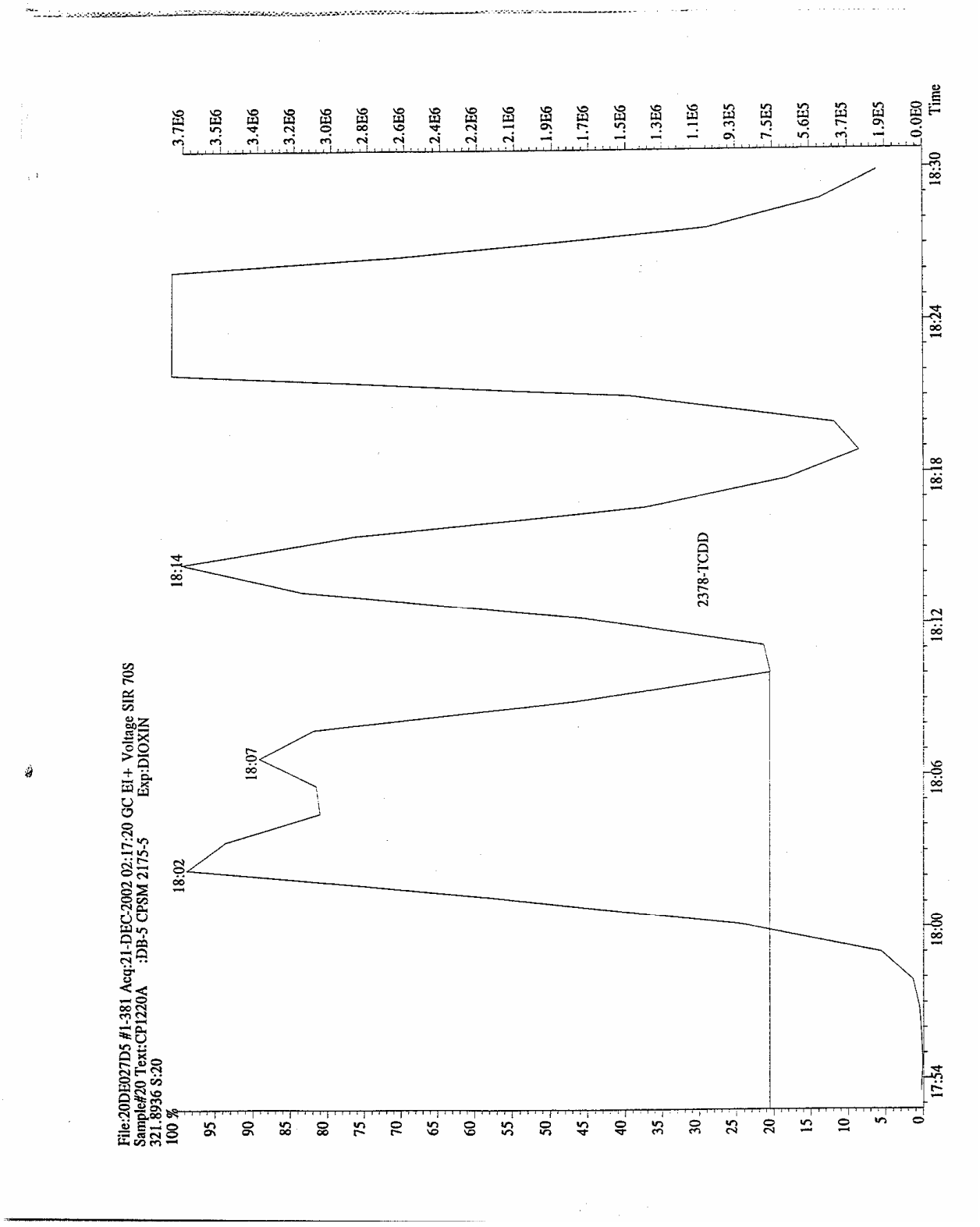


Dibenzodioxin



Dibenzofuran

FIGURE 2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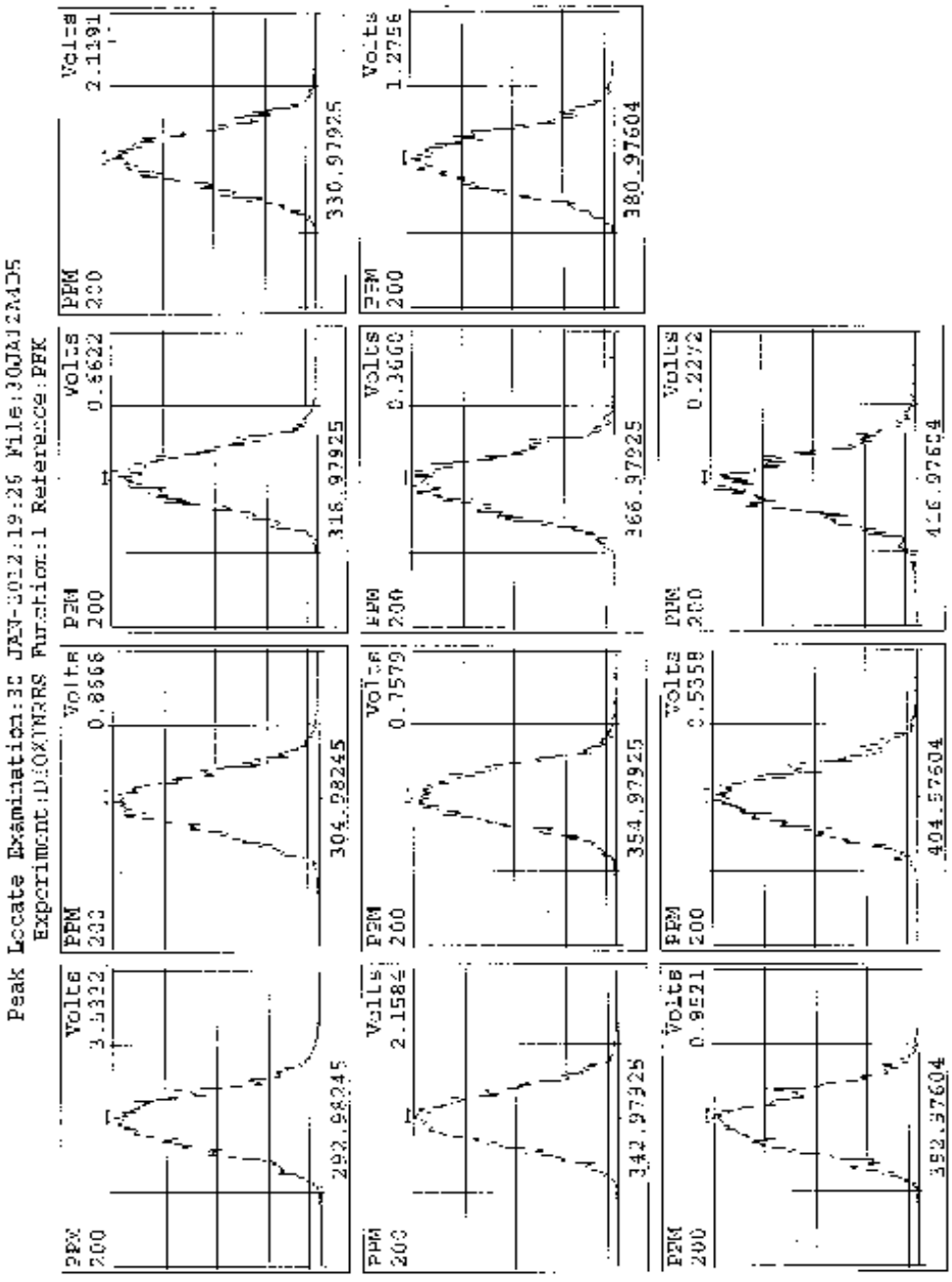
Sample#20 Text:CP1220A :DB-5 CPSM 2175-5 Exp:DIOXIN
321.8936 S:20
100 %
18:02
18:07
18:14
2378-TCDD
Time
17:54
18:00
18:06
18:12
18:18
18:24
18:30
0.0E0
1.9E5
3.7E5
5.6E5
7.5E5
9.3E5
1.1E6
1.3E6
1.5E6
1.7E6
1.9E6
2.1E6
2.2E6
2.4E6
2.6E6
2.8E6
3.0E6
3.2E6
3.4E6
3.5E6
3.7E6

Figure 3


Peak Locate Examination:3S JAN-2012:19:25 File:30JAJ2A35
Experiment:D:\OXINSRS Function:1 Reference:PFK
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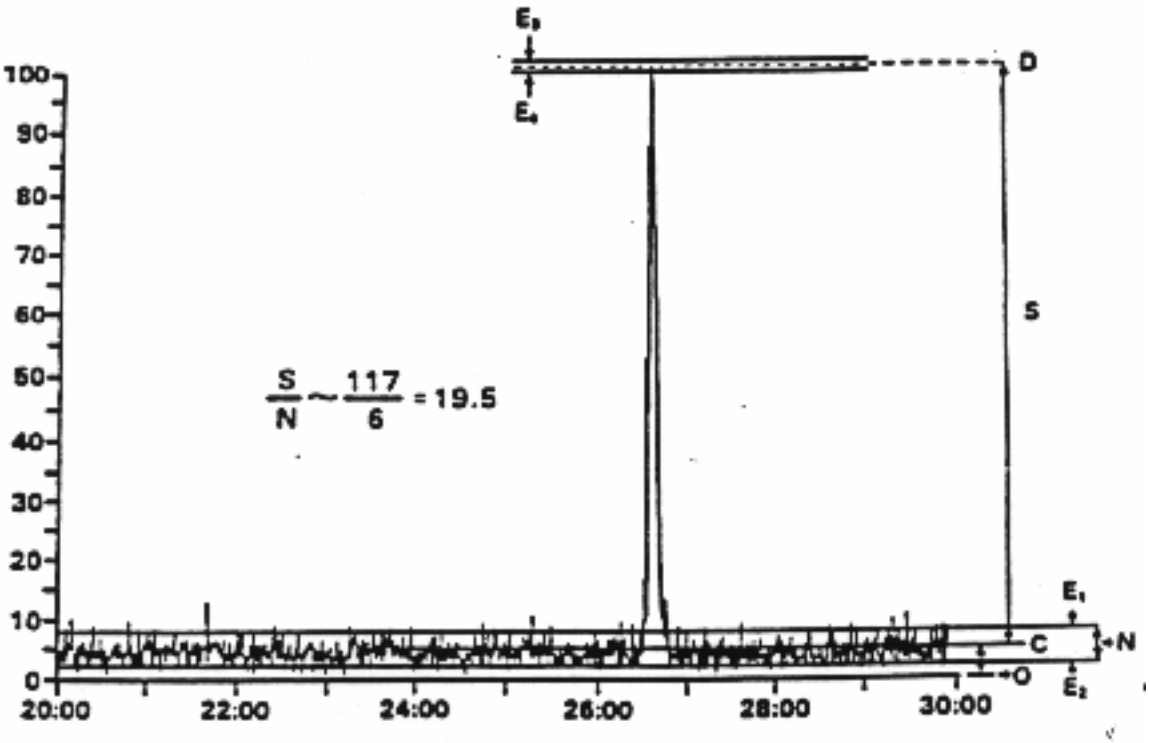
200
Volts
3.5332
292.98245
PPM
200
Volts
0.8668
304.98245
PPM
200
Volts
0.9622
316.97925
PPM
200
Volts
2.1191
330.97925
PPM
200
Volts
2.1582
342.97925
PPM
200
Volts
0.7579
354.97925
PPM
200
Volts
0.3660
366.97925
PPM
200
Volts
1.2756
380.97604
PPM
200
Volts
0.9521
392.97604
PPM
200
Volts
0.5358
404.97604
PPM
200
Volts
0.2272
416.97604

FIGURE 4



Manual determination of S/N.

The peak height (S) is measured between the mean noise (lines C and D). These mean signal values are obtained by tracing the line between the baseline average noise extremes, E1 and E2, and between the apex average noise extremes, E3 and E4, at the apex of the signal.

NOTE: It is imperative that the instrument interface amplifier electronic zero offset be set high enough so that negative going baseline noise is recorded.

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November 1992

APPENDIX A

This procedure is designed for the periodic evaluation of potential contamination by 2,3,7,8-substituted PCDD/PCDF congeners of the working areas inside the laboratory.

PERFORMING WIPE TEST

Perform the wipe tests on surface areas of two inches by one foot with laboratory wipers saturated with distilled-in-glass acetone or appropriate solvent using a pair of clean stainless steel forceps. Use one wiper for each of the designated areas. Combine the wipers to one composite sample in an extraction jar containing 200 mL distilled-in-glass hexane. Place an equal number of unused wipers in 200 mL hexane and use this as a control.

SAMPLE PREPARATION

Close the jar containing the wipes and 200 mL hexane and extract for 20 minutes using a wrist-action shaker. Use an appropriate means to reduce the volume to approximately 1.0 mL. Put through an alumina column to clean up potential interfering compounds. Add appropriate amount of internal standard.

EXTRACT ANALYSIS

Concentrate the contents of the vial to a final volume of 20 µL (either in a minivial or in a capillary tube). Inject 2 µL of each extract (wipe and control) onto a capillary column and analyze for 2,3,7,8-substituted PCDDs/PCDFs as specified in the analytical method Section 11 (this exhibit). Perform calculations according to Section 12 (this exhibit).

REPORTING FORMAT

Report the presence of 2,3,7,8-substituted PCDDs and PCDFs as a quantity (pg or ng) per wipe test experiment (WTE). Under the conditions outlined in this analytical protocol, a lower limit of calibration of 25 pg/WTE is expected for 2,3,7,8-TCDD. A positive response for the blank (control) is defined as a signal in the TCDD retention time window at any of the masses monitored which is equivalent to or above 8 pg of 2,3,7,8-TCDD per WTE. For other congeners, use the multiplication factors listed in Table 1, footnote (a) (e.g., for OCDD, the lower MCL is 25 x 5 = 125 pg/WTE and the positive response for the blank would be 8 x 5 = 40 pg). Also, report the recoveries of the isotope dilution analytes during the simplified cleanup procedure.

FREQUENCY OF WIPE TESTS

Wipe tests should be performed when there is evidence of contamination in the method blanks.

CORRECTIVE ACTION

An upper limit of 25 pg per TCDD isomer and per wipe test experiment is allowed. (Use multiplication factors listed in footnote (a) from Table 1 for other congeners.) This value corresponds to the lower calibration limit of the analytical method. Steps to correct the contamination must be taken whenever these levels are exceeded. To that effect, first vacuum the working places (hoods, benches, sink) using a vacuum cleaner equipped with a high-efficiency particulate absorbent (HEPA) filter and then wash with a detergent. A new set of wipes should be analyzed before anyone is allowed to work in the dioxin area of the laboratory.

The test results and the decontamination procedure must be reviewed with EH&S.

Title: Analysis of Tetra- through Octa Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS by Method 1613B [Method 1613B]

Approvals (Signature/Date):

Robert Hrabak — 6/12/17
Department Manager — Date

Joe Schairer — 6/13/2017
Health & Safety Manager / Coordinator — Date

Lisa Stafford — 6/13/17
Quality Assurance Manager — Date

Crystal Pollock — 6.14.17
Laboratory Director — Date

1. SCOPE AND APPLICATION

1.1. The procedures outlined within this SOP are appropriate for the analysis of samples and the determination of tetra-through-octa chlorinated dibenzo-p-dioxins and dibenzofurans, and 2,3,7,8-TCDD/TCDF associated with the Clean Water Act (CWA – as amended 1987); the Resource Conservation and Recovery Act (RCRA - as amended 1986); the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA - as amended 1986), and the EPA Methods 1613 rev A (1990) and 1613 rev B (1994) Preparation of samples is addressed in SOP WS-IDP-0007.

1.2. Specificity is provided for determination of the seventeen 2, 3, 7, 8-substituted polychlorinated dibenzo-p-dioxins (PCDD) and polychlorinated dibenzofurans (PCDF). See Table 8 for list of analytes and nominal reporting limits.

1.3. When undertaking projects for Department of Defense (DOD) or the Department of Energy (DOE), the relevant criteria in QA Policy WS-PQA-021 must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. Samples containing coarse solids are prepared for extraction by grinding or homogenization. Drinking water samples are extracted with methylene chloride using separatory funnel procedures. Non drinking water type samples are filtered and then extracted with methylene chloride using separatory funnel procedures; the particulates from the water samples, soils, and other finely divided solids are extracted using a combined Soxhlet (or equivalent) extraction/Dean-Stark azeotropic distillation with toluene (Reference 7, Method 1613).

2.1.1. Internal Standards consisting of stable isotopically labeled analogs of 15 of the PCDDs and PCDFs and are added to each sample prior to extraction. Prior to cleanup and analysis, the extracts of the filtered water and the particulates are combined. (Note: Internal standards are named “Isotope Dilution Analytes” or IDA throughout this SOP.)

2.2. After extraction, $^{37}Cl_4$-labeled 2, 3, 7, 8-TCDD is added to each extract to measure the efficiency of the cleanup process. Samples are then permeated through silica gel, acidic alumina, and activated carbon chromatography columns. High performance liquid chromatography (HPLC), basic alumina, or back extraction with sulfuric acid/water/NaOH/water can be used for further isolation of the 2, 3, 7, 8-isomers or other specific isomers or congeners.

2.3. After cleanup, the extract is concentrated to near dryness and a known amount of recovery standard is added to each extract. The recovery standard contains two $^{13}C_{12}$-labeled analogs ($^{13}C_{12}$-1,2,3,4-TCDD and $^{13}C_{12}$-1,2,3,7,8,9-HxCDD). Each extract is concentrated to a final volume in tetradecane. A 1-2uL aliquot of the extract is injected

into the gas chromatograph/mass spectrometer. (Note: Recovery standards are called "Internal Standards" or IS throughout the rest of this SOP.)

2.4. The analytes are separated by the GC and detected by a high resolution ($\geq$10,000) mass spectrometer. Two exact masses (m/z's) are monitored for each analyte. The isotopically labeled compounds serve to correct for the variability of the analytical technique.

2.5. Dioxins and furans are identified by comparing GC retention times and the ion abundance ratios of the m/z's with the corresponding retention time ranges of authentic standards and the theoretical ion abundance ratios of the exact m/z's. Isomers and congeners are identified when the retention times and m/z abundance ratios agree within pre-defined limits. This is accomplished for the specific 2,3,7,8 substituted isomers by using a GC column or columns capable of resolving the 2,3,7,8-substituted isomers from other PCDD/PCDF isomers.

2.6. Quantitative analysis is performed by GC/MS using selected ion current profile (SICP) areas, in one of two ways.

2.6.1. For the fifteen 2,3,7,8-substituted isomers for which labeled analogs are available (see Table 3), the GC/MS system is calibrated and the compound concentration is determined using an isotope dilution technique. Although a labeled analog of the octa-chlorinated dibenzofuran (OCDF) is available, using high-resolution mass spectrometry, it produces an m/z that may interfere with the identification and quantitation of the unlabeled octa-chlorinated dibenzo-p-dioxin (OCDD). Therefore, this labeled analog has not been included in the calibration standards, and the unlabeled OCDF is quantitated against the labeled OCDD. Because the labeled analog of 1,2,3,7,8,9-HxCDD is used as an internal standard (i.e., not added before extraction of the sample), it cannot be used to quantitate the unlabeled compound by strict isotope dilution procedures. Therefore, the unlabeled 1,2,3,7,8,9-HxCDD is quantitated using the average of the responses of the labeled analogs of the two 2,3,7,8-substituted HxCDD's, $^{13}C_{12}$-1,2,3,4,7,8-HxCDD, and $^{13}C_{12}$-1,2,3,6,7,8-HxCDD. As a result, the concentration of the unlabeled 1,2,3,7,8,9-HxCDD is quantitated using the average recovery of the two $^{13}C_{12}$-HxCDD internal standards.

2.6.2. For non-2,3,7,8-substituted isomers, the total concentrations of all isomers within a level of chlorination (i.e., total TCDD), are determined using the average response factor from the calibration of 2,3,7,8-substituted isomers at the same level of chlorination.

2.7. The quality of the analysis is assured through reproducible calibration and testing of the extraction, cleanup, and GC/MS systems.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs): compounds (Figure 1) that contain from one to eight chlorine atoms. The seventeen 2,3,7,8-substituted PCDDs and PCDFs are shown in Table 3. The number of isomers at different chlorination levels is shown in Table 1.

3.3. Homologous series: A series of compounds in which each member differs from the next by a constant amount. The members of one level in the series are called homologs – a group of chlorinated dibenzodioxins or dibenzofurans having a specific number of chlorine atoms.

3.4. Isomer: Chemical compounds that contain the same number of atoms of the same elements, but differ in structural arrangement and properties. For example, 1,2,3,4-TCDD and 2,3,7,8-TCDD are different structural isomers.

3.5. Congener: Any isomer of any homologous series.

3.6. Internal Dilution Analyte (IDA): An IDA is a $^{13}C_{12}$-labeled analog of a congener chosen from the compounds listed in Table 3. IDA's are added to all samples including method blanks and quality control samples before extraction, and they are used to quantitate the concentration of the target analytes. Fifteen IDA's are used in this method. There is one for each of the 2,3,7,8-substituted dioxin and furan isomers (except for OCDF and 1,2,3,7,8,9-HxCDD see section 2.6.1) with the degree of chlorination ranging from four to eight. Additional IDA's may be added to act as retention time references, but they are not used for quantitation.

3.7. Isotope Dilution Analyte (IDA) Solution: A solution (isooctane or toluene) containing the fifteen IDA, which is used to spike all samples prior to extraction.

3.8. Cleanup Recovery/Surrogate Standard (SU): A $^{37}Cl_4$-2,3,7,8-TCDD analog that is added to each sample following extraction to measure the efficiency of the cleanup process.

3.9. Cleanup Recovery/Surrogate Standard Solution: A solution (isooctane or toluene) containing the cleanup recovery standard.

3.10. Internal Standard (IS): Two internal standards are used to determine the percent recoveries for the internal standards. The ^{13}C-1,2,3,4-TCDD is used to measure the percent recoveries of the tetra- and pentachlorinated internal standards while ^{13}C-1,2,3,7,8,9-HxCDD is used to determine the recovery of the hexa-hepta- and octachlorinated internal standards . ^{13}C-1,2,3,7,8,9-HxCDD also acts as a retention

time reference for the unlabeled analog present in sample extracts. They are added to the final sample extract before HRGC/HRMS instrument analysis.

3.11. Internal Standard Solution: A solution (tetradecane, toluene, or isooctane) containing the two recovery standards, which is added to the final sample extract before the HRGC/HRMS analysis.

3.12. DB-5 Column Performance Standard Mixture (DB5 CPSM)(See Table 6): A tetradecane solution containing a mixture of selected PCDD/PCDF standards including the first and last eluting compounds from each homologous series (a window defining mixture), which is used to demonstrate continued acceptable performance of the DB-5 capillary column (i.e., ≤25% valley separation of 2,3,7,8-TCDD from other TCDD isomers) and to define the homologous PCDD/PCDF retention time windows.

3.13. (NOTE: The CPSM contains the second to the last eluting HxCDD isomer [1,2,3,4,6,7-HxCDD], however, the IDA Solution [present in all calibration standards and samples] does contain the last eluting HxCDD [1,2,3,7,8,9-HxCDD])

3.14. DB-225 Column Performance Standard Mixture (DB-225 CPSM)(See Table 6): A tetradecane solution containing a mixture of selected TCDF isomers used to demonstrate continued acceptable performance of the DB-225 capillary column (i.e., ≤25% valley separation of 2,3,7,8-TCDF from other TCDF isomers).

3.15. Signal to Noise Ratio (S/N): The ratio of the mass spectrometer response of a GC peak to the background noise signal.

3.16. Tuning (Mass Resolution Check): Standard method used to demonstrate a static resolving power of 10,000 minimum (10 percent valley definition).

3.17. Method Calibration Limits: For a given sample size, a final extract volume, and the lowest and highest concentration calibration solutions, the lower and upper calibration limits delineate the region of quantification for which the HRGC/HRMS system was calibrated with standard.

3.18. Matrix Spike Fortification Solution (also known as the Native Spike Solution): Solution used to prepare the laboratory control sample (LCS), matrix spike (MS), and matrix spike duplicate (SD) samples. It contains all unlabeled analytes listed in Table 3.

3.19. PFK: Perfluorokerosene; used to calibrate the exact m/z scale in the HRMS.

3.20. Estimated Detection Limit (EDL): The sample specific estimated detection limit (EDL) is the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background noise level.

3.21. Estimated Maximum Possible Concentration (EMPC): The calculated concentration of a signal having the same retention time as a PCDD/PCDF congener, but which does not meet the other qualitative identification criteria defined in the method.

3.22. Lower Calibration Limit (LCL): The level at which the entire analytical system must give a recognizable signal and acceptable calibration. It is equivalent to the concentration of the lowest calibration standard, assuming that all method-specified sample weights, volumes, and cleanup procedures have been employed.

4. INTERFERENCES

4.1. Solvents, reagents, glassware and other sample processing hardware may yield artifacts and/or elevated baselines causing misinterpretation of chromatograms. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required. Where possible, reagents are cleaned by extraction or solvent rinse.

4.2. Proper cleaning of glassware is extremely important because glassware may not only contaminate the samples, but may also remove the analytes of interest by absorption on the glassware surface.

4.2.1. Glassware is rinsed with solvent and washed with a detergent solution as soon after use as practical. Sonication of glassware containing a detergent solution for approximately 30 seconds may aid in cleaning. Glassware with removable parts, particularly separatory funnels with Teflon stopcocks, must be disassembled prior to detergent washing.

4.2.2. After detergent washing, glassware is immediately rinsed with tap water or DI water, then with acetone, then with toluene. The toluene rinse is followed by hexane, and then methylene chloride.

4.2.3. Do not bake reusable glassware in an oven as a routine part of cleaning. Baking may be warranted after particularly dirty samples are encountered, but should be minimized, as repeated baking of glassware may cause active sites on the glass surface that will irreversibly absorb PCDDs/PCDFs.

4.2.4. Immediately prior to use, Soxhlet (or equivalent) extraction glassware is pre-extracted with toluene for approximately 4 hours (1 hour for Soxtherm extraction glassware) (See Method 1613A, Section 11.2.3). Separatory funnels are rinsed sequentially with acetone, toluene, hexane and methylene chloride.

4.3. All materials used in the analysis shall be demonstrated to be free from interferences by running reference blanks initially and with each sample set (samples started through the extraction process on a given 12-hour shift, to a maximum of 20 samples).

4.3.1. The reference matrix blank must simulate, as closely as possible, the sample matrix under test. Reagent water is used to simulate water samples; playground sand, white quartz sand, or sodium sulfate can be used to simulate soils; filter paper or Soxhlet extraction thimbles are used to simulate paper and similar materials; and other materials can be used to simulate other matrices.

4.4. Interferences co-extracted from samples will vary considerably from source to source, depending on the diversity of the site being sampled. Interfering compounds may be present at concentrations several orders of magnitude higher than the PCDDs and PCDFs. The most frequently encountered interferences are: chlorinated-biphenyls; methoxy biphenyls; hydroxy biphenyl ethers; benzyl phenyl ethers; polynuclear aromatics; and pesticides. Because very low levels of PCDDs and PCDFs are measured by this method, the elimination of interferences is essential. The cleanup steps given in WS-IDP-0007 can be used to reduce or eliminate these interferences and thereby permit reliable determination of the PCDDs and PCDFs at the levels shown in Table 8.

4.4.1. If South Carolina samples show diphenyl ethers at levels that could contribute to positive furan hits, a clean-up to remove them must be performed.

5. 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 reponsibility 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, nonabsorbant shoes are a minimum. All work must be stopped in the event of a known or potential compromise to the health or safety of an associate. The situation must be reported immediately to a supervisor, the EH&S Staff, or a senior manager.

5.1. Specific Safety Concerns or Requirements

5.1.1. The effluents of sample splitters for the gas chromatograph and roughing pumps on the HRGC/HRMS system are passed through either a column of activated charcoal or be bubbled through a trap containing oil or high-boiling alcohol.

5.1.2. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex and vinyl gloves provide no protection against most of the organic solvents used in this method. Nitrile or similar gloves must be used. Latex gloves may be used for methanol.

5.1.3. Exposure to chemicals must be maintained as low as reasonably achievable;

therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.4. Laboratory procedures such as repetitive use of pipettes, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

5.2.1. The following is a list of the materials used in this method, which have a serious or significant hazard rating.

NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Tetradecane	Irritant	None established	Inhalation of vapors may cause difficulty breathing, headache, intoxication and central nervous system damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Preventive and routine maintenance is described in Section 11.7 of this SOP.

6.2. High-Resolution Gas Chromatograph/High-Resolution Mass Spectrometer/Data System (HRGC/HRMS/DS).

6.2.1. Gas Chromatograph: HP 6890 GC, or equivalent.

6.2.1.1. The GC must be equipped for temperature programming.

6.2.1.2. All required accessories must be available, such as syringes, gases, and capillary columns.

6.2.1.3. The GC injection port must be designed for capillary columns.

- The use of splitless injection techniques is recommended. The use of a moving needle injection port is also acceptable.

6.2.1.4. When using the method described in this protocol, a 2-µL injection volume is used consistently (i.e., the injection volumes for all extracts, blanks, calibration solutions and the performance check samples are 2 µL).

- 1 µL injections are allowed; however, laboratories are encouraged to remain consistent throughout the analyses by using the same injection volume at all times on a given HRGC/HRMS/DS.

6.2.2. Gas Chromatograph/Mass Spectrometer (GC/MS) Interface

6.2.2.1. The GC/MS interface components should withstand 350° C.

6.2.2.2. The interface must be designed so that the separation of 2,3,7,8-TCDD from the other TCDD isomers achieved in the gas chromatographic column is not appreciably degraded.

6.2.2.3. Cold spots or active surfaces (adsorption sites) in the GC/MS interface can cause peak tailing and peak broadening.

6.2.2.4. It is recommended that the GC column be fitted directly into the mass spectrometer ion source without being exposed to the ionizing electron beam.

6.2.2.5. Graphite ferrules should be avoided in the injection port because they may adsorb the PCDDs and PCDFs. Vespel® or equivalent ferrules are recommended.

6.2.3. Mass Spectrometer

6.2.3.1. The static resolving power of the instrument must be maintained at a minimum of 10,000 (10 percent valley).

6.2.3.2. The mass spectrometer must be operated in a selected ion monitoring (SIM) mode with a total cycle time (including the voltage reset time) of one second or less.

6.2.4. Data System

6.2.4.1. A dedicated data system is employed to control the rapid multiple ion monitoring process and to acquire the data.

6.2.4.2. Quantification data (peak areas or peak heights) and SIM traces (displays of intensities of each ion signal being monitored including the lock-mass ion as a function of time) must be acquired during the analyses and stored.

6.2.4.3. Quantifications may be reported based upon computer-generated peak areas or upon measured peak heights (chart recording).

6.2.4.4. The data system must be capable of acquiring data for a minimum of 10 ions in a single scan.

6.2.4.5. The data system is capable of switching to different sets of ions (descriptors) at specified times during an HRGC/HRMS acquisition.

6.2.4.6. The data system is able to provide hard copies of individual ion chromatograms for selected gas chromatographic time intervals.

6.2.4.7. The data system is also able to acquire mass-spectral peak profiles and provide hard copies of peak profiles to demonstrate the required resolving power.

6.2.4.8. The data system permits the measurement of noise on the base line.

6.2.4.9. Capable of collecting, recording and storing MS data. The VG70 and Autospec Ultima systems utilize Opus version 3.6 software and the Autospec Premiere system utilizes MassLynx version 4.1 software for collecting the data, and Chrom Version 2.2x to process the data.

6.3. GC Column

6.3.1. In order to have an isomer-specific determination for 2,3,7,8-TCDD and to allow the detection of OCDD/OCDF within a reasonable time interval in one HRGC/HRMS analysis, the 60-m DB-5 fused-silica capillary column is recommended.

6.3.2. At the beginning of each 12-hour period during which samples are analyzed and after tuning, acceptable compound separation on the GC column must be demonstrated through the analysis of a column performance check solution.

6.4 Data Acquisition, Processing, and Reporting

6.4.1 Instrument acquisition uses either OpusQuan v 3.6 or MassLynx v 4.1

6.4.2 Data processing uses CHROM rev 2.2

6.4.3 Data reporting uses TALS

7. REAGENTS AND STANDARDS

7.1. Solvents and Standards:

7.1.1. High-purity, distilled-in-glass or highest available purity: Tetradecane.

7.1.2. Standards are purchased as solutions or mixtures with certification of their purity, concentration, and authenticity, or prepared from materials of known purity and composition.

7.1.2.1. If the chemical purity is 98% or greater, the weight may be used without correction to compute the concentration of the standard.

7.1.2.2. When not being used, standards can be stored in the dark at room temperature or refrigerated in screw-capped vials with fluoropolymer-lined or Teflon ® - lined caps.

7.1.2.3. A mark is placed on the vial at the level of the solution so that solvent loss by evaporation can be detected. If solvent loss has occurred, the solution should be replaced.

7.1.3. Sealed ampules may be used until the manufacturer's expiration date is exceeded.

7.1.3.1. If no expiration date is provided, then the expiration date will be 10 years from the date the ampule is opened.

7.1.3.2. The solvent level should be monitored prior to each use to assure there has been no concentration of the standard over time.

7.1.4. All calibration, daily isotope dilution analytes, daily internal standards, surrogates, and daily native analyte spiking solutions are stable for one year from preparation.

7.1.4.1. After 1 year, solutions may be re-verified. The re-verified solution may be used for an additional year, or until there is evidence of compound degradation or concentration.

7.1.4.2. The re-verification must be performed using an unexpired, not previously re-verified solution from a second lot or second vendor.

7.2. Stock Solutions

7.2.1. Preparation – Prepare in nonane or equivalent solvent per the steps below or

purchase as dilute solutions (Cambridge Isotope Laboratories, Cambridge, MA, or equivalent). Observe standard safety precautions.

7.2.2. Dissolve an appropriate amount of assayed reference material in solvent. For example, weigh 1 to 2mg of 2,3,7,8-TCDD to three significant numbers in a 10mL vial with Teflon ® - lined cap.

7.2.3. Stock standard solutions should be checked for signs of degradation prior to the preparation of calibration or performance test standards. Reference standards that can be used to determine the accuracy of calibration standards are available from Cambridge Isotope Laboratories and may be available from other vendors.

7.2.4. Secondary standard – Using stock solutions prepare secondary standard solutions if necessary which can be further diluted the spiking standard solutions (see below).

7.3. Precision and Recovery Standard (PAR) – Also known as the Matrix Spike or Native Spike solution. Contains all the 2,3,7,8-substituted unlabeled analytes listed in Table 4. Prepare using the appropriate standards made in section 7.5 to yield a spiking solution with a concentration of 4.0ng/ml for the tetra- CDDs/CDFs, 20ng/ml for the penta-, hexa-, and hepta- CDDs/CDFs, and 40ng/ml for the octa- CDD/CDF.

7.3.1. This is prepared by the dioxin Hi-Res department.

7.4. Sample Fortification Solution (Isotope Dilution Analyte) – Also known as the Daily Isotope Dilution Analyte. From stock solutions or purchased mixtures, prepare this solution in isooctane (or toluene) to contain the fifteen isotope dilution analytes at the nominal concentrations that are listed in Table 4.

7.4.1. This is prepared by the dioxin Hi-Res department.

7.5. Internal Standard Solution – This solution contains the two internal standards ($^{13}C_{12}$-1,2,3,4-TCDD and $^{13}C_{12}$-1,2,3,7,8,9-HxCDD). This solution is preferably prepared in tetradecane. See Table 4 for nominal concentrations.

7.6. Surrogate Standard Solution (CRS) – Prepare $^{37}Cl_4$-2,3,7,8-TCDD at the concentration shown in Table 4, in isooctane (or toluene).

7.6.1. This is prepared by the dioxin Hi-Res department.

7.7. Calibration Standards (L1 through L6)

7.7.1. High-Resolution Concentration Calibration Solutions (Table 5) – Combine the spiking solutions in sections 7.4 through 7.6 to produce the five tetradecane

calibration solutions listed in Table 5. These solutions permit the relative response to be measured as a function of concentration.

7.7.1.1. The L4 (midpoint) standard is routinely used for calibration verification (Note: any other calibration point within the lower or upper calibration solution may be used for calibration verification).

7.7.1.2. Store the calibration solutions in appropriate containers and at room temperature in the dark.

8. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1. The sample collection, shipping, handling, and chain-of-custody procedures are not described in this document. Sample collection personnel will, to the extent possible, homogenize samples in the field before filling the sample containers. This should minimize or eliminate the necessity for sample homogenization in the laboratory.

8.2. Samples are collected in amber glass containers following conventional sampling practices (Method 1613). Freely flowing aqueous samples are collected in refrigeration bottles using automatic sampling equipment. Solid samples are collected as grab samples using wide mouth jars.

8.3. Samples are stored at 0-4ºC in the dark from the time of collection until extraction (fish tissues must be stored at - 20°C).

8.3.1. A measurement of residual chlorine is performed using Standard Methods 4500-Cl B, C, D, E, F, or G. The residual chlorine will be checked at the laboratory for all aqueous samples. If residual chlorine is present in aqueous samples, the sampler or analyst adds 80mg of sodium thiosulfate per liter of water.

8.4. Sample extraction is performed within 365 days of sampling. Sample analysis is performed within forty (40) days of extraction or within 1 year if extract is frozen

9. QUALITY CONTROL

9.1. Batches are defined at the sample preparation step. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the QC program document (WS-PQA-0003) for further details of the batch definition.

9.1.1. The quality control batch is a set of up to 20 samples of the same matrix processed using the same procedure and reagents within the same time period. The quality control batch must contain a laboratory control sample (LCS) and a method blank (MB). Laboratory generated QC samples (MB, LCS, MS/MSD) do not count toward the maximum 20 samples in a batch. Field

QC samples are included in the batch count. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. If insufficient sample is available for an MS/MSD and batch specific precision is required, an LCSD may be substituted. In the event that multiple MS/MSDs are run with a batch due to client requirements, the additional MS/MSDs do not count toward the maximum 20 samples in a batch.

9.2. One method blank must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. For aqueous samples, the method blank is an aliquot of laboratory reagent water. Use Ottawa sand as the method laboratory matrix when solids are extracted. Use a mixture of Ottawa sand and canola oil as the matrix when tissues are extracted. Take the method blank through all steps detailed in the analytical procedure. The method blank is processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a non-conformance memo (NCM), then implemented when target analytes are detected in the method blank above the reporting limit or when surrogate or IDA recoveries are outside of the control limits. Re-extraction of the blank, other batch QC, and the affected samples are required when the method blank is deemed unacceptable. See policy WS-PQA-0003 for specific acceptance criteria.

9.2.1. The method blank must not contain any analyte at or above the reporting limit, or at or above 10% of the measured concentration of that analyte in the associated samples, whichever is higher.

9.2.2. If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.

9.2.3. Projects performed under the auspices of the DoD/DOE QSM must meet specific criteria for method blanks. Results are acceptable if the blank contamination is less than ½ of the reporting limit for each analyte, or less than 1/10 of the regulatory limit, or less than 1/10 of the sample result for the same analyte, whichever is greater. If the method blank does not meet the acceptance criteria, the source of contamination must be investigated and measures taken to correct, minimize or eliminate the problem. If contamination remains, the contaminated samples should be re-prepared and reanalyzed with a new MB and batch-specific QC samples.

9.2.4. If method blank contamination is present, check solvents, reagents, fortification solutions, apparatus and glassware to locate and eliminate the source of contamination before any further samples are extracted and analyzed.

9.2.4.1. OCDD is a ubiquitous laboratory contaminant.

9.2.4.2. A method blank and the associated samples are deemed acceptable if the OCDD concentration is less than 5 times the specified reporting limit.

9.2.4.3. If the OCDD is greater than 5 times the specified limit, flag data appropriately.

9.2.4.4. The analyst is expected to investigate and eliminate potential sources of systematic contamination.

9.2.4.5. If a target analyte is detected in the blank but the associated samples are ND (not detected), then the data may be reported, unless otherwise directed by the client. Note the action in the narrative.

9.2.4.6. If a target analyte is detected in the blank, but the concentration of the contaminant in the samples is greater than 10 times the blank concentration, then the data may be reported, unless otherwise directed by the client. Note the action in the narrative.

9.2.4.7. Method blank contamination of non-target analytes (i.e., non-2,3,7,8-substituted isomers) does not require corrective action, but a non-conformance memo is generated explaining the contamination and impact on sample data. "Totals" concentrations are not flagged to reflect blank contamination because totals are considered estimated values.

9.2.4.8. If the method blank is contaminated with any 2,3,7,8-substituted target analyte above the lower calibration limit (other than the exception noted above for OCDD), and the associated sample contains a positive result for that same analyte above the LCL and less than 10 times the blank concentration. Re-extraction of the associated sample is required, unless otherwise stipulated by the client, where the occurrence shall be documented in the non-conformance memo.

9.2.5. If new batches of reagents or solvents contain interfering contaminants, purify or discard them.

9.3. A Laboratory Control Sample (LCS) must be extracted with every process batch of similar matrix, not to exceed twenty (20) environmental samples. The LCS is an aliquot of laboratory matrix (e.g. water, Ottawa sand, sodium sulfate, etc.) spiked prior to extraction with analytes of known identity and concentration. The LCS must be processed in the same manner and at the same time as the associated samples.

9.3.1. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside control limits provided on the LIMS or by the client. Limits for Method 1613B are defined by the method, and are listed in Table 9.

9.3.2. Re-extraction of the blank, other batch QC and all associated samples are required if the LCS is deemed unacceptable.

9.3.2.1. Control charts are evaluated per WS-PQA-0003 to assure these limits are met.

9.3.3. A LCS is deemed acceptable if control analytes are above control limits and the associated samples are non-detect, unless otherwise specified by the client. Note any actions in the narrative.

9.3.4. For drinking water samples for the State of Arizona, a second LCS spiked at the method reporting limit must be prepared. For clarity it will be named the Laboratory Fortified Blank (LFB). One LFB must be prepared for each day drinking water samples are extracted for the State of Arizona. All compounds in the LFB must have a recovery between 50-150%. (For Arizona samples see WS-WI-0014 – Method 1613B for Arizona Compliant Samples)

9.4. The assessment of matrix effects on method performance, as required by NELAP, is met in Method 1613, as in all isotope dilution techniques, with the use of isotopically labeled compounds.

9.4.1. These isotopically labeled compounds are analogs of target analytes and are spiked into each sample. Therefore, matrix effects on method performance can be judged by the recovery of these analogs. Sample analysis acceptance is controlled by the performance of these analogs in each sample.

9.4.2. A Matrix Spike/Matrix Spike Duplicate (MS/MSD or MS/SD) pair is extracted at the client's request only and is not required by Method 1613B. An MS/MSD pair are aliquots of a selected field sample spiked with analytes of known identity and concentration. The MS/MSD are also spiked prior to extraction with the same $^{13}C_{12}$-labeled IDA as the method blank, laboratory control sample, and field samples. The MS/MSD pair shall be processed in the same manner and at the same time as the associated samples.

9.4.2.1. The results obtained from the MS and MSD samples (percent recovery and concentrations of 2,3,7,8-substituted PCDDs/PCDFs) shall agree within 50 percent relative difference. Report all results, flag any outliers, and file a non-conformance memo for any outliers. Re-extraction is not required when a MS/MSD pair fail to meet one or more QC criteria.

9.5. An Instrument Blank must be evaluated after calibration standards are injected and before sample analysis may begin. This may be accomplished by analysis of a method blank. If a method blank is not available, an instrument blank must be analyzed.

9.5.1. An instrument blanks consists of solvent (isooctane, toluene, or tetradecane). It is evaluated by inspection for contamination that may affect sample analysis.

9.6. Isotope Dilution Analyte recoveries are flagged if they are outside the recovery goals. Quantitation by isotope dilution generally precludes any adverse effect on data quality due to isotope dilution analyte recoveries being outside QC limits as long as the signal-to-noise is greater than 10:1.

9.6.1. Evaluate data quality for usability, flag and submit a non-conformance memo for any analytes outside of the recovery criteria, and report if data is deemed not adversely effected.

9.6.2. Re-extraction of samples should be performed if signal-to-noise for any isotope dilution analyte is less than 10:1 or if isotope dilution analyte recoveries fall below 10%.

9.6.2.1. Re-extraction may be necessary under other circumstances when data quality has been determined to be adversely affected.

9.7. Duplicates – Upon client request, duplicates may be processed. A duplicate injection of a sample extract may be performed at the clients request to display instrument precision. The results of the laboratory duplicates (percent recovery and concentrations of 2,3,7,8-substituted PCDD/PCDF compounds) should agree within 50 percent relative difference. Report all results and flag any outliers, re-extraction is not required when RPD limits are not met.

9.8. Field Blanks – A batch of samples may contain a field blank sample of nominally uncontaminated soil, sediment or water that is to be processed for analysis. Treat this sample as all others.

9.9. Rinsate Samples – In addition to the field blank, a batch of samples may include a rinsate, which is a portion of the solvent (usually trichloroethylene) that was used to rinse sampling equipment. The rinsate is analyzed to assure that the samples were not contaminated by the sampling equipment. The rinsate sample must be processed like a regular sample.

9.10. Recommended Corrective Actions and Troubleshooting Steps

9.10.1. Verify satisfactory instrument performance.

9.10.2. If possible, verify that no error was made while weighing the sample portions.

9.10.3. Review the analytical procedures with the performing laboratory personnel.

10. CALIBRATION AND STANDARDIZATION

10.1. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to Policy CA-Q-P-003, "Calibration Curves and Selection of Calibration Points".

10.2. Two types of calibration procedures are required.

10.2.1. One type***, initial calibration***, is required before any samples are analyzed and is required intermittently throughout sample analysis as indicated by the results of the continuing calibration procedures described below.

10.2.2. The other type, ***continuing calibration***, consists of analyzing the column performance check solution and a calibration solution (typically L4). No samples are to be analyzed until acceptable calibration is demonstrated in sections 10.6 and 10.7.

10.3. The following GC operating conditions are typically used:

DB-5 Column
Injector temp: 280ºC
Interface temp: 280 ºC
Initial temp and time: 190 ºC, 1 min
Temp program: 190-240ºC at 4 ºC/min
240ºC for 16 min
240-320 ºC at 20 ºC/min
320 ºC for 10 min

DB-225 Column
Injector temp: 240ºC
Interface temp: 240ºC
Initial temp and time: 190ºC, 1 min
Temp program: 190-240ºC at 3.5ºC/min
240ºC for 20 min

10.3.1. For South Carolina samples, the retention time of ^{13}C-1,2,3,4-TCDD must exceed 25 minutes on the DB-5 column and 15 minutes on the DB-225 column.

Method 1613 suggests the following as a temperature program that can be used to meet these criteria:

DB-5 Column
Injector temp: 270ºC
Interface temp: 290 ºC
Initial temp and time: 200 ºC, 2 min
Temp program: 200-220ºC at 5 ºC/min
220ºC for 16 min
220-235 ºC at 5 ºC/min
235ºC for 7 min
235-330^{o}C at 5^{o}C/min

NOTE: All portions of the column, which connect the GC to the ion source, remain at the interface temperature specified above during analysis, to preclude condensation of less volatile compounds.

10.4. Mass spectrometer (MS) resolution - tune the mass spectrometer to achieve a minimum resolving power of 10,000 (at 10 percent of peak height) at m/z 304.9824 (PFK). Other masses monitored but not required by the method are as follows: 292.98245.

Function 1: *[292.98245], [318.97925], [330.97925], [342.97925], [354.97925], [366.97925], [380.97604], [392.97604], [404.97604], [416.97604]*

Function 2: *[330.97925], [342.97925], [354.97925], [366.97925], [380.97604], [392.97604], [404.97604], [416.97604]*

Function 3: *[366.97925], [380.97604], [392.97604], [404.97604], [416.97604], [430.97284], [442.97284], [454.97284]*

Function 4: *[404.97604], [416.97604], [430.97284], [442.97284], [454.97284], [466.97284], [480.96967]*

Function 5: *[430.97284], [442.97284], [454.97284], [466.97284], [480.96967], [492.96967], [504.96967], [516.96967]*

While in peak examine mode, print the peak profile display for all functions required, demonstrating that this minimum resolution criterion has been met.

10.4.1. Operate the mass spectrometer in mass drift correction mode.

10.4.1.1. A lock-mass ion from the reference compound PFK (perflourokerosene) is used to correct for mass drift in each group of masses (function) in the mass spectrometer experiment.

10.4.1.2. Excessive variations of the lock-mass can indicate the presence of coeluting interferences, which may significantly reduce the sensitivity of the mass spectrometer.

10.4.1.3. Re-extraction of a smaller aliquot or dilution of the original extract may be required to reduce these interferences to an acceptable level.

NOTE: Excessive PFK may cause elevated noise levels and a dirty ion source.

For the following injections, use the GC conditions specified in section 10.2 above.

10.4.2. Monitor each group (function) of masses (see Table 10) in succession as a function of retention time to ensure that all PCDDs and PCDFs are detected.

10.4.2.1. Inject 1-2 µL of concentrated sample extract (containing recovery standards) or standard solution using spitless injection.

10.4.2.2. Start GC isothermal hold and temperature program immediately upon injection.

10.4.2.3. Start MS data collection after the solvent peak has eluted.

10.4.2.4. At the end of the temperature program, (after the octachloro-dibenzodioxin and dibenzofuran have eluted), return the GC to the initial temperature for analysis of the next sample or standard.

10.5. Retention time windows

10.5.1. Analyze a 1-2 µL of the window defining mixture (WDM) to demonstrate that the GC/MS experiment is monitoring the masses for each PCDD and PCDF chlorination level from the retention time of the first eluting isomer until that of the last eluter.

10.5.2. Isomer specificity; Analyze the isomer specificity test standard (the CPSM).

10.5.2.1. This standard solution has been combined with the window defining mixture so that both parameters can be checked with a single GC/MS injection.

10.5.2.2. Compute the percent valley between the GC peaks that elute most closely to 2,3,7,8-TCDD and TCDF on their respective columns.

10.5.2.3. Verify that the height of the valley between the most closely eluted isomers and the 2,3,7,8-isomers does not exceed 25 percent of the height of the latter.

- If this criterion is not met, GC maintenance will likely be necessary.

10.5.2.4. **NOTE**: The CPSM/WDM is required prior to sample analysis, but need not be acquired prior to the ICAL and is not provided as part of the ICAL standards package.

10.6. Initial Calibration

10.6.1. A five-point calibration curve encompassing the concentration range is used for each compound to be determined (see Table 5).

10.6.2. To calibrate the analytical system, inject a 1-2 µL aliquot of each of the 6 calibration standards (L1 to L6; Table 5). (L1 ultra low level is optional)

10.6.3. Print hardcopies of the mass chromatograms (masses listed in Table 9 and 13).

10.6.4. Verify that the signal-to-noise ratio (S/N) is greater than or equal to 10 for all PCDD/PCDF ions.

10.6.4.1. Generally the unlabeled TCDD/TCDF ions for solution L1 and L2 are the ones for which meeting this requirement can be difficult.

10.6.4.2. Reducing the level of PFK being bled into the source and attempting to retune the mass spectrometer for better sensitivity are two measures which often remedy low signal-to-noise ratio.

- For Arizona samples, the level of PFK metered into the HRMS during analysis should be adjusted so that the amplitude of the most intense selected lock-mass m/z signal (regardless of the descriptor number) does not exceed 10% of the full-scale deflection for a given set of detector parameters.

10.6.5. Compute ion abundance ratios for the two ions monitored for each labeled compound and analyte; verify that these are within the limits given in Table 2.

10.6.6. Calculate the relative response factor (RRF) of each unlabeled compound vs. its isotope dilution analyte, and of each isotope dilution analyte vs. its internal standard (Table 10).

10.6.6.1. Compute the mean RRF, standard deviation and percent compound.

10.6.6.2. If the relative standard deviation exceeds 20 percent for any native compound, or 30 percent for any labeled compound, the mean RRF may not be used.

10.6.7. A relative response factor is calculated for each unlabeled PCDD/PCDF vs. its 13C-labeled analogue standard using the area responses of both the primary and secondary ions (specified in Table 9). These relative response factors are calculated as follows:

$$RRF = \frac{(A_{s1} + A_{s2})\, C_{IDA}}{(A_{IDA1} + A_{IDA2})\, C_s}$$

Where:

A_{s1} and A_{s2} are the areas of the primary and secondary ions for the compound to be calibrated (NOTE: only one ion is used for 37Cl4-2, 3 ,7, 8-TCDD).

A_{IDA1} and A_{IDA2} are the area of the primary and secondary ions for the isotope dilution analyte (or internal standard when a surrogate or cleanup recovery standard is being calibrated).

C_{IDA} is the concentration of the isotope dilution analyte (or internal standard when an surrogate or cleanup recovery standard is being calibrated).

C_S is the concentration of the compound to be calibrated

10.7. Initial calibration verification standard (ICV): A second source calibration standard is analyzed following the initial calibration curve, prior to samples. Criteria are as follows: All native (unlabelled) compounds must be within ± 30% of expected value. IDA (labeled) compounds must meet the requirements of Table 9/9a for the VER.

10.8. Continuing Calibration:

At the beginning of each 12-hour shift during which samples are to be analyzed, GC/MS system performance and calibration are verified for all unlabeled and labeled compounds.

10.8.1. MS Resolution - A static resolving power of at least 10,000 (10 percent valley definition) must be demonstrated at appropriate masses before any analysis is performed.

10.8.1.1. Static resolving power checks are performed at the beginning and ending of each analytical sequence.

- An analytical sequence is defined as 12 hours. The beginning of a 12 hour sequence is the injection time of the first analysis following the beginning resolution check. The end is marked by the injection time of the last analysis within the 12 hour sequence..

10.8.1.2. The MS Resolution Checks bracket all samples analyzed in a 12 hour sequence, during which time no instrument parameters are

changed. Static mass resolving power checks must be performed at the beginning and end of each 12 hour run.

10.8.2. Analyze the CPSM/WDM mixture at the beginning of each 12-hour period to ensure that the retention time window and isomer specificity criteria are met.

10.8.2.1. This mixture can be analyzed either immediately before or immediately after the calibration verification standard.

10.8.3. The L4 calibration standard ("VER Standard," Table 5) is analyzed and quantitated using the relative response factors calculated from the initial calibration.

10.8.4. The calculated concentration of each unlabeled and labeled compound is compared to the range given in the Table 9. If all of the following criteria are met, calibration has been verified:

10.8.4.1. Ion abundance ratios for all PCDDs and PCDFs are within the limits given in Table 2.

10.8.4.2. The peaks representing each unlabeled and labeled compound, calculated using averaged relative response factors (RRFs) from the initial calibration curve, is within the limits specified in Table 9.

10.8.4.3. If any of these criteria are not met, try retuning the mass spectrometer and reinjecting the VER Standard.

10.8.4.4. If each peak's S/N ratio does not meet 10:1, the instrument sensitivity is inadequate and needs attention. Note: filament replacement may solve this issue, otherwise, replacing the ion source with a clean one will probably be necessary.

10.8.4.5. If, after two or more injections, sensitivity of the mass spectrometer is adequate but the concentrations found in the VER Standard continue to fall outside the limits stated in Table 9, the initial calibration should be repeated, if no additional problem with the instrument can be found.

10.8.4.6. As a standard practice to increase efficiency, samples may have been analyzed before the Continuing Calibration Standard (CCV) has been checked.

10.8.4.7. If any CCV criteria or CPSM criteria is not met, review the sample data for usability. If there is no adverse impact to data quality, document using an NCM, and the data may be reported otherwise

reinject. Perform corrective action before the next analytical sequence.

10.9. Retention times and GC resolution – The relative retention times (RRT) of unlabeled and labeled PCDDs and PCDFs should be within the limits specified in Table 8. In addition the absolute retention times of $^{13}C_{12}$-1,2,3,4-TCDD and $^{13}C_{12}$- 1,2,3,7,8,9-HxCDD internal standards must be within +/- 15 seconds of the relative retention time obtained during calibration. Routine column maintenance is performed and may cause minor retention time variations which may result in RT shifts greater than +/-15 seconds. In this case, data is evaluated and if no adverse impact to the data quality is observed, the RT shift will be documented using an NCM.

11. PROCEDURE

11.1. HRGC/HRMS analysis

11.1.1. Analyze the sample extracts under the same instrument operating conditions used to perform the instrument calibration.

11.1.2. Record the analysis information in the instrument logbook. Peer review of each analytical sequence is required and must be documented with date and initials. Document and verify each of the following in the instrument logbook: Data File ID, Sample #, Sample ID, and Autosampler position.

11.2. Ongoing precision and recovery – Analyze the extract of the diluted precision and recovery standard (PAR), known as the LCS. Calculate the concentration of each unlabeled and labeled PCDD and PCDF and compare it with the limits for ongoing accuracy in Table 9.

11.2.1. If all compounds meet acceptable criteria, system performance is acceptable. If, however, any individual concentration falls outside the range given, the associated sample set should be evaluated to determine whether there is adverse impact to data quality.

11.2.1.1. If data quality has been unaffected (for example, an LCS unlabeled analyte has a high percent recovery, but all associated samples are ND) anomalize using an NCM, and report the data.

11.2.2. If it is determined that data quality has been adversely affected (for example, in the LCS an unlabeled analyte has a high recovery and the associated samples are positive) re-extract the adversely affected samples. (For Arizona samples see WS-WI-0014 – Method 1613B for Arizona Compliant Samples)

11.2.3. LCS (OPR) samples (method blanks, spiked with unlabeled PCDDs and PCDFs) are run at the rate of one for every 20 samples extracted from a given

matrix, or one per extraction batch of a given matrix. The LCS is usually prepared at a mid-level concentration. A second LCS (LCSD) is required for each extraction batch if MSQC (MS/MSD pair) is not performed. For (State of Arizona sample protocol only) drinking water samples a low level LCS (LLCS) is prepared at ½ the reporting limit concentration. Only one LLCS is prepared for each drinking water extraction batch that contain State of Arizona regulated samples. All compounds in the LLCS must have a recovery between 50-150%.

11.2.4. All results, including those that fall outside the OPR specifications in Table 9 are entered into TALS so that an ongoing precision and recovery database for each compound in each matrix can be maintained

11.2.5. Blank – Method Blank may be analyzed either after the CCV or after the solvent blank following a CCV or CPSM.

11.2.5.1. For South Carolina samples, any positive result in the method blank greater than 1/3 the reporting limit will be reported.

11.3. Qualitative identification: For a GC peak to be identified as a PCDD or PCDF (either an unlabeled or a labeled compound), it must meet all of the criteria below.

11.3.1. The signals for the two m/z's being monitored (See Table 10) must be present and must maximize within $\pm$ 2 seconds of each other.

11.3.2. The signal-to-noise (S/N) ratio of both the m/z's must be greater than or equal to 2.5 for a sample extract, and greater than or equal to 10 for a calibration standard.

11.3.3. The ratio of the integrated areas of the two exact m/z's monitored shall be within the specified limits (See Table 2) or within $\pm$10% of the ratio in the midpoint (L4) calibration.

11.3.4. The relative retention time (RRT) of the peaks for a 2,3,7,8-substituted PCDD or PCDF must be within the RRT limits (See Table 8). The retention time of peaks representing non 2,3,7,8-substituted PCDDs/PCDFs must be within the retention time windows established by the window defining mixture injected before sample analysis.

11.3.5. Confirmatory analysis—Any sample in which 2, 3, 7, 8-TCDF is identified above the lower calibration limit must be confirmed on a DB-225 column, SP-2331, or equivalent GC column.

11.3.5.1. The operating conditions may be adjusted to optimize the analysis on the second GC column.

11.3.5.2. The GC/MS must meet the mass resolution and calibration specifications in Section 10.

11.3.6. No signal shall be observed in the corresponding PCDPE ion channel having a S/N ratio greater than 2.5 at the retention time of any presumptive PCDF.

11.3.6.1. Chemical interference, rather than an interfering PCDPE may cause a signal to be observed at the m/z of the corresponding DPE.

11.3.6.2. A professional analytical judgement is necessary to determine whether a particular unlabelled analyte peak should be deleted from the "total", or the 2,3,7,8 substituted set to a detection limit because of possible DPE interference.

11.3.6.3. Additionally, professional analytical judgement is necessary to determine whether the peak in question should be qualified as a positive by a reporting flag ("JA"), or whether the peak should be counted as a positive with no qualification because no applicable interference is present.

11.4. Analysis of complex samples.

11.4.1. Some samples contain high levels (>10 ng/L; >1000 ng/kg) of the compounds of interest, interfering compounds, and/or polymeric materials. In the case of high levels of one or more analytes (such that the concentration in the extract exceeds the upper calibration limit and/or causes saturation of the MS detector), the extract is diluted with tetradecane to bring the concentration (s) of the analyte (s) in question below the limit of the detector and into the calibration range if possible.

11.4.2. If high levels of coextracted interfering compounds are a problem, a smaller aliquot of the sample is extracted and analyzed. As noted above, complex dilution may be required. The client will be contacted to determine whether or not they will require this procedure.

11.4.3. For isotope dilution analyte recoveries outside the recommended recovery range the data should be evaluated for adverse impact. If no significant adverse impact was observed the data should be reported and an NCM filed.

11.4.3.1. For samples that may or are adversely impacted an additional aliquot of the original extract or diluted extract should be analyzed.

- If the recoveries are still outside of the range for the re-injected extract and it is determined that significant adverse impact to data quality has occurred, the sample(s) may be re-extracted, or reprocessed from an archive portion if available.

- A smaller aliquot should be used for the re-extraction unless it is suspected that the recovery problem is not matrix related.
- If the re-extracted sample(s) still does not meet the limits for labeled isotope dilution analyte recovery, the data is evaluated for adverse impact and a NCM filed.
- All recoveries outside of method criteria will be flagged. The best quality data is reported from one of the two extractions unless the client requests that both sets of data be reported.

11.4.4. Optional procedure for use in the analysis of 2,3,7,8-TCDF and/or 2,3,7,8-TCDD only.

11.4.4.1. 2,3,7,8-TCDD and/or 2,3,7,8-TCDF can either be analyzed by using the procedures above, but the sample spiking solution can be modified to include only the labeled and unlabeled analytes of interest. This can reduce the cost of the spiking standards used. If this procedure is used, the spiking mixes can be QC'd against the existing "full-list" calibration curve.

11.4.4.2. The spiking solutions, including stock and secondary standards, are modified such that only the unlabeled and labeled 2,3,7,8-TCDD and 2,3,7,8-TCDF analytes are added, including the ^{37}Cl-2,3,7,8-TCDD (Cleanup Recovery Standard or Surrogate) and ^{13}C-1,2,3,4-TCDD (Internal Standard).

11.4.4.3. The silica column/alumina column cleanup (as described in WS-IDP-0007) may be optimized for TCDD and TCDF.

11.4.4.4. Carbon Column (as described in WS-IDP-0007) may be optimized for TCDD and TCDF.

11.4.4.5. The temperature program may be modified to optimize the GC run for separation of the TCDD and CDF isomers, and to shorten the run as appropriate.

11.4.4.6. All aspects of calibration and quantitation also apply when they refer to the Tetra analytes and isotope dilution analytes only.

11.5. Data review

11.5.1. When an entire project lot or batch is completed, a checklist representing the entire data package is completed. All manual integrations must be documented according the Manual Integration SOP, S-Q-004.

11.5.2. A second analyst must verify all qualitative peak identifications.

11.5.2.1. If discrepancies are found, the data should be returned to the analyst who performed the initial peak identification for resolution.

11.5.2.2. Any changes made directly by the reviewing analyst must be dated and initialed by the reviewing analyst.

11.5.3. All hand calculations and data entry into calculation programs, databases, or spreadsheets must be checked by a second analyst at a frequency of 100 percent.

11.5.3.1. If discrepancies are found, the data should be returned to the analyst who performed the initial calculation.

11.5.3.2. Any changes made directly by the reviewing analyst must be dated and initialed by the reviewing analyst.

11.5.4. Both the analyst who performed the initial qualitative and quantitative analysis and the analyst who performed the second level review must check all items on the data review checklist.

11.6. Common observations with "real world" sample data analysis:

11.6.1. If an ion ratio is outside ratio identification limits, but within the retention time window, the analyst, using professional judgement based on historical data or other samples within the sample lot, identify the peak in question as positive with an estimated concentration based on the theoretical ratio. The CHROM software will automatically adjust the ion areas to reflect the theoretical ion ratio based on the ion peak with the smallest area and then quantitate an estimated concentration. The "Q" flag is used which states that the peak has been positively identified but the concentration is estimated. (Note: non 2,3,7,8 substituted totals peaks are not qualified)

11.6.1.1. The first eluting PeCDD isomer often has a chemical interference in the m/z 357.8516 ion causing the ion ratio to be outside the ratio identification window for PeCDDs.

- If this peak is the only positive PeCDD identified for "total" PeCDD, the detection limit is elevated to reflect the estimated concentration and the total is "ND" at this value.
- If there are other positive PeCDDs within the window, the isomer will be "Q" flagged as estimated on the totals summary sheet, and included in the "total" concentration. (Note that "totals" are always considered estimated and are not flagged on the final result sheet.) No NCM is required.

11.6.1.2. The 1,2,3,4,6,7,8-HpCDF isomer often has a chemical interference with the m/z 407.7818 ion that causes the ion ratio with m/z 410 to be outside the method limits.

- Often, re-integration of the m/z 407.7818 ion is possible, but if this is not possible, qualify positive values for the 1,2,3,4,6,7,8-HpCDF isomer as "positively identified, but estimated quantitation" using the "Q" qualifier because the quantitation is based on the theoretical ion ratio. A NCM observation is filed.

11.6.2. The following 2,3,7,8-substituted target analytes often require re-integration due to closely eluting non-2,3,7,8-substituted isomers on the DB-5 column. In all cases, the ^{13}C-labeled analog should be used to determine where the split between the isomers should occur.

11.6.2.1. 2,3,7,8-TCDD elutes just after the 1,2,3,7/1,2,3,8-TCDD elutes. Even though the DB-5 column is isomer specific for 2,3,7,8-TCDD, the software often integrates these isomers together when the 2,3,7,8-TCDD concentration is much less that that of the other isomer pair.

11.6.2.2. 1,2,3,4,7,8-HxCDF elutes just after the 1,2,3,4,6,7-HxCDF isomer; 2,3,4,6,7,8-HxCDF elutes just after the 1,2,3,6,8,9-HxCDF isomer, and 1,2,3,7,8,9-HxCDF elutes just prior to the 1,2,3,4,8,9-HxCDF isomer.

11.6.2.3. 1,2,3,7,8,9-HxCDD elutes just after the 1,2,3,4,6,7-HxCDD isomer.

11.6.3. Biological samples often exhibit a typical Diphenyl Ether pattern, while Incineration Ash samples often exhibit chemical interference in the DPE traces that does not contribute ions to the native furans.

11.7. Recommended Preventative Maintenance

As Needed Maintenance:
Full Bake-Out.
Change oil in rotary pump.
Change oil in diffusion pump. Replace o-rings.
Solvent rinse the flight tube.
Clean the first field free region.
Check detector voltages.
Clean and dust connectors, etc on the outside of the instrument.
Check the vacuum: ~5 x. 10^{-7} MBAR on both analyzer ion gauges, and ~5 x 10^{-6} MBAR on the source, with no helium flowing.
Check isolation valve for leaks, correct if needed.
Check for thermal trip by taking the magnet to maximum current, and verify that the coolant flow is acceptable.
Replace septum.

Clean injector port. Cut off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required. Replace injection port liner when front portion of capillary column is removed. Clean Source, including all ceramics and lenses - the source cleaning is indicated by a variety of symptoms including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination. Replace filaments when performance indicates need for replacement.
Daily Maintenance:
Check resolution sensitivity. Check stability. Check for sufficient gas supply. Check for correct column flow and/or inlet pressure. Check temperatures of injector, detector. Verify temperature programs. Check inlets, septa. Check baseline level. Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds. Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.

12. CALCULATIONS/DATA REDUCTION

12.1. Isotope dilution: By adding a known amount of a labeled compound to every sample prior to extraction, correction for recovery of the unlabeled compound can be determined. The correction for the unlabeled compound can be made because the unlabeled compound and its labeled analog exhibit similar effects upon extraction, cleanups, concentration, and gas chromatography mass spectrometry. Relative response factors (RRF) values are used in conjunction with calibration data described in Section 10 to determine concentration directly, using the following equation:

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Where:

C_n is the concentration of the unlabeled compound in the extract.

A_n is the area of the native in the lighter mass (1) and heavier mass (2).

C_{IDA} is the concentration of the isotope dilution analyte.

RRF_n is the relative response factor from the calibration curve for that compound.

Vol is the sample size used for the extraction and/or cleanups (in milliliters, liters, grams, or extract.

12.1.1. Reporting conventions are as follows:

12.1.1.1. Aqueous results in pg/L (parts per quadrillion);

12.1.1.2. Solid results (soils, sediment, filter cake, compost, waste, etc) in pg/g or ng/kg (parts per trillion);

- Solid samples may be reported on a dry weight basis by reporting and correcting for percent solids.

12.1.1.3. Extracts or filters in pg/sample or pg/extract (parts per quadrillion).

12.1.1.4. Tissue sample results in pg/g or ng/kg of wet tissue, not on the basis of the lipid content of the sample. Report the percent lipid content if requested.

12.1.2. Because of a potential interference, the labeled analog of OCDF is not added to the sample. Therefore, this unlabeled analyte is quantitated against the labeled OCDD. As a result, the concentration of unlabeled OCDF is corrected by the recovery of the labeled OCDD.

12.1.2.1. In instances where OCDD and OCDF behave differently during sample extraction, concentration, and cleanup procedures, a decrease in the accuracy of OCDF may result. However, given the low toxicity of this compound relative to other dioxins and furans, the potential decrease in accuracy is not considered significant.

12.1.3. Because the labeled analog of 1,2,3,7, 8, 9-HxCDD is used as an internal standard (i.e. not added before extraction of the sample), it cannot be used to quantitate the unlabeled compound by strict isotope dilution procedures.

12.1.3.1. The unlabeled 1,2,3,7,8,9-HxCDD is quantitated using the averages of the responses of the labeled analogs of the other two 2,3,7,8-substituted HxCDD's, 1, 2, 3, 4, 7, 8-HxCDD and 1, 2, 3, 6, 7, 8-HxCDD. As a result, the concentration of the unlabeled 1, 2, 3, 7, 8, 9-HxCDD is corrected for by the average recovery of the other two HxCDD IDA.

12.1.4. Any peaks representing non-2, 3,7,8-substituted dioxins or furans are quantitated using an average of the response factors from all of the 2,3,7,8-isomers in the same level of chlorination.

12.1.5. Report results at or above the minimum level (also known as the Lower Calibration Limit – See Table 5).

12.1.6. Report results below the minimum level as not detected. Some clients or regulatory agencies require sample/analyte specific estimated detection limits. If these are required, there is a three-tiered approach to reporting and detection limits.

12.1.6.1. In the absence of target analytes, a sample specific estimated detection limit (EDL) is calculated based on signal-to-noise (S/N) ratios at the retention time of the target analyte.

- The target analyte is then reported as "not detected" at the EDL.

12.1.6.2. When target analytes meet method identification criteria and are free of interferences, they are reported as unqualified positives down to the lowest calibration standard concentration (the LCL, See Table 5).

- Below the lower calibration limit, qualitatively confirmed analytes are reported as "estimated" down to the project detection limit. The default is the EDL but that can change based on specific project requirements.
- To denote the less certain quantitation the value is "J" flagged as estimated.

12.1.6.3. The TDL is a value set by the lab at which there is no significant chance of a false positive (usually set at ½ the LCL value).

- If there is a peak below the TDL, a detection limit based on the ion peaks is calculated and the target analyte is reported as "not detected" at the calculated detection limit.
- Second column confirmation will be performed only for 2,3,7,8-TCDF positives greater than the lower calibration limit (Note: "Totals" peaks, i.e. non-2,3,7,8-substituted isomers are not considered target analytes, but are identified using the above criteria. "Totals" concentrations are always considered estimated and are never flagged.)

12.1.7. If the concentration of any target analyte exceeds the upper calibration range of the initial calibration curve, but does not saturate the instrument detector, the analyte is qualified by the "E" flag and an NCM is submitted. ("Totals" concentrations are always considered as estimates and are not flagged.)

12.1.7.1. Historical data indicates that for the isotope dilution method, dilution and re-injection will not produce significantly different results from those reported with the "E" qualifier.

12.1.7.2. Clients may request dilution and re-injection to bring target compounds within the calibration range. (Note: dilution of an extract to bring target compounds within range also dilutes the isotope dilution analytes which may adversely impact the data).

12.1.8. When target analyte peaks saturate the detector, TestAmerica Sacramento will provide one dilution to bring the response below the saturation level and/or the upper calibration level as part of the initial analysis cost.

12.1.8.1. Additional dilutions or re-extraction at a smaller sample aliquot will be performed at client request only. (Note: Complex dilution is one where the pre-spiked isotope dilution analytes are diluted out and must be re-added to the extract. The sample analyte concentrations are then calculated using an internal standard method and all results from this dilution are therefore estimated.)

12.1.9. Results for samples that required dilution may be reported from more than one injection. For example, some analyte results may be reported from the original injection, while others are taken from the dilution.

12.1.9.1. Isomers reported from a dilution shall be qualified with a "D" qualifier, or narrated if all or most of the congeners are reported from the dilution analysis. (Note: Dilution will not always eliminate the "E" flag qualifier.)

12.1.10. The total concentration in each level of chlorination is the sum of the concentrations of all isomers identified in that level greater than the target detection limit, including any non-2,3,7,8-substituted isomers, which elute within the CPSM window.

12.1.10.1. The total concentration is calculated by the CHROM software as follows: The total area of 2,3,7,8-substituted isomers is subtracted from the total area of all isomers to get a non-2,3,7,8 substituted total area. A non-2,3,7,8-total concentration is then calculated using the average RRF value. The 2,3,7,8-substituted isomer concentrations are added to the non-2,3,7,8-total concentration to get an "actual" total for all isomers.

12.1.11. Results are reported to two significant figures. For aqueous samples, the units are pg/L. For solid samples (soils, sludges, sediments, etc) the units are pg/g.

12.2. Isotope Dilution Analyte : Compute the concentrations of the ^{13}C-labeled analogs and the ^{37}Cl-labeled Surrogate in the extract using the response factors determined from calibration curve and the following equation:

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Where:

C_{IDA} is the concentration of the isotope dilution analyte in the extract (pg/g, pg/L)

A_{IDA} the area of the isotope dilution analyte in the lighter mass (1) and the heavier mass (2).

A_{IS} is the area of the internal standard in the lighter mass (1) and heavier mass (2).

Q_{IS} is the amount of internal standard spiked.

RRF_{IDA} is the response of the isotope dilution analyte relative to the response of the internal standard in the initial calibration curve.

12.3. Calculate the isotope dilution analyte percent recovery using the following equation:

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(Note: There is only one m/z for the ^{37}Cl-labeled standard.)

13. METHOD PERFORMANCE

13.1. Method Detection Limit

Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in QA Policy #: CA-Q-S-006 and WS-QA-0006. MDLs are available in the Quality Assurance department.

13.2. The group/team leader has the responsibility to ensure that this procedure is performed by an associate, who has been properly trained in its use and has the required experience.

13.3. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QC files.

13.4. The laboratory operates a formal quality assurance program. The minimum requirements of this program consist of an initial demonstration of capability (IDOC), analysis of samples spiked with compounds to evaluate and document data quality, and analysis of standards and blanks as tests of continued performance.

13.4.1. The analyst shall make an initial demonstration of the ability to generate acceptable accuracy and precision by this method. This ability is established by generating four laboratory control samples that meet recovery criteria. For each alternative sample matrix, four aliquots of the alternative reference matrix are used.

13.5. The laboratory must make a one time initial demonstration of capability for each individual method. Demonstrations of capability for both solid and aqueous matrices are required. This requires analysis of QC check samples containing all of the standard analytes for the method.

13.5.1. Four aliquots of the QC check samples are analyzed using the same procedure used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid-level calibration.

13.5.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these results with the acceptance criteria in Table 9.

13.5.3. If any analyte does not meet the acceptance criteria, then the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.5.4. In recognition of advances that are occurring in analytical technology, and to allow the analyst to overcome sample matrix interferences, the analyst is permitted certain options to improve separations or lower costs of measurements. These options include alternate extraction, concentration, cleanup procedures, and changes in columns and detectors. Unique changes are to be documented using a non-conformance memo. Permanent modifications or additions should be documented using an addendum to this SOP.

14. POLLUTION CONTROL

It is TestAmerica 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 Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.1. Standards should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards that must be discarded.

15. 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 WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Autovials contaminated with methylene chloride, toluene, tetradecane and other solvents. As the autovials are removed from the instrument after analysis, they are stored in vial files in the instrument lab for at least ninety days, depending on client requirements. After at least ninety days, the vial files are transferred to the waste disposal area where they are drummed and shipped as PCB waste after no more than 90 days.

15.2. Waste methylene chloride generated during glassware and sodium sulfate cleaning and various rinses. Keep waste methylene chloride separated from waste flammable solvents. Collect the waste methylene chloride in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel methylene chloride waste drum in the H3 closet. When the drum is full to six inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment.

16. REFERENCES/CROSS-REFERNCES

16.1. EPA Method 1613, Revision A, October 1990.

16.2. EPA Method 1613, Revision B, October 1994.

16.3. Federal Register, 40 CFR Part 136, Guidelines Establishing Test Procedures for the Analysis of Pollutants; EPA Method 1613, Final Rule, 10/1994, NTIS PB95-104774.

17. METHOD MODIFICATIONS

17.1. Deviations from referenced method:

17.1.1. Section 10.2.4 of Method 1613 reads “The absolute retention time of ^{13}C-1,2,3,4-TCDD shall exceed 25.0 minutes on the DB-5 column, and the retention time of ^{13}C-1,2,3,4-TCDD shall exceed 15.0 minutes on the DB-225 column; otherwise the GC temperature program shall be adjusted and this test repeated until the above-stated minimum retention time criteria are met.” Our retention time on both columns deviate from the above method, but using section 1.5 of Method 1613 “the analyst is permitted to modify the method to lower the cost of measurements, provided that all performance criteria in this method are met,” we have modified the GC program to provide a shorter run time while still meeting method performance criteria thus lowering the cost of analysis.

17.1.2. Section 10.2.1.2 of Method 1613 reads “The lock-mass for each group of m/z’s shall be monitored and shall not vary by more than $\pm$20% throughout its respective retention time window.” Data is evaluated on a sample by sample basis to establish whether the lock-mass deviations have adversely affected data quality. “Totals” are always considered estimated values, so lock-mass dips effecting a non-2,3,7,8-substituted isomer may be considered inconsequential. If a target 2,3,7,8-substituted isomer is adversely affected by a lock-mass deviation the sample may be diluted and re-injected or re-injected on a different instrument.

17.1.3. Section 10.1.2.1 of Method 1613 reads “The level of PFK metered into the

HRMS during analysis should be adjusted so that the amplitude of the most intense selected lock-mass m/z signal (regardless of the descriptor number) does not exceed 10% of the full-scale deflection for a given set of detector parameters." The qualifier "should" indicates that action is suggested but not required (see Section 24.0 of Method 1613). The exception to this is state of Arizona drinking water samples. For Arizona drinking water samples, following section 10.1.2.1 is required.

17.1.4. Section 17.6.1.4.2 of Method 1613 reads "Blanks – report results above one-third the method limit." We report blanks using the same criteria as that used for samples.

17.1.5. Section 17.6.2 of Method 1613 reads "Results for the PCDDs/PCDFs in samples that have been diluted are reported at the least dilute level at which the areas at the quantitation m/z's are within the calibration range." Data quality is always taken into account. Every effort is made to accommodate this method suggestion, but exceptions may occur when actual data results are analyzed.

17.1.6. Section 18.3 of Method 1613 reads "Chlorodiphenyl Ethers – If chromatographic peaks are detected at the retention time of any PCDDs/PCDFs in any of the m/z channels being monitored for the chlordiphenyl ethers, cleanup procedures must be employed until these interferences are removed." Chlorodiphenyl Ethers (DPE) peaks will only interfere with PCDFs, so references to PCDDs are not applicable. PCDF data is analyzed to determine whether DPE interference is present. If DPE peaks are present at –2 to +2 seconds from the retention time of a peak in a PCDF trace, that peak is eliminated from the "total" concentration when there are other positive PCDFs present without DPE interference. Otherwise, these DPE interference peaks are taken into consideration when determining a "total" detection limit.

18. ATTACHMENTS

18.1. Tables or figures referenced in body of SOP.

18.1.1. Figure 1 - Compound Structure

18.1.2. Table 1 - Isomers of Chlorinated Dioxins and Furans

18.1.3. Table 2 - Theoretical Ion Abundance Ratios and QC Limits

18.1.4. Table 3 - The Seventeen 2,3,7,8-Substituted PCDD and PCDF Congeners

18.1.5. Table 4 – Concentration of Stock and Spiking Solutions

18.1.6. Table 5 - Concentrations of Calibration Solutions

18.1.7. Table 6 – GC Retention Time Window Defining Test

18.1.8. Table 7 - List of CDDs/CDFs Determined by ID/IS/HRGC/HRMS

18.1.9. Table 8 – Retention Time Reference, Relative Retention Time

18.1.10. Table 9 – Acceptance Criteria for Performance Test

18.1.11. Table 9.1 – Acceptance Criteria for Performance Test (Tetras-Only Analysis)

18.1.12. Table 10 – Descriptors, Exact m/z's

19. REVISON HISTORY

19.1. WS-ID-0007, Revision 4.1, Effective 06/15/2017

19.1.1. Changed all "CS" references to "L" (example: CS-4 is now L4).

19.1.2. Removed Section 3.15, "Performance Evaluation Materials (PEM's): Representative sample portions containing known amounts of certain unlabeled PCDD/PCDF congeners (in particular the ones having a 2,3,7,8-substitution pattern). Representative interferences may be present. PEMs may be obtained from the EPA EMSL-LV or other sources and submitted to potential contract laboratories, which must analyze these and obtain acceptable results before being awarded a contract for sample analyses (see IFB Pre-Award Bid Confirmations). PEMs may also be included as unspecified ("blind") quality control (QC) samples in any sample batch submitted to a laboratory for analysis."

19.1.3. Added Section 15.2, "Waste methylene chloride generated during glassware and sodium sulfate cleaning and various rinses. Keep waste methylene chloride separated from waste flammable solvents. Collect the waste methylene chloride in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel methylene chloride waste drum in the H3 closet. When the drum is full to six inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment."

19.1.4. Editorial changes.

19.2. WS-ID-0007 Revision 4.0, Effective 06/09/2016

19.2.1. Section 1.1 – Added referenced service methods.

19.2.2. Added Section 6.4 – Data acquisition, processing and reporting information.

19.2.3. Section 10.6.6.2 – removed :"The complete calibration curve for that compound may be used over the five point range in place of the mean RRF."

19.2.4. Section 10.8.1.1 – Clarified resolving power checks statement with updated software.

19.2.5. Section 11.2.3 - Revised paragraph and included conditions for Arizona regulatory samples.

19.2.6. Editorial comments

19.3. WS-ID-0007, Revision 3.9, Effective 01/08/2015

19.3.1. Changed copyright statement on cover page.

19.3.2. Section 10.7 – changed to, "Initial calibration verification standard (ICV). A second source calibration standard is analyzed following the initial calibration curve, prior to samples. Criteria are as follows: All native (unlabelled) compounds must be within ± 30% of expected value. IDA (labeled) compounds must meet the requirements of Table 9/9a for the VER."

19.3.3. Editorial changes.

19.4. WS-ID-0007, Revision 3.8, Effective 03/13/2015

19.4.1. Changed all references to DOD to be DOE inclusive – Sections 1.3 and 9.2.3.

19.4.2. Inserted Section 10.7, "Initial calibration verification standard (ICV): A second source calibration standard is analyzed following the initial calibration curve, prior to samples. Criteria are as follows: All native (unlabelled) compounds must be within ± 30% of expected value. IDA (labeled) compounds must meet the requirements of Table 9/9a for the VER. "

19.5. WS-ID-0007, Revision 3.8, Effective 03/13/2015

19.5.1. Section(s) 10.2.2 and 11.3.3 – Changed CS3 to CS4.

19.5.2. Removed Section 10.7.2.2 – "In either case, inject an instrument blank or method blank (see Section 9.3 above) prior to analyzing samples. Note that the CPSM/WDM does not define the beginning of a 12-hour sample acquisition period. The 12-hour sample acquisition period begins with the calibration verification standard (see below)."

19.5.3. Editorial changes.

19.6. WS-ID-0007, Revision 3.7, Effective 02/07/2014

19.6.1. Renamed Internal Standards (as per the method) to Isotope Dilution Analytes (as per our LIMS) to clarify for personnel.

19.6.2. Renamed Recovery Standards (as per the method) to Internal Standards (as per the LIMS) to clarify for personnel.

19.6.3. Renamed Cleanup Recovery Standards (as per the method) to Surrogate Standards (as per the LIMS) to clarify for personnel.

19.6.4. Editorial Revisions

19.6.5. The revision history prior to 2010 has been removed, and is available in earlier versions of this SOP.

19.7. WS-ID-0007, Revision 3.6, Effective 05/15/2012

19.7.1. Revised Table 10 to reflect correct mass/charge ratios and elemental composition of analytes.

19.7.2. Editorial revisions.

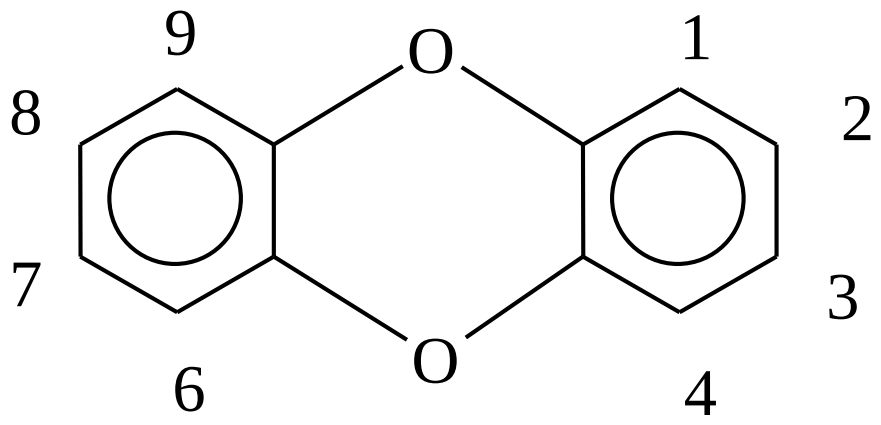
19.8. WS-ID-0007, Revision 3.5, Effective 04/15/2011

19.8.1. Section 11.2.4: Changed to - “Method Blank may be analyzed either after the CCV or after the solvent blank following CCV.

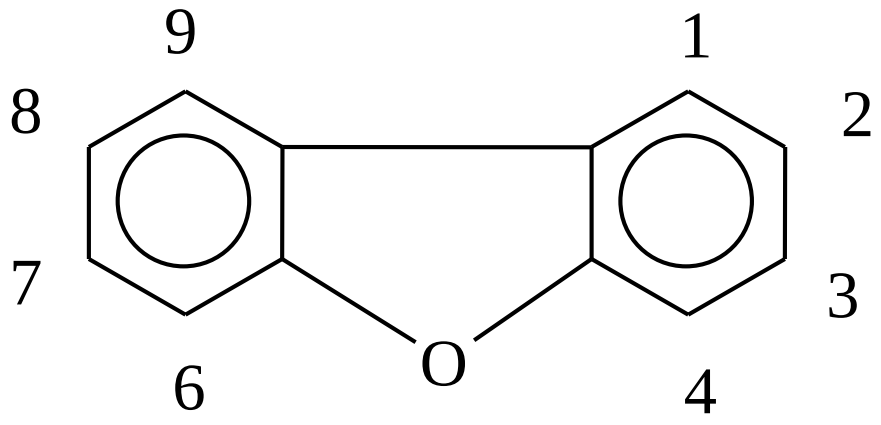
19.8.2. Deleted Section 17.1.4.

19.8.3. Editorial changes.

Figure 1
Structure of Dibenzodioxin and Dibenzofuran



Dibenzodioxin



Dibenzofuran

Table 1: Isomers of Chlorinated Dioxins and Furans as a Function of the Number of Chlorine Atoms

# of Chlorine Atoms	# of Dioxin Isomers	# of 2,3,7,8 Isomers	# of Furan Isomers	# of 2,3,7,8 Isomers
1	2	---	4	---
2	10	---	16	---
3	14	---	28	---
4	22	1	38	1
5	14	1	28	2
6	10	3	16	4
7	2	1	4	2
8	1	1	1	1
Total	**75**	**7**	**135**	**10**

Table 2: Theoretical Ion Abundance Ratios and QC Limits

# of Chlorine Atoms	Ion Type	Theoretical Ratio	Control Limits[1]	
			Lower	Upper
4[2]	M / M+2	0.77	0.65	0.89
5	M+2 / M+4	1.55	1.32	1.78
6	M+2 / M+4	1.24	1.05	1.43
6[3]	M / M+2	0.51	0.43	0.59
7[4]	M / M+2	0.44	0.37	0.51
7	M+2 / M+4	1.04	0.88	1.20
8	M+2 / M+4	0.89	0.76	1.02

(1) QC limits represent 15% windows around the theoretical ion abundance ratios.
(2) Does not apply to $^{37}Cl_4$-2,3,7,8-TCDD (Cleanup Recovery Standard).
(3) Used for $^{13}C_{12}$-HxCDF only.
(4) Used for $^{13}C_{12}$-HpCDF only.

Table 3: The Seventeen 2,3,7,8-Substituted PCDD and PCDF Congeners

PCDD	PCDF
2,3,7,8-TCDD(*)	2,3,7,8-TCDF(*)
1,2,3,7,8-PeCDD(*)	1,2,3,7,8-PeCDF(*)
1,2,3,6,7,8-HxCDD(*)	2,3,4,7,8-PeCDF(*)
1,2,3,4,7,8-HxCDD(*)	1,2,3,6,7,8-HxCDF(*)
1,2,3,7,8,9-HxCDD(+)	1,2,3,7,8,9-HxCDF(*)
1,2,3,4,6,7,8-HpCDD(*)	1,2,3,4,7,8-HxCDF(*)
1,2,3,4,5,6,7,8-OCDD(*)	2,3,4,6,7,8-HxCDF(*)
	1,2,3,4,6,7,8-HpCDF(*)
	1,2,3,4,7,8,9-HpCDF(*)
	1,2,3,4,5,6,7,8-OCDF

(*)The 13C-labeled analog is used as an internal standard.
(+)The 13C-labeled analog is used as a recovery standard.

Table 4: Concentration of Stock and Spiking Solutions Containing CDDS/CDDFs and Labeled Compounds (ng/mL = pg/µL)

Analyte	Type	Labeled Compound Stock Solution (ng/mL)	Labeled Compound Spiking Solution[a] (ng/mL)	PAR Stock Solution (ng/mL)	PAR Spiking Solution[b] (ng/mL)
Isotope Dilution Standards					
$^{13}C_{12}$-2,3,7,8-TCDD	IDA	100	2	--	--
$^{13}C_{12}$-1,2,3,7,8-PeCDD	IDA	100	2	--	--
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	IDA	100	2	--	--
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	IDA	100	2	--	--
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	IDA	100	2	--	--
$^{13}C_{12}$-OCDD	IDA	100	4	--	--
$^{13}C_{12}$-2,3,7,8-TCDF	IDA	100	2	--	--
$^{13}C_{12}$-1,2,3,7,8-PeCDF	IDA	100	2	--	--
$^{13}C_{12}$-2,3,4,7,8-PeCDF	IDA	100	2	--	--
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	IDA	100	2	--	--
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	IDA	100	2	--	--
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	IDA	100	2	--	--
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	IDA	100	2	--	--
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	IDA	100	2	--	--
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	IDA	100	2	--	--
Internal Standards					
$^{13}C_{12}$-1,2,3,4-TCDD	IS	200	100[d]	--	--
$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	IS	200	100[d]	--	--
Surrogate Standard					
$^{37}Cl_4$-2,3,7,8-TCDD	CRS	200	0.8[c]	--	--
Native Standard					
2,3,7,8-TCDD	NS	--	--	40	4
1,2,3,7,8-PeCDD	NS	--	--	200	20
1,2,3,4,7,8-HxCDD	NS	--	--	200	20
1,2,3,6,7,8-HxCDD	NS	--	--	200	20
1,2,3,7,8,9-HxCDD	NS	--	--	200	20
1,2,3,4,6,7,8-HpCDD	NS	--	--	200	20
OCDD	NS	--	--	400	40
2,3,7,8-TCDF	NS	--	--	40	4
1,2,3,7,8-PeCDF	NS	--	--	200	20
2,3,4,7,8-PeCDF	NS	--	--	200	20
1,2,3,4,7,8-HxCDF	NS	--	--	200	20
1,2,3,6,7,8-HxCDF	NS	--	--	200	20
2,3,4,6,7,8-HxCDF	NS	--	--	200	20
1,2,3,7,8,9-HxCDF	NS	--	--	200	20
1,2,3,4,6,7,8-HpCDF	NS	--	--	200	20
1,2,3,4,7,8,9-HpCDF	NS	--	--	200	20
OCDF	NS	--	--	400	40

(a) typical spike amount is 1.0mL

(b) typical spike amount is 50µL
(c) typical spike amount is 1.0mL
(d) typical spike amount is 20µL

Table 5: Concentration of CDD's/CDF's in Calibration and Calibration Verification Solutions

Compound	Concentration (ng/mL)				
	L2	L3	L4	L5	L6
Native CDDs and CDFs					
2,3,7,8-TCDD	0.5	2	10	40	200
2,3,7,8-TCDF	0.5	2	10	40	200
1,2,3,7,8-PeCDD	2.5	10	50	200	1000
1,2,3,7,8-PeCDF	2.5	10	50	200	1000
2,3,4,7,8-PeCDF	2.5	10	50	200	1000
1,2,3,4,7,8-HxCDD	2.5	10	50	200	1000
1,2,3,6,7,8-HxCDD	2.5	10	50	200	1000
1,2,3,7,8,9-HxCDD	2.5	10	50	200	1000
1,2,3,4,7,8-HxCDF	2.5	10	50	200	1000
1,2,3,6,7,8-HxCDF	2.5	10	50	200	1000
1,2,3,7,8,9-HxCDF	2.5	10	50	200	1000
2,3,4,6,7,8-HxCDF	2.5	10	50	200	1000
1,2,3,4,6,7,8-HpCDD	2.5	10	50	200	1000
1,2,3,4,6,7,8-HpCDF	2.5	10	50	200	1000
1,2,3,4,7,8,9-HpCDF	2.5	10	50	200	1000
OCDD	5.0	20	100	400	2000
OCDF	5.0	20	100	400	2000
Labeled CDDs and CDFs					
$^{13}C_{12}$-2,3,7,8-TCDD	100	100	100	100	100
$^{13}C_{12}$-2,3,7,8-TCDF	100	100	100	100	100
$^{13}C_{12}$-1,2,3,7,8-PeCDD	100	100	100	100	100
$^{13}C_{12}$-1,2,3,7,8-PeCDF	100	100	100	100	100
$^{13}C_{12}$-2,3,4,7,8-PeCDF	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100	100	100	100	100
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100	100	100	100	100
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100	100	100	100	100
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	100	100	100	100	100
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	100	100	100	100	100
$^{13}C_{12}$-OCDD	200	200	200	200	200
Surrogate Standard/ FS					
$^{37}Cl_4$-2,3,7,8-TCDD	0.5	2	10	40	200
Internal Standards					
$^{13}C_{12}$-1,2,3,4-TCDD	100	100	100	100	100
$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	100	100	100	100	100

Table 6: GC Retention Time Window Defining Test and Isomer Specificity Test Solution

DB-5 column GC retention-time window defining solution		
CDD/CDF	**First Eluter**	**Last Eluter**
TCDF	1,3,6,8-	1,2,8,9-
TCDD	1,3,6,8-	1,2,8,9-
PeCDF	1,3,4,6,8-	1,2,3,8,9-
PeCDD	1,2,4,7,9-	1,2,3,8,9-
HxCDF	1,2,3,4,6,8-	1,2,3,4,8,9-
HxCDD	1,2,4,6,7,9-	1,2,3,4,6,7-[(a)]
HpCDF	1,2,3,4,6,7,8-	1,2,3,4,7,8,9-
HpCDD	1,2,3,4,6,7,9-	1,2,3,4,6,7,8-
DB-5 Column TCDD Specificity Test Standard[(b)]		
	1,2,3,7+1,2,3,8-TCDD 2,3,7,8-TCDD 1,2,3,9-TCDD 1,2,3,4-TCDD	
DB-225 Column TCDF Isomer Specificity Test Standard[(c)]		
	2,3,4,7-TCDF 2,3,7,8-TCDF 1,2,3,9-TCDF	

(a) The 1,2,3,4,6,7-HxCDD isomer is not the last eluting isomer. 1,2,4,7,8,9-HxCDD is the last eluting HxCDD isomer and is present in the internal standard solution.
(b) Column performance criteria is met on the DB-5 column when the percent valleys between the 2,3,7,8-TCDD analyte and the closest eluting analytes are ≤25%.
(c) Column performance criteria is met on the DB-225 column when the percent valleys between the 2,3,7,8-TCDF analyte and the closest eluting analytes are ≤25%.

Table 7: Chlorinated Dibenzo-p-Dioxins and Furans Determined by Isotope Dilution and Internal Standard High Resolution Gas Chromatography (HRGC)/High Resolution Mass Spectrometry (HRMS)

CDDs/CDFs (1)	CAS registry	Labeled analog	CAS registry
2,3,7,8-TCDD	1746-01-6	$^{13}C_{12}$-2,3,7,8-TCDD	76523-40-5
		$^{37}Cl_4$-2,3,7,8-TCDD	85508-50-5
Total TCDD	41903-57-5		
2,3,7,8-TCDF	51207-31-9	$^{13}C_{12}$-2,3,7,8-TCDF	89059-46-1
Total TCDF	55722-27-5		
1,2,3,7,8-PeCDD	40321-76-4	$^{13}C_{12}$-1,2,3,7,8-PeCDD	109719-79-1
Total PeCDD	36088-22-9		
1,2,3,7,8-PeCDF	57117-41-6	$^{13}C_{12}$-1,2,3,7,8-PeCDF	109719-77-9
2,3,4,7,8-PeCDF	57117-31-4	$^{13}C_{12}$-2,3,4,7,8-PeCDF	116843-02-8
Total PeCDF	30402-15-4		
1,2,3,4,7,8-HxCDD	39227-28-6	$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	109719-80-4
1,2,3,6,7,8-HxCDD	57653-85-7	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	109719-81-5
1,2,3,7,8,9-HxCDD	19408-74-3	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	109719-82-6
Total HxCDD	34465-46-8		
1,2,3,4,7,8-HxCDF	70648-26-9	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	114423-98-2
1,2,3,6,7,8-HxCDF	57117-44-9	$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	116843-03-9
1,2,3,7,8,9-HxCDF	72918-21-9	$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	116843-04-0
2,3,4,6,7,8-HxCDF	60851-34-5	$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	116843-05-1
Total HxCDF	55684-94-1		
1,2,3,4,6,7,8-HpCDD	35822-46-9	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	109719-83-7
Total HpCDD	37871-00-4		
1,2,3,4,6,7,8-HpCDF	67562-39-4	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	109719-84-8
1,2,3,4,7,8,9-HpCDF	55673-89-7	$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	109719-94-0
Total HpCDF			
OCDD	3268-87-9	$^{13}C_{12}$-OCDD	114423-97-1
OCDF	39001-02-0	Not Used	

(1) Chlorinated dibenzo-p-dioxins and chlorinated dibenzofurans

TCDD = Tetrachlorodibenzo-p-dioxins
TCDF = Tetrachlorodibenzofurans
PeCDD = Pentachlorodibenzo-p-dioxins
PeCDF = Pentachlorodibenzofurans
HxCDD = Hexachlorodibenzo-p-dioxins
HxCDF = Hexachlorodibenzofurans
HpCDD = Heptaachlorodibenzo-p-dioxins
HpCDF = Heptachlorodibenzofurans
OCDD = Octachlorodibenzo-p-dioxins
OCDF = Octachlorodibenzofurans

Table 8: Retention Time References, Quantitation References, Relative Retention Times, and Minimum Levels for PCDDs and PCDFs. (pg/L = ppq, ng/kg = ppt, and pg/uL = ppb)

			Minimum Level (1)		
CDD/CDF	**Retention Time and Quantitation Reference**	**Relative Retention Time**	**Water (pg/L)**	**Solid (ng/kg)**	**Extract (pg/µL)**
Compounds using $^{13}C_{12}$-1,2,3,4-TCDD as the Injection Internal Standard (Recovery Standard)					
2,3,7,8-TCDF	$^{13}C_{12}$-2,3,7,8-TCDF	0.999-1.003	10	1	0.5
2,3,7,8-TCDD	$^{13}C_{12}$-2,3,7,8-TCDD	0.999-1.002	10	1	0.5
1,2,3,7,8-PeCDF	$^{13}C_{12}$-1,2,3,7,8-PeCDF	0.999-1.002	50	5	2.5
2,3,4,7,8-PeCDF	$^{13}C_{12}$-2,3,4,7,8-PeCDF	0.999-1.002	50	5	2.5
1,2,3,7,8-PeCDD	$^{13}C_{12}$-1,2,3,7,8-PeCDD	0.999-1.002	50	5	2.5
$^{13}C_{12}$-2,3,7,8-TCDF	$^{13}C_{12}$-1,2,3,4-TCDD	0.923-1.103	--	--	--
$^{13}C_{12}$-2,3,7,8-TCDD	$^{13}C_{12}$-1,2,3,4-TCDD	0.976-1.043	--	--	--
$^{37}Cl_4$-2,3,7,8-TCDD	$^{13}C_{12}$-1,2,3,4-TCDD	0.989-1.052	--	--	--
$^{13}C_{12}$-1,2,3,7,8-PeCDF	$^{13}C_{12}$-1,2,3,4-TCDD	1.000-1.425	--	--	--
$^{13}C_{12}$-2,3,4,7,8-PeCDF	$^{13}C_{12}$-1,2,3,4-TCDD	1.001-1.526	--	--	--
$^{13}C_{12}$-1,2,3,7,8-PeCDD	$^{13}C_{12}$-1,2,3,4-TCDD	1.000-1.567	--	--	--
Compounds using $^{13}C_{12}$-1,2,3,7,8,9-HxCDD as the Injection Internal Standard (Recovery Standard)					
1,2,3,4,7,8-HxCDF	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	0.999-1.001	50	5	2.5
1,2,3,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	1.000-1.005	50	5	2.5
1,2,3,7,8,9-HxCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	0.999-1.001	50	5	2.5
2,3,4,6,7,8-HxCDF	$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	0.999-1.001	50	5	2.5
1,2,3,4,7,8-HxCDD	$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	0.999-1.001	50	5	2.5
1,2,3,6,7,8-HxCDD	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	0.999-1.004	50	5	2.5
1,2,3,7,8,9-HxCDD	(2)	1.000-1.019	50	5	2.5
1,2,3,4,6,7,8-HpCDF	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	0.999-1.001	50	5	2.5
1,2,3,4,7,8,9-HpCDF	$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	0.999-1.001	50	5	2.5
1,2,3,4,6,7,8-HpCDD	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	0.999-1.001	50	5	2.5
OCDF	$^{13}C_{12}$- OCDD	0.999-1.001	100	10	5.0
OCDD	$^{13}C_{12}$- OCDD	0.999-1.001	100	10	5.0
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	$^{13}C_{12}$-1,2,3, 7,8,9-HxCDD	(3)	--	--	--
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3, 7,8,9-HxCDD	0.949-0.975	--	--	--
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	$^{13}C_{12}$-1,2,3, 7,8,9-HxCDD	0.977-1.047	--	--	--
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3, 7,8,9-HxCDD	0.959-1.021	--	--	--
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	$^{13}C_{12}$-1,2,3, 7,8,9-HxCDD	0.977-1.000	--	--	--
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	$^{13}C_{12}$-1,2,3, 7,8,9-HxCDD	0.981-1.003	--	--	--
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	$^{13}C_{12}$-1,2,3, 7,8,9-HxCDD	1.043-1.085	--	--	--
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	$^{13}C_{12}$-1,2,3, 7,8,9-HxCDD	1.057-1.151	--	--	--
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	$^{13}C_{12}$-1,2,3, 7,8,9-HxCDD	1.086-1.110	--	--	--
$^{13}C_{12}$- OCDD	$^{13}C_{12}$-1,2,3, 7,8,9-HxCDD	1.032-1.311	--	--	--

(1) The Minimum Level (ML) for each analyte is defined as the level at which the entire analytical system must give a recognizable signal and acceptance calibration point. It is equivalent to the concentration of the lowest calibration standard, assuming that all method-specified sample weights, volumes, and cleanup procedures have been employed.

(2) The retention time reference for 1,2,3,7,8,9-HxCDD is $^{13}C_{12}$-1,2,3,6,7,8-HxCDD, and 1,2,3,7,8,9-HxCDD is quantified using the averaged responses for $^{13}C_{12}$-1,2,3,4,7,8-HxCDD and $^{13}C_{12}$-1,2,3,6,7,8-HxCDD.

(3) Method 1613 has many typographical errors within this table. Most have been corrected. This RRT is not present in Method 1613, but the internal standard is clearly identifiable and there is no adverse impact for this error.

Table 9: Acceptance Criteria for Performance Tests (Note that ng/mL = pg/µL) (1)

CDD/CDF	Test Conc. (ng/mL)	IPR (2)(3)		OPR (LCS) (ng/mL)	VER (L4) (ng/mL)
		S (ng/mL)	X (ng/mL)		
2,3,7,8-TCDD	10	2.8	8.3-12.9	6.7-15.8	7.8-12.9
2,3,7,8-TCDF	10	2.0	8.7-13.7	7.5-15.8	8.4-12.0
1,2,3,7,8-PeCDD	50	7.5	38-66	35-71	39-65
1,2,3,7,8-PeCDF	50	7.5	43-62	40-67	41-60
2,3,4,7,8-PeCDF	50	8.6	36-75	34-80	41-61
1,2,3,4,7,8-HxCDD	50	9.4	39-76	35-82	39-64
1,2,3,6,7,8-HxCDD	50	7.7	42-62	38-67	39-64
1,2,3,7,8,9-HxCDD	50	11.1	37-71	32-81	41-61
1,2,3,4,7,8-HxCDF	50	8.7	41-59	36-67	45-55
1,2,3,6,7,8-HxCDF	50	6.7	46-60	42-65	44-57
1,2,3,7,8,9-HxCDF	50	6.4	42-61	39-65	45-56
2,3,4,6,7,8-HxCDF	50	7.4	37-74	35-78	44-57
1,2,3,4,6,7,8-HpCDD	50	7.7	38-65	35-70	43-58
1,2,3,4,6,7,8-HpCDF	50	6.3	45-56	41-61	45-55
1,2,3,4,7,8,9-HpCDF	50	8.1	43-63	39-69	43-58
OCDD	100	19	89-127	78-144	79-126
OCDF	100	27	74-146	63-170	63-159
$^{13}C_{12}$-2,3,7,8-TCDD	100	37	28-134	20-175	82-121
$^{13}C_{12}$-2,3,7,8-TCDF	100	35	31-113	22-152	71-140
$^{13}C_{12}$-1,2,3,7,8-PeCDD	100	39	27-184	21-227	62-160
$^{13}C_{12}$-1,2,3,7,8-PeCDF	100	34	27-156	21-192	76-130
$^{13}C_{12}$-2,3,4,7,8-PeCDF	100	38	16-279	13-328	77-130
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100	41	29-147	21-193	85-117
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	100	38	34-122	25-163	85-118
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100	43	27-152	19-202	76-131
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100	35	30-122	21-159	70-143
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	100	40	24-157	17-205	74-135
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	100	37	29-136	22-176	73-137
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100	35	34-129	26-166	72-138
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100	41	32-110	21-158	78-129
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDD	100	40	28-141	20-186	77-129
$^{13}C_{12}$- OCDD	200	95	41-276	26-397	96-415
$^{37}Cl_4$-2,3,7,8-TCDD	10	3.6	3.9-15.4	3.7-15.8	7.9-12.7

(1) All specifications are given as concentration in the final extract, assuming a 20□L final volume.
(2) s = standard deviation of the concentration.
(3) X = average concentration.

Table 9.1: Acceptance Criteria for Performance Tests (Tetras – Only Analysis) Note that ng/mL = pg/µL

CDD/CDF	Test Conc. (ng/mL)	IPR [2][3]		OPR (LCS) (ng/mL)	VER (L4) (ng/mL)
		S (ng/mL)	X (ng/mL)		
2,3,7,8-TCDD	10	2.7	8.7 – 12.4	7.3 – 14.6	8.2 – 12.3
2,3,7,8-TCDF	10	2.0	9.1 – 13.1	8.0 -14.7	8.6 - 11.6
$^{13}C_{12}$-2,3,7,8-TCDD	100	35	32 – 115	25 – 141	85 – 117
$^{13}C_{12}$-2,3,7,8-TCDF	100	34	35 – 99	26 - 126	76 - 134
$^{37}Cl_4$-2,3,7,8-TCDD	10	3.4	4.5 – 13.4	3.7-15.8	8..3 – 12.1

Table 10: Descriptors, Exact m/z's, m/z types, and Elemental Compositions of the CDDs and CDFs

Descriptor	Exact m/z [1]	m/z Type	Elemental Composition	Substance [2]
1	292.9825	QC	C_7F_{11}	PFK
	303.9016	M	$C_{12}H_4{}^{35}Cl_4O$	TCDF
	305.8987	M+2	$C_{12}H_4{}^{35}Cl_3{}^{37}ClO$	TCDF
	315.9419	M	$^{13}C_{12}H_4{}^{35}Cl_4O$	TCDF [3]
	317.9389	M+2	$^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO$	TCDF [3]
	319.8965	M	$C_{12}H_4{}^{35}Cl_4O_2$	TCDD
	321.8936	M+2	$C_{12}H_4{}^{35}Cl_3{}^{37}ClO_2$	TCDD
	327.8847	M	$C_{12}H_4{}^{37}Cl_4O_2$	TCDD [4]
	330.9792	Lock	C_7F_{13}	PFK
	331.9368	M	$^{13}C_{12}H_4{}^{35}Cl_4O_2$	TCDD [3]
	333.9339	M+2	$^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO_2$	TCDD [3]
	339.8597	M+2	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF
	341.8567	M+4	$C_{12}H_3{}^{35}Cl_3{}^{37}ClO$	PeCDF
	375.8364	M+2	$C_{12}H_4{}^{35}Cl_5{}^{37}ClO$	HxCDPE
	409.7974	M+2	$C_{12}H_3{}^{35}Cl_6{}^{37}ClO$	HpCDPE
2	330.9792	QC	C_7F_{13}	PFK
	339.8597	M+2	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF
	341.8567	M+4	$C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$	PeCDF
	342.9792	Lock	C_8F_{12}	PFK
	351.9000	M+2	$^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF
	353.8970	M+4	$^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF [3]
	354.9792	Lock	C_9F_{13}	PFK
	355.8546	M+2	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$	PeCDD
	357.8516	M+4	$C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$	PeCDD
	366.9793	QC	C_9F_{13}	PFK
	367.8949	M+2	$^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$	PeCDD [3]
	369.8919	M+4	$^{13}C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$	PeCDD [3]
	409.7974	M+2	$C_{12}H_3{}^{35}Cl_6{}^{37}ClO$	HpCDPE
3	373.8208	M+2	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO$	HxCDF
	375.8178	M+4	$C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O$	HxCDF
	380.9760	Lock	C_8F_{15}	PFK
	383.8639	M	$^{13}C_{12}H_2{}^{35}Cl_6O$	HxCDF [3]
	385.8610	M+2	$^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO$	HxCDF [3]
	389.8157	M+2	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$	HxCDD
	391.8127	M+4	$C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$	HxCDD
	392.9760	Lock	C_9F_{15}	PFK
	401.8559	M+2	$^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$	HxCDD [3]
	403.8529	M+4	$^{13}C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$	HxCDD [3]
	430.9728	QC	C_9F_{17}	PFK
	445.7550	M+4	$C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2O$	OCDPE

Descriptor	Exact m/z [1]	m/z Type	Elemental Composition	Substance [2]
4	392.9760	QC	C_9F_{15}	PFK
	407.7818	M+2	$C_{12}H^{35}Cl_6{}^{37}ClO$	HpCDF
	409.7789	M+4	$C_{12}H^{35}Cl_5{}^{37}Cl_2O$	HpCDF
	417.8253	M	$^{13}C_{12}H^{35}Cl_7O$	HpCDF [3]
	419.8220	M+2	$^{13}C_{12}H^{35}Cl_6{}^{37}ClO$	HpCDF [3]
	423.7766	M+2	$C_{12}H^{35}Cl_6{}^{37}ClO_2$	HpCDD
	425.7737	M+4	$C_{12}H^{35}Cl_5{}^{37}Cl_2O_2$	HpCDD
	430.9729	Lock	C_9F_{17}	PFK
	435.8169	M+2	$^{13}C_{12}H^{35}Cl_6{}^{37}ClO_2$	HpCDD [3]
	437.8140	M+4	$^{13}C_{12}H^{35}Cl_5{}^{37}CL_2O_2$	HpCDD [3]
	479.7165	M+4	$C_{12}H^{35}Cl_7{}^{37}Cl_2O$	NCDPE
5	392.9760	QC	C_9F_{15}	PFK
	441.7428	M+2	$C_{12}{}^{35}Cl_7{}^{37}ClO$	OCDF
	442.9728	Lock	$C_{10}F_{17}$	PFK
	443.7399	M+4	$C_{12}{}^{35}Cl_6{}^{37}Cl_2O$	OCDF
	457.7377	M+2	$C_{12}{}^{35}Cl_7{}^{37}ClO_2$	OCDD
	459.7348	M+4	$C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$	OCDD
	469.7779	M+2	$^{13}C1_2{}^{35}Cl_7{}^{37}ClO_2$	OCDD [3]
	471.7750	M+4	$^{13}C1_2{}^{35}Cl_6{}^{37}Cl_2O_2$	OCDD [3]
	479.7165	M+4	$C_{12}Cl_8{}^{37}Cl_2O$	NCDPE
	513.6775	M+4	$^{13}C_{12}{}^{35}Cl_8{}^{37}Cl_2O$	DCDPE

(1) Nuclidic masses used:

H	=	1.007825
O	=	15.994915
C	=	12.00000
^{13}C	=	13.003355
^{35}Cl	=	34.968853
^{37}Cl	=	36.965903
F	=	18.9984

(2) Nomenclature

TCDD = Tetrachlorodibenzo-p-dioxin
PeCDD = Pentachlorodibenzo-p-dioxin
HxCDD = Hexachlorodibenzo-p-dioxin
HpCDD = Heptachlorodibenzo-p-dioxin
OCDD = Octachlorodibenzo-p-dioxin
HxCDPE = Hexachlorodiphenyl ether
HpCDPE = Heptachlorodiphenyl ether
NCDPE = Nonachlorodiphenyl ether
OCDPE = Octachlorodiphenyl ether
DCDPE = Decachlorodiphenyl ether
TCDF = Tetrachlorodibenzofuran
PeCDF = Pentachlorodibenzofuran
HxCDF = Hexachlorodibenzofuran

HpCDF = Heptachlorodibenzofuran
OCDF = Octachlorodibenzofuran
PFK = Perfluorokerosene

(3) Labeled compound
(4) There is only one m/z for $^{37}Cl_4$-2,3,7,8-TCDD (Cleanup Recovery Standard)

Title: Preparation of Samples for Analysis of Polychlorinated Dioxins and Furans for Analysis HRGC/HRMS

[Methods 8290, 8290A & TO-9A]

Approvals (Signature/Date):

Robert Hrabak 1/26/17 Technical Manager Date	Joe Schairer 1/27/17 Health & Safety Manager / Coordinator Date
Lisa Stafford 1/26/2017 Quality Assurance Manager Date	Crystal Pollock 1.30.17 Laboratory Director Date

1. SCOPE AND APPLICATION

1.1. This method provides procedures for the preparation of samples prior to the analysis of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD), polychlorinated dibenzo-p-dioxins (tetra- through octachlorinated homologs; PCDDs), and polychlorinated dibenzofurans (tetra- through octachlorinated homologs; PCDFs) in a variety of environmental matrices at part-per-trillion (ppt) concentrations by SW 846 Method 8290. The analytical method calls for the use of high-resolution gas chromatography and high-resolution mass spectrometry (HRGC/HRMS) on purified sample extracts. Refer to Table 1 for the list of analytes. Analysis is by SOP WS-ID-0005.

1.2. The sensitivity of this method is dependent upon the level of interferences within a given matrix.

1.3. This method is designed for use by analysts who are experienced with residue analysis.

1.4. Samples containing concentrations of specific congeners (PCDDs and PCDFs) that are greater than the calibration limit should be analyzed by a protocol designed for such concentrations, such as 8280A/B.

2. SUMMARY OF METHOD

2.1. This procedure uses matrix-specific extraction and analyte-specific cleanup techniques.

2.2. A specified amount (see Table 1) of soil, sediment, fly ash, water, leachate, sludge (including paper pulp), still-bottom, fuel oil, chemical reactor residue, air sample (QFF, PUF or XAD media) or fish tissue, is spiked with a solution containing specified amounts of each of nine isotopically (^{13}C) labeled PCDDs/PCDFs listed in Table 2. The sample is then extracted according to a matrix-specified extraction procedure. The extraction procedures are: a) toluene Soxhlet (or equivalent), Soxtherm, or Microwave extraction, for soil, sediment, fly ash samples, aqueous sludges, and solid air matrices (XAD, QFF, PUF); b) methylene chloride liquid-liquid extraction or solid phase extraction for water samples; c) dilution of a small sample aliquot in solvent for wastes/chemical products; and d) toluene (or hexane/methylene chloride) Soxhlet (or equivalent), Soxtherm, or Microwave extraction for fish tissue. This method can also use solid phase extraction (SPE); however, Test America Sacramento is not currently certified for its use.

2.3. If interferences are present, extracts may be cleaned as described below. The extracts are submitted to an acid and/or base washing treatment and dried. Following a solvent exchange step, the residue is cleaned up by column chromatography on acid/base silica, acid alumina and carbon on silica. The preparation of the final extract for HRGC/HRMS analysis is accomplished by adding 20 µL of a tetradecane solution containing 100 pg/µL of each of the two recovery standards $^{13}C_{12}$-1,2,3,4-TCDD and

$^{13}C_{12}$ -1,2,3,7,8,9-HxCDD (Table 2) to the concentrated eluate. The former is used to determine the percent recoveries of tetra- and penta-chlorinated PCDD/PCDF isotope dilution analytes while the latter is used for the determination of hexa-, hepta- and octa-chlorinated PCDD/PCDF isotope dilution analyte percent recoveries. Upon client approval, less final volume can be used to decrease detection limit and more final volume can be used to decrease severe interferences.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

3.3. Isotope Dilution Analyte (IDA): An isotope dilution analyte is a ^{13}C-labeled analog of a congener chosen from the compounds listed in Table 2. Isotope dilution analytes are added to all samples including method blanks and quality control samples before extraction, and they are used to quantitate the concentration of the analytes. Nine isotope dilution analytes are used in this method. There is one for each of the dioxin and furan homologs (except for OCDF) with the degree of chlorination ranging from four to eight. Additional isotope dilution analytes may be added to act as retention time references, but they are not used for quantitation.

3.4. Internal Standard (IS) (named "recovery standard" in the reference methods): Two internal are used to determine the percent recoveries for the isotope dilution analytes. The $^{13}C_{12}$-1,2,3,4-TCDD is used to measure the percent recoveries of the tetra- and pentachlorinated isotope dilution analytes while $^{13}C_{12}$-1,2,3,7,8,9-HxCDD is used to determine the recovery of the hexa-hepta- and octachlorinated isotope dilution analytes. $^{13}C_{12}$-1,2,3,7,8,9-HxCDD also acts as a retention time reference for the unlabeled analog present in sample extracts. They are added to the final sample extract before HRGC/HRMS instrument analysis.

3.5. Surrogate Standard (SU) (named "cleanup recovery standard" in the reference methods): A $^{37}Cl_4$-2,3,7,8-TCDD analog that is added to each sample following extraction to measure the efficiency of the cleanup process.

3.6. Target Analyte (TA) (named "unlabeled analytes" or "native spike" in the reference methods): The seventeen target analytes are listed in Table 3. The target analytes are added to the LCS/LCSD and MS/MSD (when prepared).

4. INTERFERENCES

4.1. Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts or elevated baselines that may cause misinterpretation of the chromatographic

data. All of these materials must be demonstrated to be free from interferents under the conditions of analysis by running laboratory method blanks. Analysts shall not use PVC gloves.

4.2. The use of high-purity reagents and solvents helps minimize interference problems. Purification of solvents by distillation in all-glass systems may be necessary.

4.3. Proper cleaning of glassware is extremely important because glassware may not only contaminate the samples, but may also remove the analytes of interest by adsorption on the glassware surface.

4.3.1. Glassware should be rinsed with solvent and washed with a detergent solution as soon after use as is practical. Sonication of glassware containing a detergent solution for approximately 30 seconds may aid in cleaning. Glassware with removable parts, particularly separatory funnels with Teflon stopcocks, must be disassembled prior to detergent washing.

4.3.2. After detergent washing, glassware should be rinsed with acetone, toluene, hexane, and then methylene chloride.

4.3.3. Do not kiln reusable glassware in an oven as a routine part of cleaning. Kilning may be warranted after particularly dirty samples are encountered, but should be minimized, as repeated kilning of glassware may cause the formation of active sites on the glass surface that will irreversibly adsorb PCDDs/ PCDFs.

4.3.4. Immediately prior to use, Soxhlet (or equivalent) extraction glassware should be pre-extracted with toluene for a minimum of 3 hours. Note: Accelerated extractors such as the Soxtherm can use a shorter cleaning cycle which exhibits subsequent extractions free of cross contamination and interferences.

Note: Re-use of glassware should be minimized to avoid the risk of contamination. All glassware that is re-used must be scrupulously cleaned as soon as possible after use, applying the following procedure:

4.4. Interferences co-extracted from samples will vary considerably from source to source, depending on the diversity of the site being sampled. Interfering compounds may be present at concentrations several orders of magnitude higher than the PCDDs and PCDFs. The most frequently encountered interferences are chlorinated-biphenyls, methoxy biphenyls, hydroxy biphenyl ethers, benzyl phenyl ethers, polynuclear aromatics, and pesticides. Because very low levels of PCDDs and PCDFs are measured by this method, the elimination of interferences is essential. The cleanup steps given in Sections 11.12 thru 11.16 can be used to reduce or eliminate these interferences.

4.4.1. If South Carolina samples show diphenyl ethers at levels that could contribute to positive furan hits, a subsequent clean-up to remove them must be performed.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) 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-toes, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. Hearing protection must be worn when using mechanical systems to grind fish, tissue, or paper/pulp samples.

5.1.2. Finely divided dry soils contaminated with PCDDs and PCDFs are particularly hazardous because of the potential for inhalation and ingestion. Such samples are to be processed in a confined environment, such as a hood or a glove box.

5.1.3. Assembly and disassembly of glassware creates a risk of breakage and cuts. All staff members shall wear Kevlar or MAPA blue latex cut-resistant gloves over chemically resistant gloves when assembling and disassembling glassware.

5.1.4. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex and vinyl gloves provide no protection against most of the organic solvents used in this method. Nitrile or similar gloves must be used. Latex gloves may be used for methanol.

5.1.5. Exposure to chemicals must be maintained as low as reasonably achievable, therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.6. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are

in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.1.7. The use of separatory funnels to extract aqueous samples with methylene chloride creates excessive pressure very rapidly. The use of separatory funnels during the partition and back extraction of sample extracts can also create excessive pressure. Initial venting should be done immediately after the sample container has been sealed and inverted. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, and a face shield must be worn over safety glasses or goggles when it is performed. Alternately, the extraction can be performed behind a closed fume hood sash on a mechanical shaker.

5.1.8. When Dean-Stark/Soxhlet clean-ups or extractions are performed overnight or unattended, special precautions must be taken. Open the chiller valves to the system about 15 minutes before the heating elements are turned on, and check every condenser to ensure that it is cold and functioning properly before turning the heating elements on. Check every condenser again about 15 minutes after turning the heating elements on to ensure that they are still cold and functioning properly. If the system is left operating overnight or unattended for an extended period, the first chemist to come back into the lab must again check every condenser to ensure that it is still cold and functioning properly.

5.1.9. Microwave ovens used for extraction or digestion of samples can create elevated pressure in the extraction/digestion containers. Microwaves used for these processes must be equipped with an automated vapor sensor and shutoff system that automatically shuts down the digestion/extraction process when vapor pressure reaches pre-specific levels. Users must follow procedures in the microwave operator's manual to ensure that the vapor sensors are functional and working properly prior to starting each extraction/digestion batch.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Benzene	Flammable Toxic Carcinogen	PEL: 1 ppm TWA ; 5 ppm 15 MIN. STEL	Causes skin irritation. Toxic if absorbed through skin. Causes severe eye irritation. Toxic if inhaled. Vapor or mist causes irritation to mucous membranes and upper respiratory tract. Exposure can cause narcotic effect. Inhalation at high concentrations may have an initial stimulatory effect on the central nervous system characterized by exhilaration, nervous excitation and/or giddiness, depression, drowsiness or fatigue. Victim may experience tightness in the chest, breathlessness, and loss of consciousness.
Cyclohexane	Flammable Irritant	300 ppm TWA	Inhalation of vapors causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. High concentrations have a narcotic effect.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Isooctane	Flammable Irritant	None established	Inhalation of vapors may cause nausea, headache, dizziness, loss of consciousness, and irritation to upper respiratory tract, pain in throat and nose, coughing, wheezing, shortness of breath.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Sulfuric Acid (1)	Corrosive Oxidizer Dehydra-dator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Tetradecane	Irritant	None established	Inhalation of vapors may cause difficulty breathing, headache, intoxication and central nervous system damage.
Toluene	Flammable Poison Irritant	200 ppm-TWA 300 ppm-Ceiling	Inhalation may cause irritation of the upper respiratory tract. Symptoms of overexposure may include fatigue, confusion, headache, dizziness and drowsiness. Peculiar skin sensations (e. g. pins and needles) or numbness may be produced. Causes severe eye and skin irritation with redness and pain. May be absorbed through the skin.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid (3-0-1)	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

The following list of items does not necessarily constitute an exhaustive compendium of the equipment needed for this analytical method.

6.1. Nitrogen evaporation apparatus with variable flow rate.

6.2. Balances capable of accurately weighing to 0.01 g and 0.0001 g.

6.3. Centrifuge.

6.4. Water bath, equipped with concentric ring covers and capable of maintaining temperature control within ± 2ºC.

6.5. Stainless steel or glass containers large enough to hold contents of one-pint sample containers.

6.6. Drying oven.

6.7. Stainless steel spoons and spatulas.

6.8. Pipettes, disposable, Pasteur, 150 mm long x 5 mm ID.

6.9. Pipettes, disposable, serological, 10 mL, for the preparation of the carbon column specified in Section 7.1.

6.10. Reacti-vial, 2 mL, silanized clear glass.

6.11. Stainless steel meat grinder with a 3- to 5-mm hole size inner plate.

6.12. Separatory funnels, 250 mL.

6.13. Separatory funnels, 1000 mL.

6.14. Teflon® boiling chips (or equivalent) washed with hexane before use.

6.15. Chromatographic column, glass, 300 mm x 10.5 mm, fitted with Teflon® stopcock.

6.16. Adapters for concentrator tubes.

6.17. Glass fiber filters, Whatman GF-D, GF-F, GMF150, or equivalent.

6.18. Solid phase extraction discs, 3M 90mm C18, or equivalent.

6.19. 30x100mm glass fiber thimble or equivalent.

6.20. Dean-Stark trap, 5 or 10 mL, with T-joints, condenser and 125 mL flask.

6.21. All-glass Soxhlet apparatus.

6.22. 500 mL round bottom flask.

6.23. Milestone microwave extraction apparatus (or equivalent)

6.23.1. Automated microwave extractor unit

6.23.2. Plastic extraction vessels with Teflon sample chambers and Teflon pressure release gaskets

6.23.3. 24 position carousel

6.24. Soxtherm extraction apparatus (or equivalent), including glass thimble holders, glass beakers, and gaskets.

6.25. Glass funnels, sized to hold 170 mL of liquid.

6.26. Desiccator.

6.27. Turbo evaporator

6.28. Rotary evaporator with a temperature controlled water bath.

6.29. High speed tissue homogenizer, equipped with an EN-8 probe or equivalent.

6.30. Glass wool, extracted with methylene chloride, dried and stored in a clean glass jar.

6.31. Vacuum extraction device for solid phase extraction, 1 Liter glass funnel with 90mm filter disc holder with a vacuum source, Kontes or equivalent.

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Sulfuric acid, concentrated, ACS grade, specific gravity 1.84.

7.1.2. Distilled water demonstrated to be free of interferents

7.1.3. 1 N HCl, ACS Grade

7.1.4. 10 N NaOH, certified (Fisher Scientific S5255 or equivalent)

7.1.5. Silica gel – 70-230 mesh (Fischer Scientific , P/N 130714A or equivalent) -- Activate at least 12 hours at 185-195°C before use. Store at 125-145°C in covered flask or bottle.

7.1.6. Sodium Sulfate, granular, anhydrous, ACS grade.

7.1.7. Vegetable Oil (for tissue extraction only), Mazola or other suitable oil, demonstrated to be free of interferences.

7.1.8. Diatomaceaous earth – Hydromatrix (Agilent P/N 198004 (4kg) or 198005 (1kg), or equivalent.

7.2. Solvents

7.2.1. High-purity, distilled-in-glass or highest available purity: Methylene chloride, hexane, methanol, tetradecane, isooctane, toluene, cyclohexane, benzene, and acetone.

7.3. Column Chromatography Reagents

7.3.1. Acid Alumina - ICN or equivalent, activated as necessary.

7.3.2. Basic Alumina - ICN or equivalent. No activation required.

7.3.3. Granular carbon/silica gel - Mix 3.6 g granular carbon and 16.4 g activated silica gel; (alternatively, prepare carbon/silica gel (5%/95%); i.e., combine 5 g precleaned carbon with 95 g silica gel). Store at room temperature in a Teflon ® lined covered jar. The first LCS prepared with a new batch of column packing material is the quality control check of the packing materials. Refer to historical control limits before accepting the new batch of material.

7.3.4. 44% H_2SO_4 /silica gel - Mix 24 mL conc. H_2SO_4 and 56 g activated silica gel. Stir and shake until free flowing. Store at room temperature.

7.4. Acid Alumina Activity Assessment

Alumina activity may vary with the matrix or environmental conditions. Monitor Isotope Dilution Analyte Standard (IDA) and Surrogate Standard (SU) recoveries in extract analysis. Low recoveries of SU may indicate loss of alumina activity. Assess

stability of alumina activity and apply corrective action as appropriate (reactivate and reprofile).

Note: a column profile should be done to show elution of all 2,3,7,8 substituted analogs so problems can be readily identified.

7.4.1. Profile each vendor lot of activated alumina as corrective action for low internal standard and CRS recoveries dictate. If necessary, proceed as follows:

7.4.1.1. Set up and label 3 acid alumina columns.

7.4.1.2. Pre-rinse with 20 mL hexane.

7.4.1.3. Add 2 mL hexane spiked with isotope dilution analytes (Section 7.8) and natives (spike amounts equivalent to those for LCS) with 2X2 mL hexane rinse of fractions.

7.4.1.4. Elute each column with 20 mL hexane. Collect and label these fractions.

7.4.1.5. Elute each column with 5 x10 mL methylene chloride/hexane at the appropriate v/v percent. Collect and label these fractions separately.

7.4.1.6. Elute each column with 10 mL of 100% methylene chloride. Collect and label these fractions. Reduce all fractions to final volume and add recovery standard.

7.4.2. Review data and select an elution scheme. Group the fraction from each solvent system as follows:

7.4.2.1. Pre-analyte fraction - consists of all eluent prior to elution of first target analytes.

7.4.2.2. Analyte fraction - consists of all that contain detectable levels of target analytes.

7.4.2.3. Post-analyte fraction - consists of all eluents after elution of the last target analyte.

7.4.3. Select the solvent system which best meets the following two conditions:

7.4.3.1. Pre-analyte fraction consists of 20mL hexane and no more than 20 mL mixed solvent.

7.4.3.2. Analyte fraction consists of no more than 20mL of mixed solvent and contains greater than 90% of all target analytes and greater than 80% of all internal standards.

7.4.4. After selection of the appropriate solvent system and fractionation pattern, perform triplicate acid alumina cleanups on spiked hexane to ensure reproducibility of the fractionation pattern. Document each elution scheme.

7.4.5. Each subsequent batch of acid alumina used in the lab (from the same vendor lot) must be checked for stable activity.

7.5. All daily isotope dilution analyte (IDA) standard, daily surrogate standards (SU), internal standard (IS), and daily spiking solutions (TA) are stable for one year from preparation. After 1 year, solutions may be re-verified. The re-verified solution may be used for an additional year, or until there is evidence of compound degradation or concentration. The re-verification must be performed using an unexpired, not previously re-verified solution from a second lot or second vendor.

7.5.1. Sealed ampules may be used until the manufacturer's expiration date is exceeded. If no expiration date is provided, then the expiration date will be 10 years from the date the ampule is opened. The solvent level should be monitored prior to each use to assure there has been no concentration of the standard over time.

7.5.2. Standards for method 8290A require storage at $\leq 6^{o}C$.

7.6. Field Surrogate Solution (air matrices)

This solution contains one ^{37}Cl labeled analog (for Method TO-9/TO-9A) or one ^{37}Cl and four ^{13}C labeled analogs (for Method 0023) at the nominal concentration indicated in Table 2. It is used to assess sample collection and recovery procedures.

7.7. Isotope Dilution Analyte Standard (IDA)

This acetone solution contains the nine IDA compounds at the nominal concentrations that are listed in Table 2. The solution contains at least one carbon-labeled standard for each homologous series, and it is used to measure the concentrations of the native substances. (Note that $^{13}C_{12}$ -OCDF is not present in the solution.)

7.8. Native Spike Standard

Also known as the Target Analyte Spike, Matrix Spike or Native Spike solution. Contains all the 2,3,7,8-substituted unlabeled analytes listed in Table 2. Prepare using the appropriate standards to yield a spiking solution with a concentration of 4.0 ng/ml for the tetra- CDDs/CDFs, 20 ng/ml for the penta-, hexa-, and hepta- CDDs/CDFs, and 40 ng/ml for the octa- CDD/CDF.

7.9. Internal Standard

This tetradecane solution contains two labeled compounds ($^{13}C_{12}$-1,2,3,4-TCDD and $^{13}C_{12}$ -1,2,3,7,8,HxCDD). An appropriate volume of this solution is spiked into each sample extract before the final concentration step.

7.10. Cleanup Recovery Standard or Surrogate (SU)
Prepare $^{37}Cl_4$-2,3,7,8-TCDD at the concentration shown in Table 2, in isooctane.

7.11. Preparation and QC of PUF material

7.11.1. The PUF material is purchased pre-cut.

7.11.2. The PUFs are rinsed by Soxhlet with acetone (or other appropriate solvent) for a minimum of 16 hours and air dried for a minimum of 2 hours in a contaminant-free area.

7.11.3. One PUF from the rinsed batch is randomly selected to be the QC sample for the batch.

7.11.4. The PUF is loaded into a pre-cleaned Soxhlet extractor charged with toluene.

7.11.5. The 1613/8290 daily IDA standard solution is spiked into the PUF and it is extracted for a minimum of 16 hours.

7.11.6. The Soxhlet extract is recovered and processed according to Section 11.11.

7.11.7. The batch of PUF is considered acceptable if no target analytes are detected at or above the laboratory or project specific reporting limit.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. The sample collection, shipping, handling, and chain-of-custody procedures are not described in this document. Sample collection personnel will, to the extent possible, homogenize samples in the field before filling the sample containers. This should minimize or eliminate the necessity for sample homogenization in the laboratory. The analyst should make a judgment, based on the appearance of the sample, regarding the necessity for additional mixing. If the sample is clearly non-homogeneous, the entire contents should be transferred to a glass or stainless steel pan for mixing with a stainless steel spoon or spatula before removal of a sample portion for analysis.

8.2. Grab and composite samples must be collected in glass containers.

8.3. Ambient air samples are collected on a Quartz Fiber Filter followed by a glass sleeve containing a polyurethane foam plug.

8.4. Samples from stationary sources are collected on glass or quartz fiber filters and XAD-2 Resin. (See WS-ID-0009 for sample preparation procedures).

8.5. Conventional sampling practices must be followed. Do not rinse the bottle with sample before collection. Sampling equipment must be free of potential sources of contamination.

8.6. Grinding or blending of fish samples.

If not otherwise specified by the client, the whole fish should be blended or ground to provide a homogeneous sample. The use of a stainless steel meat grinder with a 3 to 5 mm hole size inner plate is recommended. In some circumstances, analysis of fillet or specific organs of fish may be requested by the client. If so requested by the client, the above whole fish requirement is superseded. More detail can be found in "Tissue Sampling and Handling for a variety of Methods" (WS-WI-0018).

Warning: Hearing protection must be worn when grinding samples.

8.7. With the exception of the fish tissues, which must be stored at - 20°C, all samples should be stored at 4°C ± 2, extracted within 30 days and completely analyzed within 45 days of collection. The 30 day hold time is recommended. PCDDs and PCDFs have demonstrated stability for greater than one year.

8.8. All extracts must be stored capped, in the dark, at room temperature (approximately 21°C to 28°C). All extracts for method 8290A must be stored capped at ≤ 6ºC.

8.9. For moisture determinations refer to SOP WS-OP-0013.

9. QUALITY CONTROL

9.1. One method blank (MB) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The method blank is an aliquot of laboratory matrix (reagent water, sodium sulfate, PUF, XAD, filter, etc.) processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when target analytes are detected in the method blank above the reporting limit or when surrogate recoveries are outside control limits. Re-extraction of the blank, other batch QC, and the affected samples are required when the method blank is deemed unacceptable. The method blank contains a PUF plug, XAD, or filter prepared from the same batch as the field samples whenever possible for air samples.

Certain programs, such as DOD, may require a more stringent evaluation of the method blank, for instance, that the blank not contain any analytes of interest at a concentration greater than ½ the lower calibration limit.

Note: Re-extraction of the blank, QC and affected samples for the air matrices (PUF, XAD, and filter) is not generally possible because the entire sample is consumed in the initial extraction.

9.1.1. If the accompanying samples are aqueous, use distilled water as a matrix. Take the method blank through all steps detailed in the analytical procedure.

9.1.2. Use sodium sulfate as the method laboratory matrix when solids are extracted. Use a mixture of sodium sulfate and canola oil as the matrix when tissues are extracted. Take the method blank through all steps detailed in the analytical procedure.

9.1.3. The method blank must be spiked prior to extraction with the same amount of ^{13}C -labeled internal standards as added to samples.

9.1.4. If method blank contamination is present, check solvents, reagents, fortification solutions, apparatus and glassware to locate and eliminate the source of contamination before any further samples are extracted and analyzed. The presence of any analyte in the method blank ate concentrations greater than the reporting limit (RL) is cause for corrective action. Refer to SOP WS-ID-0005 for method blank acceptance criteria

9.2. A Laboratory Control Sample (LCS) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The LCS is an aliquot of laboratory matrix (e.g. water, sodium sulfate, PUF, XAD, etc.) spiked with analytes of known identity and concentration. An LCSD is required if a MS/MSD pair is not extracted with the batch. The LCS/LCSD must be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, other batch QC and all associated samples are required if the LCS/LCSD is deemed unacceptable. See policy WS-PQA-003 for specific acceptance criteria. When associated with PUF samples, the LCS should contain a PUF plug prepared from the same batch as the field samples whenever possible.

Note: Re-extraction of the blank, QC and affected samples for the air matrices (PUF, XAD, and filter) are not generally possible because the entire sample is consumed in the initial extraction.

9.2.1. A LCS is deemed acceptable if control analytes are above control limits and the associated samples are ND, unless otherwise specified by the client. Note any actions in the narrative.

9.3. The assessment of matrix effects on method performance, as required by NELAP, is met in Method 8290 and 8290A, as in all isotope dilution techniques, with the use of isotopically labeled compounds. These isotopically labeled compounds are analogs of target analytes and are spiked into each sample. Therefore, matrix effects on method performance may be judged by the recovery of these analogs. Sample analysis acceptance is controlled by the performance of these analogs in each sample. A Matrix Spike/Matrix Spike Duplicate (MS/MSD or MS/SD) pair are extracted at the client's request only. Method 8290A does not address analysis of MS/MSD. An exception to this rule is a batch containing South Carolina samples for Method 8290. These batches must have an MS/MSD prepared. However, South Carolina requires Method 8290A

after December 31, 2008. An MS/MSD pair are aliquots of a selected field sample spiked with analytes of known identity and concentration. When requested by the client, the MS/MSD pair shall be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spike analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, the LCS, the selected field sample, and the MS/MSD may be required after evaluation and review. Matrix Spike/ Matrix Spike Duplicates are not generally applicable for air samples due to the difficulty in collecting identical or representative samples. An LCS/LCSD may be extracted to show precision of the extraction and analysis process.

9.3.1. Matrix Spike (MS): A sample, which is spiked with a known amount of the matrix spike fortification solution prior to the extraction step. The recoveries of the matrix spike compounds are determined; they are used to estimate the effect of the sample matrix upon the analytical methodology.

9.3.2. Matrix Spike Duplicate (MSD): A second portion of the same sample as used in the matrix spike analysis and which is treated like the matrix spike sample.

9.3.3. Locate the sample for the MS and MSD analyses (the sample may be labeled "double volume").

9.3.4. Add an appropriate volume of the target analyte solution, adjusting the fortification level as specified in Table 1, under IDA Spiking Levels.

9.3.5. The results obtained from the MS and MSD samples (percent recovery and concentrations of 2,3,7,8-substituted PCDDs/PCDFs) should agree within 20 percent relative difference. Report all results and flag outliers.

9.3.6. IDA Standard recoveries are flagged if they are outside the recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any internal standard is less than 10:1.

9.4. Duplicates

9.4.1. Upon client request, duplicates may be processed. Locate the sample specified for duplicate analysis, and prepare and analyze a second 10-g soil or sediment sample portion or 1 L water sample, or an appropriate amount of the type of matrix under consideration. Duplicate samples are not generally applicable for air samples due to the difficulty in collecting identical or representative samples. A duplicate injection of a sample extract may be performed to display instrument precision.

9.4.1.1. The results of the laboratory duplicates (percent recovery and concentrations of 2,3,7,8-substituted PCDD/PCDF compounds) should agree within 25 percent relative difference. Report all results and flag outliers.

9.4.2. IDA Standard recoveries are flagged if they are outside the recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any internal standard is less than 10:1.

9.5. Field Blanks

9.5.1. Each batch of samples may contain a field blank sample of nominally uncontaminated soil, sediment or water that is to be processed for analysis.

9.5.1.1. Weigh a 10-g portion or use 1 L (for aqueous samples) of the specified field blank sample and add the appropriate amount of IDA Standard to yield 100 pg/µL in the final extract.

9.5.1.2. Extract by using the procedures described in Section 11. As applicable, add the appropriate amount of Internal Standard to yield 100 pg/µL in the final extract. Analyze a 1-2 µL aliquot of the concentrated extract using SOP WS-ID-0005.

9.6. Rinsate Samples

9.6.1. In addition to the field blank, a batch of samples may include a rinsate, which is a portion of the solvent (usually trichloroethylene) that was used to rinse sampling equipment. The rinsate is analyzed to assure that the samples were not contaminated by the sampling equipment.

9.6.2. The rinsate sample must be processed like a regular sample.

Take a 100-mL (± 0.5 mL) portion of the sampling equipment rinse solvent (rinsate sample), filter, if necessary, and add the appropriate amount of IDA Standard to yield 100 pg/µL in the final extract.

9.6.3. Using appropriate methods, concentrate to approximately 10 mL.

9.6.4. Just before analysis, add the appropriate amount of Internal Standard to yield 100 pg/µL in the final extract. Reduce the volume to a final volume of 20 µL, as necessary. No column chromatography is required.

9.6.5. Analyze an aliquot following the same procedures used to analyze samples.

9.7. Surrogate/Clean Up Recovery Standard

A surrogate compound may be spiked into all air media samples prior to collection. For all other matrices, Surrogate Standard is spiked following extraction and prior to cleanup, in order to monitor relative loss of Isotope Dilution Analyte Standard during both extraction and cleanup.

9.8. Isotope Dilution Analyte Standard (IDA Standard)

An internal standard is a ^{13}C -labeled analog of a PCDD/PCDF congener. Internal standards are added to all samples including method blanks and quality control samples before extraction, and they are used to quantitate the concentration of the analytes. Nine internal standards are used in this method. There is one for each of the dioxin and furan homologs (except for OCDF) with the degree of chlorination ranging from four to eight. Additional internal standards may be added to act as retention time references, but they are not used for quantitation.

9.8.1. A 2000 pg aliquot of the IDA Standard mixture is added to all samples, regardless of sample size. As an example, for $^{13}C_{12}$ -2,3,7,8-TCDD, a 10-g soil sample requires the addition of 2000 pg of $^{13}C_{12}$ -2,3,7,8-TCDD to give the requisite fortification level.

9.8.2. IDA Standard must be spiked into all samples, QC samples, and included in all calibrations.

9.8.3. For each sample and QC aliquot, calculate the percent recovery. The percent recovery should be between 40 percent and 135 percent for all nine internal standards.

9.8.4. A low or high percent recovery for a blank does not require discarding the analytical data but it may indicate a potential problem with future analytical data. IDA Standard recoveries are flagged if they are outside the recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any internal standard is less than 10:1.

9.9. Internal Standard: Two labeled compounds are used to determine the percent recoveries for the internal standards. The $^{13}C_{12}$ -1,2,3,4-TCDD is used to measure the percent recoveries of the tetra- and pentachlorinated internal standards while $^{13}C_{12}$ -1,2,3,7,8,9-HxCDD is used to determine the recovery of the hexa-hepta- and octachlorinated internal standards. $^{13}C_{12}$ -1,2,3,7,8,9-HxCDD also acts as a retention time reference for the unlabeled analog present in sample extracts. They are added to the final sample extract before HRGC/HRMS instrument analysis.

9.10. Recommended Corrective Actions and Troubleshooting Steps

- Verify satisfactory instrument performance.
- If possible, verify that no error was made while weighing the sample aliquots.
- Review the analytical procedures with the performing laboratory personnel.

10. CALIBRATION

10.1. On a daily basis, calibrate any balance to be used in accordance with SOP WS-QA-0041.

10.2. On a daily basis, calibrate any autopipettor to be used in accordance with SOP WS-QA-0004.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.2. Refer to SOP WS-IDP-0009 for the preparation of stationary source samples.

11.3. Sample Pre-Treatment

11.3.1. Paper Pulp Sludges are generally air-dried and ground prior to extraction following Section 11.5. Because of the drying procedure, a Dean-Stark water separator is optional for extraction.

11.3.2. Fly Ash — Fly ash samples are pretreated with HCl prior to extraction by both soxhlet and separatory funnel techniques.

11.3.2.1. Weigh 2-10g of sample aliquot into a clean glass jar.

11.3.2.2. Add 1.0mL of the internal standard mixture with 2 mL of acetone.

11.3.2.3. Add 150 mL of 1N hydrochloric acid and shake for 4 hours.

11.3.2.4. If the sample reacts violently with acid, then allow the sample to equilibrate for 4 hours with no shaking.

11.3.2.5. Filter the contents of the jar through a glass fiber filter.

11.3.2.6. Extract the solids as per Section 11.5, omitting the daily IDA standard for the samples.

11.3.2.7. Extract the aqueous filtrate as per Section 11.9, using 100 mL of toluene for the first shake, and 100 mL of hexane for subsequent shakes.

11.3.2.8. Concentrate the combined toluene solutions to near dryness on a rotary evaporator at 50°C. Proceed with Section 11.16 as necessary.

Note: As an option, a Soxhlet/Dean Stark extractor system may be used, with toluene as the solvent. No sodium sulfate is added when using this option.

11.4. Waste Dilution (Still-Bottom/Fuel Oil, and other solvent-miscible materials).

11.4.1. Weigh 1 g of the waste (organic liquids, fuel oils, and solids that will dissolve in a solvent) into a vial.

11.4.2. Add 40 mL of toluene (or other solvent if the material is not miscible/soluble in toluene). Shake gently to dissolve.

11.4.3. Remove a 4.0 mL aliquot (0.1g sample equivalent) and place in a culture tube. Add 1.0 mL of daily IDA standard and 1.0 mL of SU standard, and proceed to Section 11.16.

11.5. Soxhlet Extraction (Solids, Tissues, Sludges, Wipes)

11.5.1. Pre-extract the glassware by heating the flask until the toluene is boiling. When properly adjusted, 1-2 drops of toluene per second will fall from the condenser tip into the receiver. Extract the apparatus for a minimum of four hours.

WARNING: Open the chiller supply valves about 15 minutes before turning on the heating element and ensure that all of the condensors are cold before you turn the heating element on. Check all of the condensors about 15 minutes after starting the heating process to ensure that they are still cold and functioning properly. If this cleaning cycle is to be left unattended (e.g., overnight) the first chemist to arrive the next morning is to check all condensers to ensure that they are still cold and functioning properly.

11.5.2. After pre-extraction, cool and disassemble the apparatus.

11.5.3. If tissues requiring % Lipids are to be extracted, for each sample weigh the concentration vessel with label and boiling chips. Record the mass on the benchsheet. Refer to SOP WS-QA-0018 “Subsampling”, for instructions on how to homogenize and subsample the container of sample.

11.5.4. Weigh a well-mixed aliquot of each sample (10 g, unless otherwise specified) into a clean thimble. Record the mass to the nearest 0.01g. Use

sodium sulfate for the batch QC (MB, LCS) for solids, and a mixture of 9.75 g sodium sulfate and 0.25 g canola oil for the batch QC for tissue matrices.

11.5.4.1. In the case of wipes, place the entire wipe sample into the Soxhlet apparatus (no thimble needed), including any liquid present with the sample. Use pre-cleaned wipes for the batch QC samples.

11.5.5. Place the thimble into a Soxhlet apparatus equipped with a Dean-Stark water separator.

11.5.6. For each sample or sample set (to a maximum of 20 samples) to be extracted during the same 12-hour shift, weigh two aliquots of the appropriate reference matrix into clean thimbles. One aliquot will serve as the blank and the other will serve as the laboratory control sample (LCS).

11.5.6.1. Into the blank, add 1 mL of daily IDA standard (2 pg/µL).

11.5.6.2. Into the LCS, add 1 mL of daily IDA standard (2 pg/µL) and 50 µL of the TA spike.

11.5.6.3. For each field sample, add 1 mL of daily IDA standard. For MS/SD aliquots, add 50 uL of the TA spike as well.

Note: This spike level will give a final concentration of 200 pg/g (based on a 10g sample).

11.5.7. Reassemble the pre-extracted apparatus and add a fresh charge (250-300 mL) of toluene to the receiver and reflux flask.

11.5.8. Reflux 16 hours, with the solvent cycling at least 5 times per hour.

WARNING: Open the chiller supply valves about 15 minutes before turning on the heating element and ensure that all of the condensors are cold before you turn the heating element on. Check all of the condensors about 15 minutes after starting the heating process to ensure that they are still cold and functioning properly. If this cleaning cycle is to be left unattended (e.g., overnight) the first chemist to arrive the next morning is to check all condensers to ensure that they are still cold and functioning properly.

11.5.9. Drain the water from the receiver if the receiver fills with water. Check and drain when necessary.

Note: If the receiver holds 10 mL of liquid, and 20 g of an approximately 10% solid sample is being extracted, then approximately 9 mL of water will end up in the receiver. In this case, the receiver will not need to be emptied (insufficient liquid to overflow), but it should be checked. If the sample amount is 50, and the percent solids

is still 10%, then 45 mL of water will end up in the receiver. In this case, frequent checking is required, and the receiver will need to be emptied at least 5 times.

11.5.10. After refluxing, allow the apparatus to cool.

11.5.11. If samples DO NOT require % lipids add 100 µL of tetradecane as a keeper to the round bottom flask.

11.5.12. Proceed to Section 11.18.

11.6. SoxTherm Extraction (Solids, Tissues, Sludges,Wipes)

11.6.1. Prior to loading samples, run the system through 2 cleaning cycles (approximately 1 hour each).

11.6.2. After pre-extraction, cool and disassemble the apparatus.

11.6.3. Weigh a well-mixed aliquot of each sample (10 g, unless otherwise specified) into a clean thimble. Record the mass to the nearest 0.01g. Use sodium sulfate for the batch QC (MB, LCS) for solids, and a mixture of 9.75 g sodium sulfate and 0.25 g canola oil for the batch QC for tissue matrices.

11.6.3.1. In the case of wipes, place the entire wipe sample into the Soxhlet apparatus (no thimble needed), including any liquid present with the sample. Use pre-cleaned wipes for the batch QC samples.

11.6.4. Place the thimble into the Soxtherm apparatus.

11.6.5. For each sample or sample set (to a maximum of 20 samples) to be extracted during the same 12-hour shift, weigh two aliquots of the appropriate reference matrix into clean thimbles. One aliquot will serve as the blank and the other will serve as the laboratory control sample (LCS).

11.6.5.1. Into the blank, add 1 mL of daily IDA standard (2 pg/µL).

11.6.5.2. Into the LCS, add 1 mL of daily IDA standard (2 pg/µL) and 50 µL of the TA spike.

11.6.5.3. For each field sample, add 1 mL of daily IDA standard. For MS/SD aliquots, add 50 uL of the TA spike as well.

Note: This spike level will give a final concentration of 200 pg/g (based on a 10g sample).

11.6.6. Reassemble the pre-extracted apparatus and add a fresh charge (150 mL) of toluene to the apparatus.

11.6.7. Program the system to boil for 1 hour, and reduce the toluene volume by 70-90 mL (volume < volume of the thimble).

11.6.8. Continue the extraction for one hour fifteen minutes, reducing the toluene volume by another 15 mL.

11.6.9. After refluxing, allow the apparatus to cool.

11.6.10. Pour the samples into round bottom flasks, and if samples DO NOT require % lipids add 100 µL of tetradecane as a keeper to the round bottom flask.

11.6.11. Proceed to Section 11.18.

11.7. Microwave Assisted Extraction (MAE)

WARNING: Microwave ovens used for extraction or digestion of samples can create elevated pressure in the extraction/digestion containers. Follow procedures in the operator's manual to ensure that the vapor sensor(s) are functional and working properly prior to starting each extraction/digestion batch.

11.7.1. Prior to loading samples, run the system through a cleaning cycle (approximately 35 minutes) using approximately 30 mL 1:1 Toluene:Acetone and the following program (cleaning run.mpr):

Step	Time (min)	Power (W)	Temperature (℃)
1	10	1200	145
2	25	1200	145
3	10	0	ambient

11.7.2. After pre-extraction, cool and disassemble the apparatus.

11.7.3. Weigh 10 g (or required sample amount) of each field sample and MS/MSD if required into the cleaned Teflon chambers and mix with approximately 5 g of diatomaceous earth. MB, LCS and if required LCSD aliquots are made using 10g of Na_2SO_4 and 5g of diatomaceous earth.

11.7.4. Add 1 mL of deionized water to each sample and QC in Teflon sample chamber. The number of sample chambers should match the number of field samples and QC samples.

11.7.5. Spike according to Section 11.6.5.1, 11.6.5.2, and 11.6.5.3.

11.7.6. Add 30 mL of 1:1 Toluene:Acetone to each Teflon sample chamber.

11.7.7. Add the Teflon cap with the Teflon pressure release valve on top of each chamber and place the setup into the plastic pressure vessel and screw on the

cap. Into one vessel (that has a special Teflon pressure release valve) carefully add the monitoring probe casing into the top of the chamber. Be careful to not force this probe casing as it is very fragile.

11.7.8. Add flared extraction vessel covers to each extraction vessel containing sample and QC aliquots.

11.7.8.1. It is important that each cover fits snugly to ensure a proper seal. The cover should not slide easily or loosely inside the extraction vessel, but should require some finger pressure to insert firmly. A cover flaring tool should be used.

11.7.8.2. For the visually wettest sample, add the thermowell liner into the extraction vessel cover to create the representative sample that the ATC temperature sensor can be inserted into.

11.7.9. Place each extraction vessel into a pressure reactor. Screw on the pressure cap/safety lid. The pressure cap should be hand tightened until the sealing valve is flush with the top of the cap.

11.7.9.1. For the representative sample created in 11.7.8.2, add the protection foil and appropriate safety lid.

11.7.10. Place all the extraction vessels into the rotor so that the pressure-release valves are facing outside of the rotor on the outside ring and inside toward the center on the inside ring.

WARNING: Microwave ovens used for extraction or digestion of samples can create elevated pressure in the extraction/digestion containers. Follow procedures in the operator's manual to ensure that the vapor sensor(s) are functional and working properly prior to starting each extraction/digestion batch.

11.7.10.1. Place the rotor in the microwave oven and insert the ATC temperature sensor into the representative sample (11.7.8.2 and 11.7.9.1).

11.7.11. Close the microwave oven and start the appropriate extraction profile.

11.7.11.1. A 10 minute ramp from 25°C to 115° C, hold for 30 minutes at 115°C, followed by a 10 minute cool down to ambient temperature.

11.7.12. Run the following program for sample extraction:

Step	Time (min)	Power (W)	Temperature (^{o}C)
1	30	1200	145
2	30	1200	145
3	10	0	(ambient)

11.7.13. Following extraction filter the extract through Na_2SO_4 and collect in a round bottom flask, rinse twice more with approximately 5 mL to 10 mL of toluene for a total of about 50 mL.

11.7.14. Add approximately 100 uL C_{14} to each round bottom flask if % lipid is not needed.

11.7.15. Proceed to Section 11.18.

11.8. Extract Splitting (Wipes)

Wipe extracts prepared using any extraction technique are split prior to further workup, to permit an archive aliquot, or analysis by an additional method. The samples and QCs in the batch should be spiked with 2X of the nominal spiking amount to achieve a a 1X spiking amount after splitting. Once the extract has been concentrated using the rotovap or Turbovap, proceed as follows:

11.8.1. Add approximately 1 mL of hexane or toluene to rinse the sides of the round bottom flask. Using a pipette, withdraw the sample from the round bottom flask and transfer the liquid into a test-tube. Use additional amounts of solvents to rinse the flask. Transfer all the liquid into the test-tube. Ensure that all traces of sample in the round bottom flask have been thoroughly rinsed from all surfaces. Bring the sample volume to 8.0 mL or 10.0 mL (or appropriate volume) with the addition of rinse solvent.

11.8.2. Upon completion of the rinsing, cap the test tube and shake vigorously. Take ½ of each sample (or an appropriate amount as instructed by the client, program manager or department manager) and transfer to a culture tube. Archive the remaining sample for future use.

11.8.2.1. If only one analysis is required, then ½ of the sample is archived and the other half is analyzed.

11.8.2.2. If “N” analyses are required, then the extract is divided into “N+1” equal portions, so that one portion is archived, and a portion is used for each test.

11.9. Aqueous Samples (liquid/liquid extraction).

11.9.1. When setting up the glassware for a batch, for each sample label one separatory funnel with the sample ID.

11.9.2. If any samples has more than 10% solid (~1inch), set sample aside and contact your supervisor or PM.

11.9.3. Weigh the sample in the bottle on the top loading balance to the nearest centigram (0.01g), and record the mass.

11.9.3.1. For leachate samples (including an aliquot of the leachate blank), measure 200 mL of leachate into a clean 1 L sample bottle. Add approximately 800 mL of reagent water to the leachate. The sample volume in TALS for leachates is 200 mL.

11.9.4. For each sample, add 1 mL of IDA standard to the sample in the original container.

11.9.5. Create a blank and LCS by adding 1 L of laboratory reagent water to a 1L AGB. Spike 50 µL of the TA standard into the LCS container. Spike each one with 1 mL of IDA.

11.9.6. Pour the entire sample (approximately 1L) into a 2L separatory funnel that is labeled with the sample ID.

11.9.7. Add 100 mL methylene chloride to the sample bottle, seal, and shake for 30 seconds to rinse the inner surface. Transfer the solvent to the separatory funnel.

11.9.8. Extract the samples by shaking each funnel for two minutes with periodic venting.

Warning: Separatory funnel extraction with methylene chloride is a high-risk activity. Pressure may build rapidly in the funnel. It should be vented after several seconds of shaking, and often enough to prevent build-up of pressure. Chemist performing separatory funnel extraction must wear a face shield over their safety glasses/goggles. Alternatively, the extraction can be performed behind a closed fume hood sash.

11.9.9. Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation.

11.9.10. Repeat the extraction two additional times with 100mL of methylene chloride each time.

11.9.11. Determine the original sample volume by re-weighing the sample bottle. Record the sample volume to the nearest centigram (0.01g).

11.9.12. Dry extract with sodium sulfate: Place glass wool in a precleaned filter funnel. Rinse glass wool with methylene chloride and load funnel with Na_2SO_4. Pour extract through Na_2SO_4 to remove water. Rinse Na_2SO_4 with fresh methylene chloride and collect in round bottom flask.

11.9.13. Transfer the extract to a 500 mL round-bottom previously labeled with the

sample ID, then add approximately 100 µL of tetradecane.

11.9.14. Perform macro-concentration as detailed in Section 11.18.

11.10. Aqueous Samples via solid phase extraction.

11.10.1. Weigh the sample in the bottle on the top loading balance to the nearest centigram (0.01g), and record the mass.

11.10.2. Create a blank and LCS by adding 1L of laboratory reagent water to 2 additional 1L bottles.

11.10.3. For each sample, add 1mL of IDA standard solution into the sample in the bottles. Each aliquot of spike mixture is added similarly.

11.10.4. To the LCS, add 50µL of the TA standard.

11.10.5. Prepare the C18 extraction discs by first soaking them in toluene for at least 5 minutes.

11.10.6. Assemble the filter holder and vacuum filtration flask and place the extraction disc onto the filter holder. Place a GF-F filter on top of the extraction disc. If the sample has a large amount of particulates a GF-D filter can be placed on top of the GF-F filter. Alternatively, a GMF-150 filter can be used in place of the two filters.

11.10.7. Place the filtering funnel onto the disc holder and clamp it in place.

11.10.8. Rinse the filter and discs with approximately 15mL of toluene and allow it to soak for about a minute. Apply vacuum and draw the toluene through the discs. Repeat the wash step using about 15mL of acetone. Apply vacuum and draw the acetone through the discs.

11.10.9. Rinse the filter and discs with approximately 15mL of methanol and allow it to soak for about a minute. Apply vacuum and draw the methanol through the discs, but **DO NOT ALLOW THE DISCS TO GO DRY**. If they do go dry, simply repeat the methanol rinse step, leaving a 1 – 2mm layer of solvent on top of the discs.

11.10.10. Rinse twice with about 50mL of reagent water, leaving a 1 – 2mm layer of water on the surface of the discs.

11.10.11. Pour the spiked method blank, LCS or sample into the reservoir and apply vacuum to begin the extraction. Adjust the vacuum such that the extraction takes approximately 10 minutes. Samples with large amounts of particulates

may take much longer.

11.10.12. After most of the sample has been pulled through the discs, rinse the sample bottle with a few mL of reagent water and add the rinse to the funnel. Rinse down the sides of the funnel with reagent water as well.

11.10.13. Allow the discs to dry, remove them from the holder and extract by soxhlet (11.5) or soxtherm (11.6) and proceed with cleanups.

11.10.14. Determine the original sample volume by re-weighing the sample bottle. Record the sample volume to the nearest centigram (0.01g).

11.11. Breaking Emulsions

There are several useful methods to decrease or eliminate emulsion in aqueous samples when extracting with methylene chloride. These methods may include stirring with a pipette to manually breakup the emulsions or to transfer the sample into centrifuge tubes and centrifuge at approximately 3000 RPM. The most useful method is to use a 10 NNaOH solution to change the pH enough to disrupt the emulsion phase, which works 90% of the time.

11.11.1. Check the pH of the sample to verify that the pH is between 3 and 7. If the pH is greater than 7, consult the supervisor and client for instructions.

11.11.2. Pour approximately 10 mL of the 10N NaOH into a 1 L amber glass bottle (AGB).

11.11.3. Drain the sample with the emulsion from the 2 L separatory funnel into the 1 L AGB and let it stand.

11.11.4. Empty the aqueous waste into the LLE waste drum.

11.11.5. Pour the solution with methylene chloride back into the same 2 L separatory funnel and drain the methylene chloride phase through Na_2SO_4 into a 500 mL round-bottom flask.

11.11.6. Empty the aqueous waste into the LLE waste drum.

11.11.7. Perform macro-concentration as detailed in Section 11.18.

11.12. Filter/PUF Samples

11.12.1. Place the glass sleeve containing the PUF and the Quartz Fiber Filter into the pre-cleaned Soxhlet extractor charged with toluene.

11.12.2. Add 2 mL (4000 pg) of 1613/8290 daily IDA Standard solution to all

samples and QC.

11.12.3. Add 50 uL of 1613/8290 TA standard to the LCS.

11.12.4. Extract the samples and QC for a minimum of 16 hours.

11.12.5. Concentrate the extract from the round bottom flask using method described in section 11/18.

11.12.6. Transfer the extract from the round bottom flask with hexane and adjust the volume.

11.12.7. Split the extract 50:50 for analysis and archive.

11.13. Extract Clean-Up

11.13.1. For all samples that are not air media, spike 1.0 mL of the Surrogate standard prior to any cleanup, into the round bottom flasks containing the samples and QC Extracts (See also Section 9.7).

11.13.2. Proceed with further cleanups as dictated by the sample matrix and extract color. The "Option C" cleanup (Section 11.14) and the IFB Upper Column cleanup (Section 11.15) are applied to samples with high levels of interferences. The IFB column cleanup (Section 11.16) is applied to all samples.

11.14. Acid Partitioning ("Option C") - **Optional cleanup**

11.14.1. Use this clean up as needed on samples with high levels of interferences. Consult with a lead chemist or department manager to determine applicability.

11.14.2. Partition the extract in 50-125 mL of hexane against 20 mL concentrated H_2SO_4 in a separatory funnel. Shake for two minutes. Remove and discard the H_2SO_4 layer (bottom). Repeat the acid washing until no color is visible in the acid layer (perform a maximum of five acid washings).

Warning: Shaking with a concentrated caustic is a high-risk activity. Analyst must wear a face shield over safety glasses/goggles, or the shaking must take behind a closed hood sash.

11.14.3. Partition the extract against 20 mL of distilled H_2O. Shake for two minutes. Remove and discard the aqueous layer (bottom). Dry the extract by pouring it through a funnel containing anhydrous sodium sulfate and collect it in a round-bottom flask. Rinse the sodium sulfate with two 15 mL portions of hexane, add the rinsates to the flask, and concentrate the hexane solution to

near dryness on a rotary evaporator (35°C water bath), making sure all traces of toluene (when applicable) are removed. (Use of blow-down with an inert gas to concentrate the extract is also permitted).

11.15. IFB Upper Column Cleanup – **Optional cleanup**

11.15.1. Use this clean up as needed on samples with high levels of interferences. Consult with a lead chemist or department manager to determine applicability.

11.15.2. Set up the upper of the two chromatography columns as depicted in Figure 2. The column (20 mm diameter) is packed in this order: a glass wool plug, 2 g activated silica gel, 4 g Acid silica gel, 2 g activated silica gel, and 1 g sodium sulfate.

11.15.3. Pre-rinse the column with 20 mL hexane, and discard the rinsate.

11.15.4. Position each column with a round bottom below to collect the extract. Add extract to the column. Rinse extract vessel 2 times with 1 mL each of hexane and add to column.

11.15.5. Elute 90 mL hexane directly onto the upper IFB column.

11.15.6. Collect the eluate, and concentrate via directions described in section 11.18 before proceeding with the IFB cleanup (Section 11.16).

11.16. IFB Column Cleanup

All samples will undergo this cleanup, either direction following concentration on the rotovap, or following the cleanup in Section 11.14 (Option C) or Section 11.15 (IFB Upper Column).

11.16.1. Set up two chromatography columns as depicted in Figure 2. The upper column (20 mm diameter) is packed in this order: a glass wool plug, 2 g activated silica gel, 4 g Acid silica gel, 2 g activated silica gel, and 1 g sodium sulfate. The lower column (15 mm diameter) is packed in this order: a glass wool plug, 6 g acid alumina, and 1 g sodium sulfate.

11.16.2. Pre-rinse each column with 20 mL hexane, and discard the rinsate.

11.16.3. Put one column above the other.

11.16.4. Add extract to the top column (silica column). Rinse extract vessel 2 times with 1 mL each of hexane and add to column.

11.16.5. Elute 50 mL hexane directly onto acid silica column (upper column).

11.16.6. Discard upper column.

11.16.7. Elute lower column with 10 mL of 20% methylene chloride/hexane. Discard in proper waste stream.

11.16.8. Elute lower column with 30 mL of 65% methylene chloride/hexane. Save and collect in culture tube.

11.16.9. Proceed with additional cleanups as necessary.

11.17. Carbon Column Clean-up (D2 Column) – Optional cleanup

Prepare an activated Carbon & Silica Gel column as described in below. Refer to the diagram in Figure 3 as well.

11.17.1. Push a glass wool plug down to the 3.5 inch mark in a pre-cut D2 column.

11.17.2. Add 1 g of 5% activated carbon/silica. Top with a glass wool plug.

11.17.3. With the column oriented with "A" on the top (and the carbon on the lower end of the column), pre-elute with 5 mL 1:1 methylene chloride:cyclohexane.

11.17.4. Discard pre-eluates.

11.17.5. Invert the column so that the column is oriented with the "B" on the top and pre-elute with 3 mL of 1:1 methylene chloride.

11.17.6. Dilute the extract to 1 mL with hexane and transfer to the column (still oriented in the "B" direction).

11.17.7. Rinse sample vial onto the column with 2x 2 mL 1:1 methylene chloride:cyclohexane.

11.17.8. Elute with 5 mL 1:1 methylene chloride:cyclohexane. Additional amount can be used if samples has high matrix interference.

11.17.9. Elute with 5 mL 75:20:5 methylene chloride:methanol:benzene.

11.17.10. Discard eluates.

11.17.11. Turn the column over (so that the "A" end is on top), and elute with 30 mL of toluene. Collect this eluate.

11.17.12. Concentrate to NEAR dryness using the Rotovap (Section 11.18) or Turbovap (Section 11.19), then proceed to the recovery standard step (Section 11.20).

11.18. Macro-concentration (Rotary Evaporator)

Concentrate the extracts in separate round bottom flasks on rotary evaporator.

11.18.1. Assemble the rotary evaporator according to manufacture's instructions, and warm the water bath. On a daily basis, preclean the rotary evaporator by solvent rinsing. Between samples, 2-3 mL rinses of toluene followed by a 2-3 mL rinse of hexane should be rinsed down the feed tube into a waste beaker.

Rotovap Conditions		
Solvent	**Bath Temperature (C)**	**Vacuum Setting (PSI)**
Toluene	80	25
Hexane	65	15
Methylene Chloride	70	No vacuum applied

11.18.2. Attach the round bottom flask containing the sample extract to the rotary evaporator. Slowly apply vacuum to the system, and begin rotating the sample flask.

11.18.3. Lower the flask into the water bath and adjust the speed of rotation and the temperature as required. At the proper rate of concentration, the flow of solvent into the receiving flask will be steady, but no bumping or visible boiling of the extract will occur.

NOTE: If the rate of concentration is too fast, analyte loss may occur.

11.18.4. For samples requiring % Lipids analysis:

11.18.4.1. Concentrate until the toluene has been completely removed. Add approximately 25 mL hexane and concentrate to ensure that only the lipids are present.

11.18.4.2. Dry the concentration vessel and let stand at room temperature. Weigh the vessel and record on the benchsheet.

11.18.4.3. Calculate % lipids as follows:

$$\%\,\text{Lipids} = \frac{\text{Final Vessel Mass - Initial Vessel Mass}}{\text{Sample Size}} \times 100\%$$

11.18.5. Proceed to extract cleanups, or transfer to a micro concentration vial for the recovery standard step (Section 11.20).

11.19. Micro-concentration (Turbovap)

Concentrate the extracts in 35 mL culture tubes in a turbo-evaporator. The turbo-evaporator model that the laboratory uses can hold up to 50-35 mL culture tubes. Other turbo-evaporator models can be used that may or may not have the same culture

tube sizes and/or capacity. Adjust temperature according to solvent (65ºC for toluene and 45ºC for hexane or hexane/ methylene chloride mixtures)

11.19.1. The evaporating times are dependent on sample volume and solvent. The following are examples and can change from sample to sample. Each sample should be checked in intermittent intervals to make sure samples do not go dry.

11.19.2. When evaporating 30 mL toluene, it will normally take approximately 30-50 minutes with the temperature setting described above.

11.19.3. When evaporating 30 mL hexane/ methylene chloride, it will normally take approximately 20-30 minutes with the temperature setting described above.

11.19.4. For samples requiring % Lipids analysis refer to Section 11.18.4.

11.19.5. Proceed to extract cleanups, or transfer to a micro concentration vial for the recovery standard step (Section 11.20).

11.20. Recovery Standard

11.20.1. Add 20 µL of the Internal standard solution (Table 2) to each extract.

11.20.2. Transfer extracts to a micro concentration vial (test tubes and other small vessels may also be used). Rinse with 1mL of Hexane 2 times and transfer solvent to micro concentration vial.

11.20.3. With a stream of dry, purified nitrogen, reduce the extract volume to 20 µL.

11.20.4. Transfer the extract to an autoinjection vial and store in the dark at room temperature. If the samples are for method 8290A, the extracts must be stored in the freezer.

11.20.5. A smaller final volume can be used to decrease the detection limit upon client approval.

11.20.6. A larger final volume can be use to decrease potential matrix interferences, if the column and acid cleanups were unsuccessful.

11.21. Sample Dilution Procedure

11.21.1. Simple dilutions: Dilutions from 2X to 50X can be achieved without respiking the final extract. The calculation to determine the final extract concentration is as follows:

$$\text{Final Conc. of Extract} = \frac{(\text{Conc. of original extract}) \text{ x } (\text{Amount of aliquot taken})}{(\text{Volume of diluted extract})}$$

Ex: $$\frac{(10\text{ g}) \text{ x } (2\ \mu\text{L})}{(20\mu\text{L}) \text{ x } (100\mu\text{L})} = \frac{1\text{ g}}{100\mu\text{L}}\ \text{FV}$$

Record the final sample concentration on the extract label.

11.21.2. Complex dilution requiring respiking of IDA and IS standard:

Dilutions greater than 50x must be done by diluting and respiking the extract with IDA and IS standard. This procedure may require serial dilution to be performed. If this procedure is done, then the sample size must be adjusted to reflect the aliquot taken.

Ex. 100X dilution (original sample with 10 g/20 µL final volume)

Take a 2 µL aliquot (1/10 of original sample) and add 18 µL of solvent keeper. Take a 2 µL aliquot of the dilution (1/100 of the original sample), respike with 1 mL IS and 20 µL IS, reduced to 20 µL FV.

Record the final sample concentration of the extract label.

12. CALCULATIONS/DATA REDUCTION

12.1. Not applicable

13. METHOD PERFORMANCE

It must be documented that all applicable system performance criteria specified were met before analysis of any sample is performed. .

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the

method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. 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 Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.1. The use of Roto-vaps and Turbo-vaps rather than Kuderna-Danish reduction allows extraction solvents to be collected and disposed of rather than released to the atmosphere.

14.2. Toluene, which is a less hazardous solvent, has been substituted for benzene as an extraction solvent.

14.3. The use of SoxTherm extraction rather than soxhlet extraction, when appropriate, reduces the volume of solvent used.

14.4. Standards should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards that must be discarded.

14.5. All waste will be disposed of in accordance with Federal, State, and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.

14.6. Do not allow waste solvent to vent into the hoods. All solvent waste is stored in capped containers unless they are being filled.

14.7. Transfer waste solvent from collection cups (tri-pour and similar containers) to jugs and/or carboys as quickly as possible to minimize evaporation.

15. 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 SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Extracted aqueous/leachate samples contaminated with methylene chloride are collected at the fume hood in a 5-gallon or smaller carboy. If the samples are not at a neutral pH, add small quantities of sodium bicarbonate to bring the waste to neutral. Stir well. Once neutralized, immediately pour the carboy contents into a blue plastic LLE drum in the H3 closet. When full to between two and six inches of the top, or after no more than 75 days, move the LLE drum to the waste collection area for shipment.

15.2. Extracted soil samples and thimbles, extracted PUF filters, XAD-2 resin, paper funnel filters, glass wool, sodium sulfate, assorted disposable glassware, fish/crawfish or similar materials, silica gel, alumina, and carbon from column clean-ups, contaminated with various solvents and eluates. Dump the materials into an orange contaminated lab trash bucket. When the bucket is full or at the end of the day, tie the plastic bag liner shut and put the lab trash into the appropriate steel collection drum (high volatiles waste) in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.3. Miscellaneous disposable glassware, test tubes, syringes, filter disks, chemical resistant gloves, bench paper and similar materials that may or may not be contaminated/ hazardous. Place contaminated materials into a yellow contaminated lab trash bucket. When the bucket is full or after no more than one year, tie the plastic bag liner shut and put the lab trash into the appropriate steel collection drum (hazardous waste landfill) in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.4. Flammable solvent and methylene chloride waste generated during glassware and sodium sulfate cleaning. Solvent waste collected during roto-vap/turbo-vap reduction of extracted samples. Assorted flammable solvents and methylene chloride waste generated during quartz fiber filter preparation, PUF adsorbent preparation, XAD-2 resin preparation, PUF/XAD-2 cartridge preparation, glassware rinsing and sodium sulfate pre-rinsing. Collect the waste solvents in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel solvent

drum in the H3 closet. When full to between two and six inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment.

15.5. Contaminated distilled water used during extract cleanup. Collect the contaminated water in a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the plastic LLE drum in the H3 closet. When full to between two and six inches of the top, or after no more than 75 days, move the plastic drum to the waste collection area for shipment.

16. REFERENCES/CROSS REFERENCES

16.1. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update IV. Method 8290Al Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by high-Resolution Mass Spectrometry, February 2007.

16.2. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update IV. Method 3546, Microwave Extraction. February 2007.

16.3. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update III. Method 8290l Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by high-Resolution Mass Spectrometryl September 1994.

16.4. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update III. Method 0023A, Sampling Method for Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans Emissions from Stationary Sources. December 1996.

16.5. Compendium Method TO-9A “Determination of Polychlorinated, Polybrominated, and Brominated, Cholorinated Dibenxo-p-dioxins and Dibenzofurans in Ambient Air”, EPA compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, second edition, January 1997.

16.6. Protocol for the Analysis of 2,3,7,8-TCDD by HRGC/HRMS". J. S. Stanley and T. M. Sack, EPA 600/4-86-004.

16.7. “Safety in Academic Chemistry Laboratories", American Chemical Society Publication, Committee on Chemical Safety (3rd Edition, 1979.)

16.8. “Carcinogens - Working with Carcinogens". Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control. National Institute for Occupational Safety and Health. Publication No. 77-206, August 1977.

16.9. "OSHA Safety and Health Standards, General Industry", (29 CFR 1910) Occupational Safety and Health Administration, OSHA 2206 (revised January 1976).

17. METHOD MODIFICATIONS

17.1. Deviations from EPA 8290 and 8290A.

17.1.1. Tetradecane instead of nonane is used as the final solvent to increase the stability of extracts and standards. Tetradecane is less volatile than nonane. Loss of analyte as a result of solvent incompatibility is monitored through recovery checks and calibration acceptance criteria.

17.1.2. Extract clean-ups are performed at the discretion of the analyst when interferences are observed. Then, the analyst should select the clean-up procedure appropriate to the interferent.

17.1.3. Section 7.4.6.4 of Method 8290 indicates that extracts should be transferred with hexane, then toluene. Toluene is used to transfer extracts to maintain compound solubility and minimize analyte loss.

17.1.4. Section 7.5.1.2 of Method 8290 specifies that a NaCl solution should be used for partitioning. Instead, the laboratory uses laboratory water only. NaCl is used to break up emulsions that may form. An analyst may use NaCl, NaOH, or any mechanical means to break up an emulsion.

17.1.5. Section 7.5.3 of Method 8290 specifies that hexane is used as a column elution solvent. The laboratory uses cyclohexane to achieve better and more reproducible separation of the target analyte from the interferent.

17.1.6. Carbon columns are packed with silica gel in place of celite. Elution solvents are changed accordingly. (SOP Section 11.4; Method 8290 Section 7.5.3.2, 8290A Section 7.3.6.).

17.2. Modifications from TO-9A method

17.2.1. Quartz Fiber Filters are cleaned by Soxhlet extraction with methylene chloride, not baked at 400 degrees C for 5 hours.

17.2.2. The PUF material may be pre-cleaned with methylene chloride or other appropriate solvent. The PUFs are not reused.

17.2.3. The $^{37}Cl_4$-2,3,7,8-TCDD surrogate is present at varying levels in the calibration curve (0.5-200 pg/ µL).

17.2.4. Samples are extracted with toluene not benzene.

17.2.5. Concentration is performed by rotary evaporation not Kuderna-Danish.

17.2.6. All cleanup procedures are optional and applied based on the analyst's

discretion.

17.2.7. The laboratory uses 2 labeled recovery standard for the quantitation of labeled internal standards.

17.2.8. The final volume is adjusted to 20 µL in tetradecane.

17.2.9. Calibration and quantitation are performed in accordance to this SOP.

18. ATTACHMENTS

18.1. Table 1 - Types of Matrices

18.2. Table 2 - Composition of Sample Fortification and Recovery Standard Solutions.

18.3. Table 3 - The Seventeen 2,3,7,8-Substituted PCDD and PCDF Congeners

18.4. Figure 1 - Analysis Flowchart

18.5. Figure 2 — IFB column cleanup

18.6. Figure 3 — D2 Column cleanup

18.7. Appendix A - Periodic Wipe Test Performance

19. REVISION HISTORY

19.1. WS-IDP-0005, Revision 2.4, Effective 2/7/2017

19.1.1. Section 2.2, added “leachate” as an applicable matrix.

19.1.2. Inserted Section 11.9.3.1, “For leachate samples (including an aliquot of the leachate blanks), measure 200 mL of leachate into a clean 1 L sample bottle. Add approximately 800 mL of reagent water to the leachate. The sample volume in TALS for leachates is 200 mL.”

19.2. WS-IDP-0005, Revision 2.3, Effective 07/08/2016

19.2.1. Section 6.14 – Changed boiling chip solvent rinse from methylene chloride to hexane.

19.2.2. Section 9.2 – Inserted 4th sentence to paragraph – “An LCSD is required if a MS/MSD is not extracted with the batch.”

19.2.3. Section 11.14.2 and 11.14.3 – Changed 40 mL of concentrated H_2SO_4 (Section 11.14.2) and 40 mL of distilled H_2O (Section 11.14.3) to 20 mL.

19.2.4. Editorial changes.

19.3. WS-IDP-0005, Revision 2.2, Effective 05/28/2015

19.3.1. Added Section 5.1.9 – "Microwave ovens used for extraction or digestion of samples can create elevated pressure in the extraction/digestion containers. Microwaves used for these processes must be equipped with an automated vapor sensor and shutoff system that automatically shuts down the digestion/extraction process when vapor pressure reaches pre-specific levels. Users must follow procedures in the microwave operator's manual to ensure that the vapor sensors are functional and working properly prior to starting each extraction/digestion batch."

19.3.2. Section 7, removed reference to Kieselgel, and updated reagent information, added reference to diatomaceous earth, and moved the "reagents" section to 7.1. Also removed information regarding 10:1 NaOH:Water.

19.3.3. Section 11.7.3 – Replaced Ottawa sand with Na2SO4.

19.3.4. Added Sections 11.7.9 – 11.7.11 specifying microwave extraction procedure.

19.3.5. Revision history prior to 2012 has been removed. It is available for review in previous versions of this SOP.

19.3.6. Editorial changes.

19.4. WS-IDP-0005 Revision 2.1, Effective 02/21/2015

19.4.1. Added Section 3.6 - Target Analyte (TA) (named "unlabeled analytes" or "native spike" in the reference methods): The seventeen target analytes are listed in Table 3. The target analyte are added to the LCS/LCSD and MS/MSD (when prepared).

19.4.2. Removed Sections 9.1.4.1 – 9.1.4.5 and amended section 9.1.4 with "Refer to SOP WS-ID-0005 for method blank acceptance criteria."

19.4.3. Changed Section 11.5.6 to-" For each sample or sample set (to a maximum of 20 samples) to be extracted during the same 12-hour shift, weigh two aliquots of the appropriate reference matrix into clean thimbles. One aliquot will serve as the blank and the other will serve as the laboratory control sample (LCS).

19.4.4. Added Section 11.5.6.1 - Into the blank, add 1 mL of daily IDA standard.

19.4.5. Added Section 11.5..6.2 - Into the LCS, add 1 mL of daily IDA standard (2

pg/µL) and 50 µL of the TA spike.

19.4.6. Added Section 11.5.6.3 – For each field sample, add 1 mL of daily IDA standard. For MS/SD aliquots, add 50 uL of the TA spike as well.

19.4.7. Added at end of Section 11.5.6 - "Note: This spike level will give a final concentration of 200 pg/g (based on a 10g sample)."

19.4.8. Editorial changes.

19.5. WS-IDP-0005, Revision 2.0, Effective 02/14/2014

19.5.1. Added procedures for Microwave Extraction. (Section 2.2, inserted Section 11.7)

19.5.2. Added detail regarding use of Soxtherm extractors. (Section 2.2, added detail to Section 11.6)

19.5.3. Renamed Internal Standards (as per the method) to Isotope Dilution Analytes (as per our LIMS) to clarify for personnel.

19.5.4. Renamed Recovery Standards (as per the method) to Internal Standards (as per the LIMS) to clarify for personnel.

19.5.5. Renamed Cleanup Recovery Standards (as per the method) to Surrogate Standards (as per the LIMS) to clarify for personnel.

19.5.6. Editorial Revisions

19.5.7. The revision history prior to 2010 has been removed, and is available in earlier versions of this SOP.

19.6. WS-IDP-0005, Revision 1.5, Effective 12/21/2012

19.6.1. Clarified extraction procedure by revising Section(s) 11.8.1- 11.8.4 and adding an extra extraction step (Section 11.8.3).

19.6.2. Editorial revisions. .

19.7. WS-IDP-0005, Revision 1.4, Effective 03/20/2012

19.7.1. Appended to Section 2.2: "This method can also use solid phase extraction (SPE), however, Test America Sacramento is in the developmental stages for this extraction type and is not currently certified for its use."

19.7.2. Editorial changes.

TABLE 1

Types of Matrices, Sample Sizes and 2,3,7,8-TCDD-Based Method Calibration Limits (Parts per Trillion)

	Water	**Soil Sediment Paper Pulp**	**Fly Ash**	**Human/ Fish Tissue**	**Adipose Tissue**	**Sludges, Fuel Oil**	**Still-Bottom**	**Ambient or Source Samples**
Lower MCL(a)	0.01	1.0	2.0	1.0	2.0	10	20	40
Upper MCL(a)	4.0	400	400	400	400	2000	4000	8000
Weight (g)	1000	10	10	10	10	2.0	1.0	1 sample
IDA Spiking Levels (ng)	2.0	2.0	2.0	2.0	2.0	2.0	2.0	4.0
Final Extract Volume (µL)	20	20	20	20	20	20	20	20

(a) For other congeners, multiply the values by 1 for TCDF, by 5 for PeCDD/PeCDF/HxCDD/HxCDF/HpCDD/HpCDF, and by 10 for OCDD/OCDF.

TABLE 2

Composition of the Isotope Dilution Analyte Solution and Internal Standard Solutions

Analyte	Isotope Dilution Analyte Solution Concentration pg/µL; Solvent: Acetone	Internal Standard Solution Concentration pg/µL; Solvent: Tetradecane
$^{13}C_{12}$ -2,3,7,8-TCDD	2[a], 100[c]	--
$^{13}C_{12}$ -2,3,7,8-TCDF	2[a], 100[c]	--
$^{13}C_{12}$ -1,2,3,4-TCDD	--	100
$^{13}C_{12}$ -1,2,3,7,8-PeCDD	2[a], 100[c]	--
$^{113}C_{12}$ -1,2,3,7,8-PeCDF	2[a], 100[c]	--
$^{13}C_{12}$ -1,2,3,6,7,8-HxCDD	2[a], 100[c]	--
$^{13}C_{12}$ -1,2,3,4,7,8-HxCDF[d]	2[a], 100[c]	--
$^{113}C_{12}$ -1,2,3,7,8,9-HxCDD	--	100
$^{13}C_{12}$-2,3,7,8-TCDD[b][c]	0.8[b], 100[c]	
	100[c]	
$^{13}C_{12}$ -2,3,4,7,8-PeCDF[c]	100[c]	
$^{13}C_{12}$ -1,2,3,6,7,8-HxCDF[c][d]	100[c]	
$^{13}C_{12}$ -1,2,3,4,7,8-HxCDD[c]	100[c]	
$^{13}C_{12}$ -1,2,3,4,7,8,9-HpCDD[c]	100[c]	
$^{13}C_{12}$ -1,2,3,4,6,7,8-HpCDD	2[a], 100[c]	--
$^{13}C_{12}$ -1,2,3,4,6,7,8-HpCDF	2[a], 100[c]	--
$^{13}C_{12}$ -OCDD	4[a], 200[c]	--

(a) Standard 8290, Method 23, Method 0023A, TO9 and TO9A Sample Fortification Solution concentrations
(b) Method TO9 and TO9A surrogate concentrations
(c) Method 23 and Method 0023A surrogate concentrations
(d) $^{13}C_{12}$ -1,2,3,6,7,8-HxCDF is used as a Sample Fortification Solution and $^{13}C_{12}$-1,2,3,4,7,8-HxCDF is used as a surrogate solution in Method 23 and Method 0023A

TABLE 3

The Seventeen 2,3,7,8-Substituted PCDD and PCDF Congeners

PCDD	PCDF
2,3,7,8-TCDD(*)	2,3,7,8-TCDF(*)
1,2,3,7,8-PeCDD(*)	1,2,3,7,8-PeCDD(*)
1,2,3,6,7,8-HxCDD(*)	2,3,4,7,8-PeCDF
1,2,3,4,7,8-HxCDD	1,2,3,6,7,8-HxCDF
1,2,3,7,8,9-HxCDD(+)	1,2,3,7,8,9-HxCDF
1,2,3,4,6,7,8-HpCDD(*)	1,2,3,4,7,8-HxCDF(*)
1,2,3,4,5,6,7,8-OCDD(*)	2,3,4,6,7,8-HxCDF
	1,2,3,4,6,7,8-HpCDF(*)
	1,2,3,4,7,8,9-HpCDF
	1,2,3,4,5,6,7,8-OCDF

(*)The ^{13}C -labeled analog is used as an internal standard.
(+)The ^{13}C -labeled analog is used as a recovery standard.

FIGURE 1 — Flowchart of Process

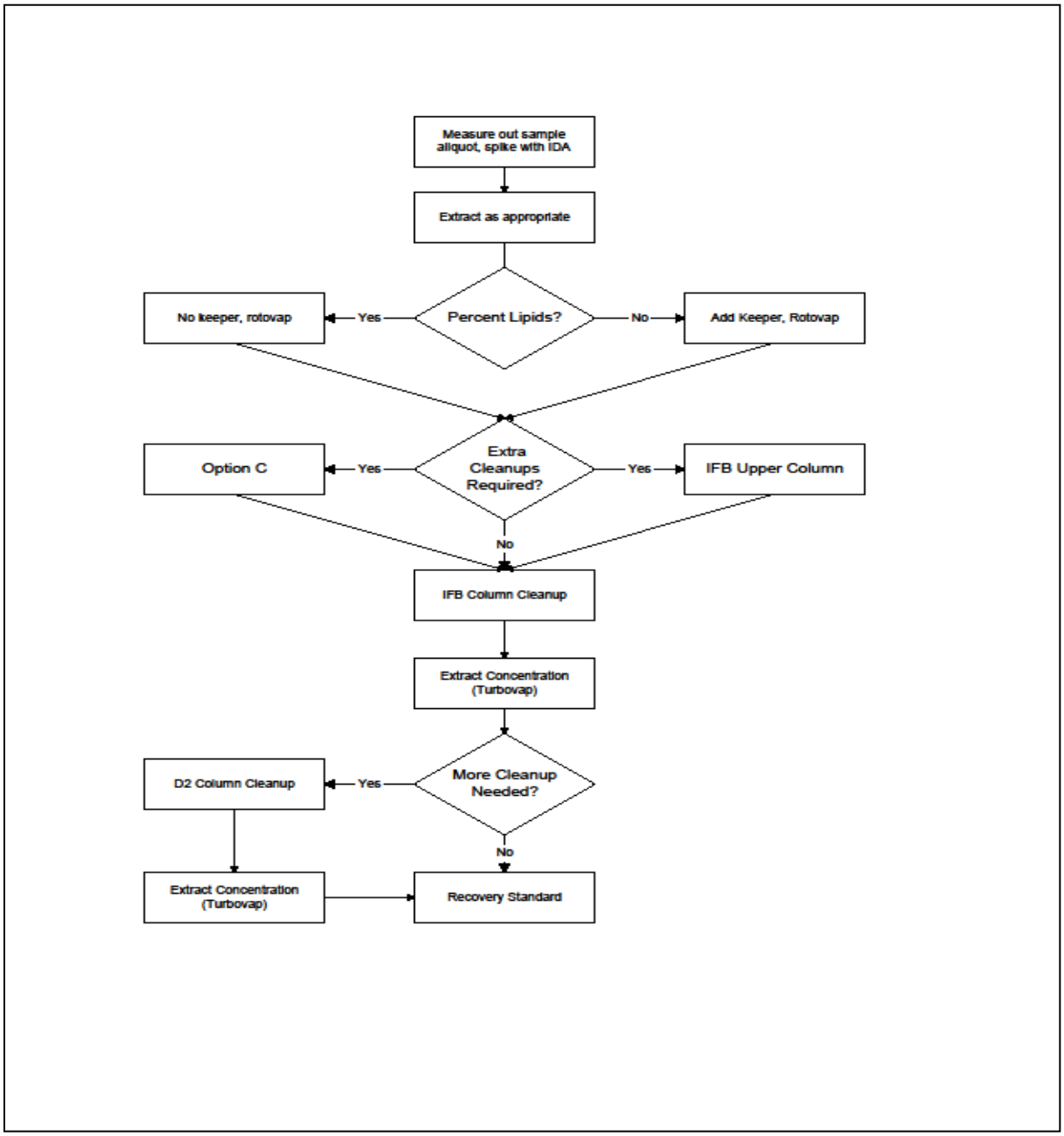
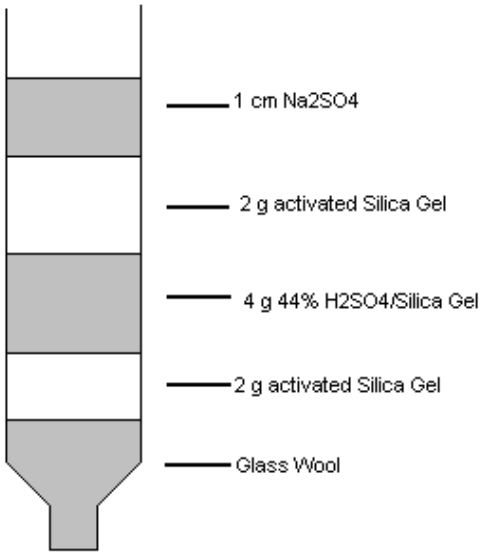
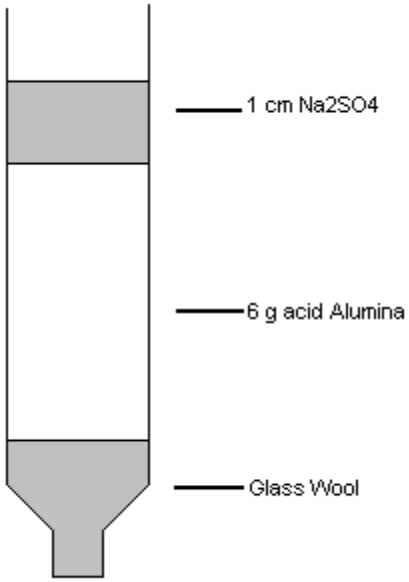


Figure 2 – Diagram of IFB Column Cleanup

Use 20 mm column for top column (IFB Column)
Use 16 mm column for bottom column* (Acid Alumina)
Note: Upper and lower columns are piggy backed for IFB cleanup, upper column only can be used for additional cleaning.

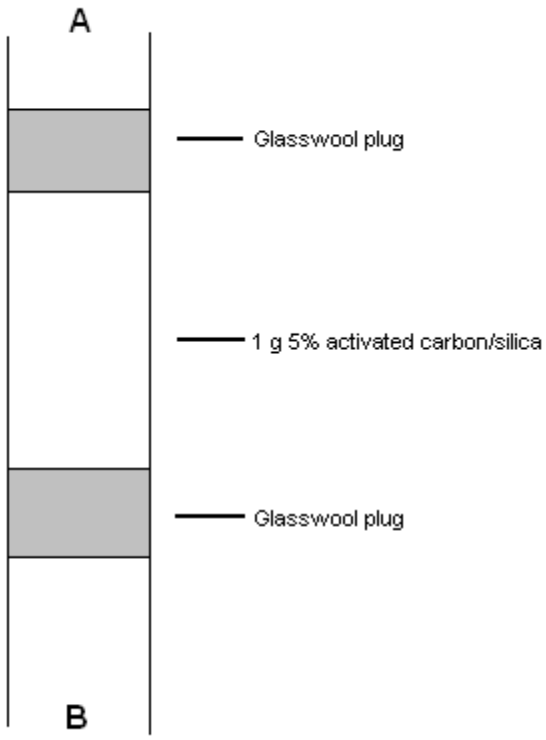


Instead of 1cm Na2SO4, it's 1g.



Instead of 1cm Na2SO4, it's 1g.

Figure 3— D2 Carbon Column:



APPENDIX A — Screening the Laboratory for 2,3,7,8 Congeners

This procedure is designed for the periodic evaluation of potential contamination by 2,3,7,8-substituted PCDD/PCDF congeners of the working areas inside the laboratory.

PERFORMING WIPE TEST
Perform the wipe tests on surface areas of two inches by one foot with laboratory wipers saturated with distilled-in-glass acetone or appropriate solvent using a pair of clean stainless steel forceps. Use one wiper for each of the designated areas. Combine the wipers to one composite sample in an extraction jar containing 200 mL distilled-in-glass hexane. Place an equal number of unused wipers in 200 mL hexane and use this as a control.

SAMPLE PREPARATION
Close the jar containing the wipes and 200 mL hexane and extract for 20 minutes using a wrist-action shaker. Use an appropriate means to reduce the volume to approximately 1.0 mL. Put through an alumina column to clean up potential interfering compounds. Add appropriate amount of recovery standard.

EXTRACT ANALYSIS
Concentrate the contents of the vial to a final volume of 20 µL (either in a minivial or in a capillary tube). Inject 2 µL of each extract (wipe and control) onto a capillary column and analyze for 2,3,7,8-substituted PCDDs/PCDFs as specified in the analytical method Section 11 (this exhibit). Perform calculations according to Section 12 (this exhibit).

REPORTING FORMAT
Report the presence of 2,3,7,8-substituted PCDDs and PCDFs as a quantity (pg or ng) per wipe test experiment (WTE). Under the conditions outlined in this analytical protocol, a lower limit of calibration of 25 pg/WTE is expected for 2,3,7,8-TCDD. A positive response for the blank (control) is defined as a signal in the TCDD retention time window at any of the masses monitored which is equivalent to or above 8 pg of 2,3,7,8-TCDD per WTE. For other congeners, use the multiplication factors listed in Table 1, footnote (a) (e.g., for OCDD, the lower MCL is 25 x 5 = 125 pg/WTE and the positive response for the blank would be 8 x 5 = 40 pg). Also, report the recoveries of the internal standards during the simplified cleanup procedure.

FREQUENCY OF WIPE TESTS
Wipe tests should be performed when there is evidence of contamination in the method blanks.

CORRECTIVE ACTION
An upper limit of 25 pg per TCDD isomer and per wipe test experiment is allowed. (Use multiplication factors listed in footnote (a) from Table 1 for other congeners.) This value corresponds to the lower calibration limit of the analytical method. Steps to correct the contamination must be taken whenever these levels are exceeded. To that effect, first vacuum the working places (hoods, benches, sink) using a vacuum cleaner equipped with a high-efficiency

particulate absorbent (HEPA) filter and then wash with a detergent. A new set of wipes should be analyzed before anyone is allowed to work in the dioxin area of the laboratory.

The test results and the decontamination procedure must be reviewed with EH&S.

Title: Preparation of Samples for Tetra- through Octa Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS by Method 1613B [Method 1613B]

Approvals (Signature/Date):

Robert Hrabak — 8/7/17
Technical Manager

Joe Schairer — 8/8/17
Health & Safety Manager / Coordinator

Lisa Stafford — 8/15/2017
Quality Assurance Manager

Crystal Pollock — 8.16.17
Laboratory Director

1. SCOPE AND APPLICATION

1.1. The procedures outlined within this SOP are appropriate for the preparation of samples for the determination of 2,3,7,8-TCDD/TCDF and tetra-through-octa chlorinated dibenzo-p-dioxins and dibenzofurans associated with the Clean Water Act (as amended 1987); the Resource Conservation and Recovery Act (as amended 1986); and the Comprehensive Environmental Response, Compensation and Liability Act (as amended 1986). Specificity is provided for determination of the seventeen 2, 3, 7, 8-substituted polychlorinated dibenzo-p-dioxins (PCDD) and polychlorinated dibenzofurans (PCDF). See Table 1 for list of analytes. Analysis is by SOP WS-ID-0007.

1.2. When undertaking projects for the Department of Defense (DOD) and/or the Department of Energy (DOE), the relevant criteria in Policy WS-PQA-021, "Federal Program Requirements", must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. Stable isotopically labeled analogs of 15 of the PCDDs and PCDFs are added to each sample prior to extraction. Samples containing coarse solids are prepared for extraction by grinding or homogenization, as needed. Drinking water samples are extracted with methylene chloride using separatory funnel procedures. Non drinking water type samples are filtered and then extracted with methylene chloride using separatory funnel procedures; the particulates from the water samples, soils, and other finely divided solids are extracted using a combined Soxhlet (or equivalent) extraction/Dean-Stark azeotropic distillation with toluene (Reference 7, Method 1613). Prior to cleanup and analysis, the extracts of the filtered water and the particulates are combined.

2.1.1. Solid samples may also be extracted using Microwave Assisted Extraction (MAE), as described in Section 11.8.

2.2. After extraction, $^{37}Cl_4$-labeled 2, 3, 7, 8, -TCDD is added to each extract to measure the efficiency of the cleanup process. Samples are then permeated through silica gel, acidic alumina, and activated carbon chromatography columns, as needed. High performance liquid chromatography (HPLC), basic alumina, or back extraction with sulfuric acid/water/NaOH/water can be used for further isolation of the 2, 3, 7, 8-isomers or other specific isomers or congeners.

2.3. After cleanup, the extract is concentrated to near dryness and a known amount of recovery standard (Internal Standard as defined in TALS) is added to each extract. The recovery standard contains two $^{13}C_{12}$-labeled analogs ($^{13}C_{12}$-1,2,3,4-TCDD and $^{13}C_{12}$-1,2,3,7,8,9-HxCDD). Each extract is concentrated to a final volume in

tetradecane. The extract is ready for analysis and delivered to the Instrument Group for HRGC/HRMS.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Laboratory Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

3.3. Isotope Dilution Analyte (IDA): An isotope dilution analyte is a ^{13}C-labeled analog of a congener chosen from the compounds listed in Table 2. Isotope dilution analytes are added to all samples including method blanks and quality control samples before extraction, and they are used to quantitate the concentration of the analytes. Fifteen isotope dilution analytes are used in this method. There is one for each of the dioxin and furan homologs (except for OCDF) with the degree of chlorination ranging from four to eight. Additional isotope dilution analytes may be added to act as retention time references, but they are not used for quantitation.

3.4. Target Analyte: analyte of interest.

3.5. Surrogate Standard (SU): A $^{37}Cl_4$-2,3,7,8-TCDD analog that is added to each sample following extraction to measure the efficiency of the cleanup process.

3.6. Internal Standard (IS): Two recovery standards are used to determine the percent recoveries for the IDA. The 13C-1,2,3,4-TCDD is used to measure the percent recoveries of the tetra- and pentachlorinated IDA while 13C-1,2,3,7,8,9-HxCDD is used to determine the recovery of the hexa-hepta- and octachlorinated IDA. 13C-1,2,3,7,8,9-HxCDD also acts as a retention time reference for the unlabeled analog present in sample extracts. They are added to the final sample extract before HRGC/HRMS instrument analysis.

4. INTERFERENCES

4.1. Solvents, reagents, glassware and other sample processing hardware may yield artifacts and/or elevated baselines causing misinterpretation of chromatograms. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required. Where possible, reagents are cleaned by extraction or solvent rinse.

4.2. Proper cleaning of glassware is extremely important because glassware may not only contaminate the samples, but may also remove the analytes of interest by absorption on the glassware surface.

4.2.1. Glassware should be rinsed with solvent and washed with a detergent

solution as soon after use as practical. Glassware with removable parts, particularly separatory funnels with Teflon stopcocks, must be disassembled prior to detergent washing.

4.2.2. After detergent washing, glassware should be immediately rinsed first with acetone, then with toluene. The toluene rinse is followed by hexane, and then methylene chloride.

4.2.3. Do not bake reusable glassware in an oven as a routine part of cleaning. Baking may be warranted after particularly dirty samples are encountered, but should be minimized, as repeated baking of glassware may cause active sites on the glass surface that will irreversibly absorb PCDDs/PCDFs.

4.2.4. Immediately prior to use, Soxhlet (or equivalent) extraction glassware should be pre-extracted with toluene for approximately 4 hours (1 hour for Soxtherm extraction glassware). Separatory funnels are rinsed sequentially with acetone, toluene, hexane, and methylene chloride.

4.3. All materials used in the analysis shall be demonstrated to be free from interferences by running reference blanks initially and with each sample set (samples started through the extraction process on a given 12-hour shift, to a maximum of 20 samples). The reference matrix blank must simulate, as closely as possible, the sample matrix under test. Reagent water is used to simulate water samples; playground sand, white quartz sand, or sodium sulfate can be used to simulate soils; filter paper or Soxhlet extraction thimbles are used to simulate paper and similar materials; other materials can be used to simulate other matrices.

4.4. Interferences co-extracted from samples will vary considerably from source to source, depending on the diversity of the site being sampled. Interfering compounds may be present at concentrations several orders of magnitude higher than the PCDDs and PCDFs. The most frequently encountered interferences are chlorinated-biphenyls, methoxy biphenyls, hydroxy biphenyl ethers, benzyl phenyl ethers, polynuclear aromatics, and pesticides. Because very low levels of PCDDs and PCDFs are measured by this method, the elimination of interferences is essential. The cleanup steps given in Sections 11.13, 11.14, and 11.15 can be used to reduce or eliminate these interferences.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual (CW-E-M-001) and this document. This work 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. All work must be stopped in the event of a known or potential compromise to the health or safety of an associate. The situation must be reported immediately to a supervisor, the EH&S Staff, or a senior manager.

5.1. Specific Safety Concerns or Requirements

5.1.1. Microwave ovens used for extraction or digestion of samples can create elevated pressure in the extraction/digestion containers. Microwaves used for these processes must be equipped with an automated vapor sensor and shutoff system that automatically shuts down the digestion/extraction process when vapor pressure reaches pre-specified levels. Users must follow procedures in the microwave operator's manual to ensure that the vapor sensor(s) are functional and working properly prior to starting each extraction/digestion batch.

5.1.2. Hearing protection must be worn when using mechanical systems to grind fish or tissue samples.

5.1.3. When dissecting crawfish abdomens with a scalpel, cut from the hand holding the abdomen toward the tail (away from you).

5.1.4. Finely divided dry soils contaminated with PCDDs and PCDFs are particularly hazardous because of the potential for inhalation and ingestion. Such samples are to be processed in a confined environment, such as a hood or a glove box.

5.1.5. The use of vacuum systems during various filtering and/or cleanup steps presents the risk of imploding glassware. All glassware used during vacuum operations must be thoroughly inspected prior to each use. Glass that is chipped, scratched, cracked, rubbed or marred in any manner must not be used under vacuum. It must be removed from service immediately and replaced.

5.1.6. The use of separatory funnels to extract aqueous samples with methylene chloride creates excessive pressure very rapidly. The use of separatory funnels during the partition and back extraction of sample extracts can also create excessive pressure. Initial venting should be done immediately after the sample container has been sealed and inverted. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, and a face shield must be worn over safety glasses or goggles when it is performed. Alternately, the extraction can be performed behind a closed fume hood sash on a mechanical shaker.

5.1.7. When Dean-Stark/Soxhlet clean-ups or extractions are performed overnight or unattended, special precautions must be taken. Open the chiller valves to

the system about 15 minutes before the heating elements are turned on, and check every condenser to ensure that it is cold and functioning properly before turning the heating elements on. Check every condenser again about 15 minutes after turning the heating elements on to ensure that they are still cold and functioning properly. If the system is left operating overnight or unattended for an extended period, the first chemist to come back into the lab must again check every condenser to ensure that it is still cold and functioning properly.

5.1.8. Assembly and disassembly of glassware creates a risk of breakage and cuts. All staff members shall wear Kevlar or MAPA blue latex cut-resistant gloves over chemically resistant gloves when assembling and disassembling glassware.

5.1.9. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex and vinyl gloves provide no protection against most of the organic solvents used in this method. Nitrile gloves must be used. Latex gloves may be used for methanol.

5.1.10. Exposure to chemicals must be maintained as low as reasonably achievable; therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.11. Laboratory procedures such as repetitive use of pipettes, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware present a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Benzene	Flammable Toxic Carcinogen	PEL: 1 PPM TWA ; 5 PPM 15 MIN. STEL	Causes skin irritation. Toxic if absorbed through skin. Causes severe eye irritation. Toxic if inhaled. Vapor or mist causes irritation to mucous membranes and upper respiratory tract. Exposure can cause narcotic effect. Inhalation at high concentrations may have an initial stimulatory effect on the central nervous system characterized by exhilaration, nervous excitation and/or giddiness, depression, drowsiness or fatigue. Victim may experience tightness in the chest, breathlessness, and loss of consciousness.
Cyclohexane	Flammable Irritant	300 ppm TWA	Inhalation of vapors causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. High concentrations have a narcotic effect.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Iso-octane	Flammable Irritant	None established	Inhalation of vapors may cause nausea, headache, dizziness, loss of consciousness, irritation to upper respiratory tract, pain in throat and nose, coughing, wheezing, shortness of breath.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Nonane	Flammable	200 ppm	Primary hazard is flammability.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Sulfuric Acid (1)	Corrosive Oxidizer Dehydrator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Tetradecane	Irritant	None established	Inhalation of vapors may cause difficulty breathing, headache, intoxication and central nervous system damage.
Toluene	Flammable Poison Irritant	200 ppm-TWA 300 ppm-Ceiling	Inhalation may cause irritation of the upper respiratory tract. Symptoms of overexposure may include fatigue, confusion, headache, dizziness and drowsiness. Peculiar skin sensations (e. g. pins and needles) or numbness may be produced. Causes severe eye and skin irritation with redness and pain. May be absorbed through the skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

Miscellaneous Equipment and Materials. The following list of items does not necessarily constitute an exhaustive compendium of the equipment needed for this analytical method.

6.1. Nitrogen evaporation apparatus with variable flow rate.

6.2. Balances capable of accurately weighing to 0.01 g and 0.0001 g.

6.3. Centrifuge.

6.4. Water bath, equipped with concentric ring covers and capable of maintaining temperature control within ± 2ºC.

6.5. Stainless steel or glass containers large enough to hold contents of one-pint sample containers.

6.6. Drying oven.

6.7. Stainless steel spoons and spatulas.

6.8. Pipettes, disposable, Pasteur, 150 mm long x 5 mm ID.

6.9. Pipettes, disposable, serological, 10 mL, for the preparation of the carbon column.

6.10. Reacti-vial, 2 mL, silanized amber glass.

6.11. Stainless steel meat grinder with a 3- to 5-mm hole size inner plate.

6.12. Separatory funnels, 125 mL and 2 L funnels.

6.13. Teflon® boiling chips (or equivalent), washed with DCM before use.

6.14. Chromatographic column, glass, 300 mm x 10.5 mm, fitted with Teflon® stopcock.

6.15. Adapters for concentrator tubes.

6.16. Glass fiber filters, Whatman GF-D, GF-F, GMF150, or equivalent.

6.17. Dean-Stark trap, 5 or 10 mL, with T-joints, condenser and 125 mL flask.

6.18. All-glass Soxhlet apparatus, 500 mL flask.

6.19. Soxtherm extraction apparatus (or equivalent)

6.20. Vacuum extraction device for solid phase extraction, 1 Liter glass funnel with 90mm filter disc holder with a vacuum source, Kontes or equivalent.

6.21. Milestone microwave extraction apparatus (or equivalent)

6.21.1. Automated microwave extractor unit

6.21.2. Plastic extraction vessels with Teflon sample chambers and Teflon pressure release gaskets

6.21.3. 24 position carrousel

6.22. Glass thimble holders for Soxtherm

6.23. Glass beakers for Soxtherm

6.24. Gaskets for Soxtherm

6.25. Glass funnels, sized to hold 170 mL of liquid.

6.26. Desiccator.

6.27. Solvent reservoir (125 mL), Kontes; 12.35 cm diameter (special order item), compatible with gravity carbon column.

6.28. Rotary evaporator with a temperature controlled water bath and vacuum control.

6.29. Turbo evaporator with a temperature controlled water bath and nitrogen pressure control.

6.30. High-speed tissue homogenizer equipped with an EN-8 probe or equivalent.

6.31. pH Strips

6.32. Residual Chlorine Check strips

6.33. Hach 2100Q Portable Turbidimeter, or equivalent. The turbidimeter consists of a nephelometer with a light source for illuminating the sample and a photo-electric detector (photo-diodes). The intensity of light scattered at right angles to the path of the incident light is measured and a direct reading of turbidity (NTU) is generated.

6.34. Glass wool, extracted with methylene chloride, dried and stored in a clean glass jar.

Note: Re-use of glassware should be minimized to avoid the risk of contamination. All glassware that is re-used must be scrupulously cleaned as soon as possible after use.

7. REAGENTS AND STANDARDS

7.1. Column Chromatography Reagents

7.1.1. Silica gel – 70-230 mesh (Fischer Scientific , P/N 130714A or equivalent) -- Activate at least 12 hours at 185-195°C before use. Store at 125-145°C in covered flask or bottle.

7.1.2. Acid Alumina - ICN or equivalent, activated as necessary.

7.1.3. Basic Alumina - ICN or equivalent. No activation required.

7.1.4. Granular carbon/silica gel - Mix 5 g granular carbon and 95 g activated silica gel; (alternatively, prepare carbon/silica gel (5%/95%)). Store at room temperature in a glass container. The first LCS prepared with a new batch of column packing material is the quality control check of the packing materials. Refer to historical control limits before accepting the new batch of material.

7.1.5. 44% H_2SO_4 /silica gel - Mix 44 mL conc. H_2SO_4 and 56 g activated silica gel. Stir and shake until free flowing. Store at room temperature in a glass container.

7.2. Reagents

7.2.1. Sulfuric acid, concentrated, ACS grade, specific gravity 1.84.

7.2.2. 10 N NaOH, certified (Fisher Scientific S5255 or equivalent)

7.2.3. Reagent water is produced by a Millipore nanopure system.

7.2.4. 80 mg/mL Sodium Thiosulfate.

7.2.5. Vegetable Oil (for tissue extraction only), Mazola or other suitable oil, demonstrated to be free of interferences.

7.2.6. Diatomaceous earth – Hydromatrix (Agilent P/N 198004 (4kg) or 198005 (1kg), or equivalent.

7.2.7. Turbidimeter calibration and verification standards: 800 NTU, 0.1 NUT, ICV (5 NTU) and ICB (0 NTU), suitable for use with the Hach Turbidimeter (Section 6.33).

7.3. Desiccating Agent

7.3.1. Sodium sulfate, granular, anhydrous, ACS grade.

7.4. Solvents and Standards:

7.4.1. High-purity, distilled-in-glass or highest available purity: Methylene chloride (DCM), hexane, benzene, methanol, tetradecane, isooctane, toluene, cyclohexane, nonane and acetone.

7.4.2. All calibration, daily IDA, daily surrogate standards, and daily spiking solutions are stable for one year from preparation. After 1 year, solutions may be re-verified. The re-verified solution may be used for an additional

year, or until there is evidence of compound degradation or concentration. The re-verification must be performed using an unexpired, not previously re-verified solution from a second lot or second vendor.

7.4.3. Standards are purchased as solutions or mixtures with certification of their purity, concentration, and authenticity, or prepared from materials of known purity and composition. If the chemical purity is 98% or greater, the weight may be used without correction to compute the concentration of the standard. When not being used, standards can be stored in the dark at room temperature or refrigerated in screw-capped vials with fluoropolymer-lined or Teflon® - lined caps. A mark is placed on the vial at the level of the solution so that solvent loss by evaporation can be detected. If solvent loss has occurred, the solution should be replaced.

7.4.4. Sealed ampules may be used until the manufacturer's expiration date is exceeded. If no expiration date is provided, then the expiration date will be 10 years from the date the ampule is opened. The solvent level should be monitored prior to each use to assure there has been no concentration of the standard over time.

7.5. Stock (Source) Solutions

7.5.1. Preparation – Prepare in nonane or equivalent solvent per the steps below or purchase as dilute solutions (Cambridge Isotope Laboratories, Cambridge, MA, or equivalent). Observe standard safety precautions.

7.5.2. Dissolve an appropriate amount of assayed reference material in solvent. For example, weigh 1 to 2 mg of 2,3,7,8-TCDD to three significant numbers in a 10 mL vial with Teflon® - lined cap.

7.5.3. Stock (Source) standard solutions should be checked for signs of degradation prior to the preparation of calibration or performance test standards. Reference standards that can be used to determine the accuracy of calibration standards are available from Cambridge Isotope Laboratories and may be available from other vendors.

7.5.4. Secondary (Intermediate) standard – Using stock solutions prepare secondary (intermediate) standard solutions if necessary which can be further diluted to create the spiking standard solutions (see below).

7.6. Precision and Recovery Standard (PAR) – Also referred to as the Native Spike solution later in this SOP. Contains all the 2,3,7,8-substituted unlabeled analytes listed in Table 2. Prepare in acetone using the appropriate standards made in Section 7.5 to yield a spiking solution with a concentration of 4.0 ng/ml for the tetra- CDDs/CDFs, 20 ng/ml for the penta-, hexa-, and hepta- CDDs/CDFs, and 40 ng/ml for the octa- CDD/CDF.

7.7. Sample Fortification Solution – Also referred to as the daily Isotope Dilution Analyte (IDA) Solution later in this SOP. From stock solutions or purchased mixtures, prepare this solution in acetone to contain the fifteen isotopically labeled compounds at the nominal concentrations that are listed in Table 2.

7.8. Recovery Standard Solution – Also referred to as the Internal Standard (IS) Solution later in this SOP. This solution contains the labeled compounds $^{13}C_{12}$-1,2,3,4-TCDD and $^{13}C_{12}$-1,2,3,7,8,9-HxCDD. This solution is preferably prepared in tetradecane. See Table 2 for nominal concentrations.

7.9. Cleanup Recovery Standard Solution (CRS) – Also referred to as the Surrogate Standard. Prepare $^{37}Cl_4$-2,3,7,8-TCDD at the concentration shown in Table 2, in isooctane (or toluene).

8. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1. The sample collection, shipping, handling, and chain-of-custody procedures are not described in this document. Sample collection personnel will, to the extent possible, homogenize samples in the field before filling the sample containers. This should minimize or eliminate the necessity for sample homogenization in the laboratory. The analyst should make a judgment, based on the appearance of the sample, regarding the necessity for additional mixing. If the sample is clearly non-homogeneous, the entire contents should be transferred to a glass or stainless steel pan for mixing with a stainless steel spoon or spatula before removal of a sample portion for analysis.

8.2. Samples are collected in amber glass containers following conventional sampling practices (Method 1613). Freely flowing aqueous samples are collected in refrigeration bottles using automatic sampling equipment. Solid samples are collected as grab samples using wide mouth jars.

8.3. Grinding or blending of fish samples.

If not otherwise specified by the client, the whole fish (frozen) should be blended or ground to provide a homogeneous sample. The use of a stainless steel meat grinder with a 3- to 5-mm hole size inner plate is recommended. In some circumstances, analysis of fillet or specific organs of fish may be requested by the client. If so requested by the client, the above whole fish requirement is superseded.

Warning: Hearing protection must be worn when grinding samples.

8.4. Aqueous samples are stored at 0-6ºC in the dark from the time of collection until extraction. A measurement of residual chlorine is performed using EPA Methods 330.4 and 330.5. The residual chlorine will be checked at the laboratory for all aqueous samples. If residual chlorine is present in aqueous samples, the sampler or analyst adds 80 mg of sodium thiosulfate per liter of water.

8.5. Similarly, if stored in the dark at < -10°C, solids, semi-solid, multi-phase, and tissue samples may be stored for up to one year.

8.6. All extracts must be stored capped and in the dark at < -10°C.

8.7. If stored in the dark at < -10°C, sample extracts may be stored for up to one year.

9. QUALITY CONTROL

9.1. The laboratory operates a formal quality assurance program. The minimum requirements of this program consist of an initial demonstration of capability (IDOC), analysis of samples spiked with compounds to evaluate and document data quality, and analysis of standards and blanks as tests of continued performance. The analyst shall make an initial demonstration of the ability to generate acceptable accuracy and precision by this method. This ability is established by generating four laboratory control samples that meet recovery criteria. For each alternative sample matrix, four aliquots of the alternative reference matrix are used.

9.1.1. In recognition of advances that are occurring in analytical technology, and to allow the analyst to overcome sample matrix interferences, the analyst is permitted certain options to improve separations or lower costs of measurements. These options include alternate extraction, concentration, cleanup procedures, and changes in columns and detectors. Unique changes are to be documented using a non-conformance memo. Permanent modifications or additions should be documented using an addendum to this SOP.

9.2. One method blank (MB) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The method blank is an equivalent aliquot of laboratory reagent water (or appropriate alternate matrix) processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a non-conformance memo, then implemented, when target analytes are detected in the method blank above the reporting limit or when surrogate or IDA recoveries are outside control limits. Re-extraction of the blank, other batch QC, and the affected samples are required when the method blank is deemed unacceptable.

Certain programs, such as DOD/DOE QSM, may require a more stringent evaluation of the method blank, for instance, that the blank not contain any analytes of interest at a concentration greater than ½ the lower calibration limit.

9.2.1. If the accompanying samples are aqueous, use distilled water as a matrix. Take the method blank through all steps detailed in the analytical procedure.

9.2.2. Use Sodium sulfate as the method laboratory matrix when solids are extracted. Use a mixture of Sodium sulfate and suitable oil as the matrix

when tissues are extracted. Take the method blank through all steps detailed in the analytical procedure.

9.2.3. The method blank must be spiked prior to extraction with the same amount of 13C-labeled internal standards as is added to samples.

9.2.4. If method blank contamination is present, check solvents, reagents, fortification solutions, apparatus and glassware to locate and eliminate the source of contamination before any further samples are extracted and analyzed.

9.2.4.1. OCDD is a ubiquitous laboratory contaminant. A method blank and the associated samples are deemed acceptable if the OCDD concentration is less than 5 times the specified reporting limit. Flag data appropriately. The analyst is expected to investigate and eliminate potential sources of systematic contamination.

9.2.4.2. If a target analyte is detected in the blank but the associated samples are ND (not detected), then the data may be reported, unless otherwise directed by the client. Note the action in the narrative.

9.2.4.3. If a target analyte is detected in the blank, but the concentration of the contaminant in the samples is greater than 10 times the blank concentration, then the data may be reported, unless otherwise directed by the client. Note the action in the narrative.

9.2.4.4. Method blank contamination of non-target analytes (i.e., non-2,3,7,8-substituted isomers) does not require corrective action, but a non-conformance memo is generated explaining the contamination and impact on sample data. “Totals” concentrations are not flagged to reflect blank contamination because totals are considered estimated values.

9.2.4.5. If the method blank is contaminated with any 2,3,7,8-substituted target analyte above the lower calibration limit (other than the exception noted above for OCDD), and the associated sample contains a positive result for that same analyte above the LCL and less than 10 times the blank concentration. Re-extraction of the associated sample is required, unless otherwise stipulated by the client, where the occurrence shall be documented in the non-conformance memo.

9.2.5. If new batches of reagents or solvents contain interfering contaminants, purify or discard them.

9.3. A Laboratory Control Sample (LCS) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The LCS is an aliquot of laboratory matrix (e.g. water, sodium sulfate, etc.) spiked prior to extraction with analytes of known identity and concentration. The LCS is also spiked prior to extraction with the same amount of $^{13}C_{12}$-labeled IDA as added to the method blank and samples. The LCS must be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, other batch QC and all associated samples are required if the LCS is deemed unacceptable. Update the accuracy assessment for each analyte on a periodic basis. Keep control charts for each matrix.

9.4. A second control sample must be prepared for batches containing drinking water samples for the State of Arizona. This will be identified as the laboratory fortified blank (LFB) or low-level LCS (LLCS) to differentiate it from the LCS. The LFB is an aliquot of water spiked prior to extraction with analytes at the minimum reporting level (MRL). This level is 5 pg/L for 2,3,7,8-TCDD. The LFB must be prepared with each batch containing drinking waters or once per day, whichever is less. The LFB is also spiked prior to extraction with the same amount of $^{13}C_{12}$-labeled IDA as added to the method blank and samples. The LFB must be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, other batch QC and all associated samples are required if the LLCS is deemed unacceptable. Update the accuracy assessment for each analyte on a periodic basis. Keep control charts for each matrix.

9.5. The assessment of matrix effects on method performance, as required by NELAP, is met in Method 1613, as in all isotope dilution techniques, with the use of isotopically labeled compounds. These isotopically labeled compounds are analogs of target analytes and are spiked into each sample. Therefore, matrix effects on method performance can be judged by the recovery of these analogs. Sample analysis acceptance is controlled by the performance of these analogs in each sample. A Matrix Spike/Matrix Spike Duplicate (MS/MSD or MS/SD) pair is extracted at the client's request only and is not required by Method 1613B. An MS/MSD pair are aliquots of a selected field sample spiked with analytes of known identity and concentration. The MS/MSD are also spiked prior to extraction with the same $^{13}C_{12}$-labeled IDA standards as the method blank, laboratory control sample, and field samples. The MS/MSD pair shall be processed in the same manner and at the same time as the associated samples. The results obtained from the MS and MSD samples (percent recovery and concentrations of 2,3,7,8-substituted PCDDs/PCDFs) should agree within 50 percent relative difference. Report all results, flag any outliers, and file a non-conformance memo for any outliers. Re-extraction is not required when a MS/MSD pair fail to meet

one or more QC criteria. An LCS is extracted to show precision of the extraction and analysis process. (An LCS/LCSD pair may be requested by the client, in which case, these usually replace requests for an MS/MSD pair)

9.6. Duplicates – Upon client request, duplicates may be processed. Locate the sample specified for duplicate analysis, and prepare and analyze a second 10 g soil or sediment sample portion or 1 L water sample, or an appropriate amount of the type of matrix under consideration. A duplicate injection of a sample extract may be performed at the clients request to display instrument precision. The results of the laboratory duplicates (percent recovery and concentrations of 2,3,7,8-substituted PCDD/PCDF compounds) should agree within 50 percent relative difference. Report all results and flag any outliers, re-extraction is not required when RPD limits are not met.

9.7. Field Blanks – Each batch of samples may contain a field blank sample of nominally uncontaminated soil, sediment or water that is to be processed for analysis. Treat this sample as all others.

9.8. Recommended Corrective Actions and Troubleshooting Steps

9.8.1. If possible, verify that no error was made while weighing the sample portions.

9.8.2. Review the analytical procedures with the performing laboratory personnel.

10. CALIBRATION AND STANDARDIZATION

10.1. On a daily basis, calibrate any balance to be used in accordance with SOP WS-QA-0041.

10.2. When using autopipettors, follow calibration guidelines in accordance with SOP WS-QA-0004.

11. PROCEDURE

The sample preparation process involves modifying the physical form of the sample so that the PCDDs and PCDFs can be extracted efficiently. In general, the samples must be in a liquid form or in the form of finely divided solids in order for efficient extraction to take place. Samples containing a solid phase and samples containing particle size larger than 1mm require preparation prior to extraction. Because PCDDs/PCDFs are strongly associated with particulates, the preparation of aqueous samples includes filtering, Soxhlet extraction, and separatory funnel shakeout. For samples expected or known to contain high levels of the PCDDs and/or PCDFs, the smallest sample size representative of the entire sample should be used, and the sample extract should be diluted, if necessary.

11.1. Preparation of aqueous samples (liquid/liquid extraction).

The extraction procedure for aqueous samples involves filtering the sample, extracting the particulate phase and filtrate separately, and combining the extracts for analysis. The aqueous portion is extracted by shaking with methylene chloride in a separatory funnel. The particulate material is extracted using Soxhlet with Dean-Stark or Soxtherm extraction.

Note- If the samples are noted as drinking water and no visible particulates are present, sample filtration is not required.

Note – Drinking water samples must be checked for pH using pH strips. The pH must be adjusted to pH 7-9 with sulfuric acid, as necessary. If any sample is below this range, the pH must be adjusted with sodium hydroxide.

Note- Drinking wter samples must be checked for residual Chlorine using Chlorine test strips. The Chlorine level must be less than 0.5 mg/L. If the sample fails the residual Chlorine test, add 1mL portions of 80mg/mL Sodium Thiosulfate unitl the sample passes the Residual Chlorine test.

Note- If the samples are noted as Arizona drinking water, the samples must be screened for Turbidity. If the turbidity is found to be greater than 1.0 NTU, the analyst will notify the project manager to see how to proceed with the samples. Please refer to Section 11.2 for Turbidity procedure.

11.1.1. Mark the original level of the sample on the sample bottle for reference. Weigh the sample in the bottle on the top loading balance to ±1g.

11.1.1.1. For leachate samples (including an aliquot of the leachate blank), measure 200 mL of leachate into a clean 1 L sample bottle. Add approximately 800 mL of reagent water to the leachate. The sample volume in TALS for leachates is 200 mL.

11.1.2. For each sample or sample set (to a maximum of 20 samples) to be extracted during the same twelve hour shift, place two 1.0 liter aliquots of reagent water into two clean 1 liter sample bottles. One aliquot will serve as the blank and the other will serve as the laboratory control sample (LCS).

11.1.2.1. Into the blank, add 1mL of daily isotope dilution analyte (IDA) standard.

11.1.2.2. Into the LCS, add 1 mL of daily IDA standard and 50 µL of the native spike.

11.1.2.3. If the sample is a drinking water then all spike volumes listed in 11.1.2.1 and 11.1.2.2 are reduced by ½.

11.1.3. For each sample, add 1 mL of IDA standard into 2 mL of acetone. Spike the diluted solution into the sample bottle. Cap the bottle and mix the sample by carefully shaking. Allow the sample to equilibrate.

11.1.4. Assemble a Buchner funnel on top of a clean 1 L filtration flask. Apply a vacuum to the flask, and pour the entire contents of the sample bottle through a glass fiber filter in the Buchner funnel, swirling the sample remaining in the bottle to suspend any particulates.

Warning: The use of a vacuum system creates the risk of glassware implosions. Carefully inspect all glassware before each use, and discard or repair any that is cracked, chipped, scratched or otherwise damaged.

11.1.4.1. Rinse the sample bottle twice with 5 mL of DCM. Shake to transfer any remaining particulates onto the filter.

11.1.4.2. Rinse any particulates off the sides of the Buchner funnel, with small quantities of DCM.

11.1.4.3. Pour aqueous sample back into sample container (if not extracted immediately).

PERFORM THE EXTRACTION USING THE PROCEDURES IN SECTION 11.3

11.2. Turbidity

11.2.1. For best results, it is recommended that the turbidimeter be left on continuously. At minimum, the instrument should be on one hour before analysis.

11.2.2. Standards and samples should be allowed to equilibrate to room temperature prior to analysis. This will prevent condensation on the turbidity cuvette.

11.2.3. All readings are to be recorded in the turbidity screening logbook.

11.2.4. The instrument should be calibrated daily or when in use, as follows:

Note- All calibration levels and standard lots must be recorded in the turbidity screening logbook.

11.2.4.1. Press **<VERIFY CAL>**. Initial value for calibration is set at 800 NTU with an acceptance criteria of 10%.

11.2.4.2. Place 800 NTU blank into machine and press **<READ>**.

11.2.4.3. After reading, press **<DONE>**.

11.2.4.4. Press **<CANCEL>**. This will take you back to the initial screen.

11.2.4.5. Since the machine does not allow you to set the next calibration value to 0.1 NTU, you can place the 0.1 NTU blank into the machine and press **<READ>**. Reading must be between 90%-110.

11.2.4.6. Press **<DONE>**. Place ICV into machine and take reading. Reading must be less than 5.5 NTU.

11.2.4.7. Take ICB reading after reading ICV. Reading must be less than 0.5 NTU.

11.2.5. Mix the samples thoroughly to disperse solids. Wait for air bubbles to disappear. Pour samples into clean turbidimeter vials and record the readings.

Note- Vials must be free of fingerprints and air bubbles to ensure accurate results. Clean the outside surface with Kimwipes if necessary.

11.2.6. Following every 10 samples, analyze a 5.0 NTU ICV. Reading must be less than 5.5 NTU.

11.2.7. Samples that pass the turbidity screening can proceed to extraction.

11.3. Extraction of filtrates

Extract the aqueous samples, blanks, and PAR aliquots according to the following procedure.

11.3.1. Pour the filtered aqueous sample from the sample bottle flask into a 2 L separatory funnel. Rinse the bottle twice with 5 mL of DCM and add these rinses to the separatory funnel. Add 100 mL DCM to the sample bottle, seal, and shake 60 seconds to rinse the inner surface. Transfer the solvent to the separatory funnel. Weigh the empty bottle on a top loading balance, and compare to weight from Section 11.1.1 to determine initial volume.

11.3.2. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for 2 minutes with periodic venting.

Warning: Separatory funnel extraction with DCM is a high-risk activity. Pressure may build rapidly in the funnel. It should be vented after several seconds of shaking, and often enough to prevent build-up of pressure. Chemist performing separatory funnel extraction must wear a face shield over their safety glasses/goggles. Alternatively, the extraction can be performed behind a closed fume hood sash.

11.3.3. Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between the layers is more than one-third the volume of the solvent layer, employ mechanical techniques to complete this operation (e.g., a glass stirring rod or centrifuge). Drain the DCM extract into the original sample container.

NOTE: Experience with aqueous samples high in dissolved organic materials (e.g., paper mill effluents) has shown that acidification of the sample prior to extraction may

reduce the formation of emulsions. Mechanical techniques may still be necessary to complete the phase separation. Refer to Section 11.4 for techniques to decrease or eliminate emulsions.

11.3.4. Extract the water sample two more times using 100 mL of fresh DCM each time. Drain solvent into original container. After the third extraction, rinse the separatory funnel with at least 20 mL of fresh DCM, and drain solvent into original container.

11.3.5. Prepare a funnel plugged with rinsed glass wool and half filled with sodium sulfate. Filter DCM from Section 11.3.4 through funnel and collect directly into 500 mL round bottom flask. Rinse the Na_2SO_4 with DCM and then add as a keeper

11.3.6. Extract the filter portion by Dean-Stark/Soxhlet or Soxtherm according to Section 11.6.

11.3.7. Perform macro concentration as detailed in Section 11.16.

11.3.8. If the sample was filtered and the particulate fraction was also extracted, combine the concentrated extracts of filtrate and particulate prior to proceeding with cleanup or micro concentration steps.

11.3.9. Proceed to Section 11.12, Extract Cleanup.

11.4. Decreasing or Eliminating Emulsions

There are several useful methods to decrease or eliminate emulsion in aqueous samples when extracting with DCM. These methods may include stirring with a pipette to manually breakup the emulsions or to transfer the sample into centrifuge tubes and centrifuge at approximately 3000 RPM.

The most useful method is to use a 10N NaOH solution to change the pH enough to disrupt the emulsion phase, which works 90% of the time. The following procedure describes how to prepare and use the solution to decrease or eliminate emulsions in aqueous samples during the liquid/liquid extraction step.

11.4.1. Check the pH of the sample to make sure pH is between 3 and 7. If pH is greater than 7, then consult supervisor and client for instructions.

11.4.2. Pour approximately 10 mL of the 10N NaOH into a 1 L amber glass bottle (AGB).

11.4.3. Drain the sample with the emulsion from the 2 L separatory funnel into the 1 L AGB and let it stand.

11.4.4. Pour the solution with DCM back into the same 2 L separatory funnel and

drain the DCM phase through Na_2SO_4 into a 500 mL round bottom. Empty the aqueous waste into the LLE waste drum.

11.5. Preparation of pulp, paper and tissue samples which are subject to grinding, homogenization or blending.

The method of reducing particle size to less then 1 mm is matrix dependent. In general, hard particulates can be reduced by grinding with a mortar and pestle. Softer particulates can be reduced by grinding in a Whiley mill or meat grinder, by homogenization, or by blending.

11.5.1. The grinding, homogenization, or blending procedures shall be carried out in a fume hood to prevent particles from contaminating the work environment.

11.5.2. Grinding- Tissue samples, certain papers and pulps, slurries, and amorphous solids can be ground in a heavy-duty grinder. In some cases, reducing the temperature of the sample to freezing or to dry ice or liquid nitrogen temperatures can aid in the grinding process. Do not allow the sample temperature to exceed 50ºC.

11.5.3. Homogenization or blending – Particles that are not ground effectively, or particles greater than 1 mm in size after grinding, can often be reduced in size by high-speed homogenization or blending.

11.5.4. Extract the aliquots using the Dean-Stark/Soxhlet or Soxtherm procedures in Section 11.6.

11.6. Dean-Stark/Soxhlet (or alternative Soxtherm [Section 7] or Microwave Assisted Extraction [Section 8]) extraction of solids – Extract the solid samples, particulates, blanks, and PAR aliquots using the following procedure.

11.6.1. Pre-extract the glassware by heating the flask until the toluene is boiling. When properly adjusted, 1-2 drops of toluene per second will fall from the condenser tip into the receiver. Extract the apparatus for a minimum of four hours.

WARNING: Open the chiller supply valves about 15 minutes before turning on the heating element and ensure that all of the condensors are cold before you turn the heating element on. Check all of the condensors about 15 minutes after starting the heating process to ensure that they are still cold and functioning properly. If this cleaning cycle is to be left unattended (e.g., overnight) the first chemist to arrive the next morning is to check all condensers to ensure that they are still cold and functioning properly.

11.6.2. After pre-extraction, cool and disassemble the apparatus.

11.6.3. Weigh a well-mixed aliquot of each sample, generally 2 –10 g, (of the same

matrix type) into a clean Soxhlet thimble. If the material to be extracted is the particulate matter from the filtration of an aqueous sample, add the filter paper to the thimble also.

11.6.4. Spike 1.0 mL of the IDA standard into the sample aliquot(s).

11.6.4.1. Do not spike filter/particulate samples from the aqueous filtration step, because theses extracts will be combined with those of the aqueous portion, and the aqueous portion is spiked already.

11.6.5. For each sample or sample set (to a maximum of 20 samples) to be extracted during the same 12-hour shift, weigh two aliquots of the appropriate reference matrix into clean Soxhlet thimbles. One aliquot will serve as the blank and the other will serve as the laboratory control sample (LCS).

11.6.5.1. Into the blank, add 1 mL of daily IDA standard.

11.6.5.2. Into the LCS, add 1 mL of daily IDA standard and 50 µL of the native spike.

11.6.5.3. Normal solid samples are spiked with the appropriate daily IDA standard (the LCS and MS/SD samples are spiked with the daily PAR solution) **after the samples are loaded into the Soxhlet (or Soxtherm) apparatus**.

11.6.6. Reassemble the pre-extracted apparatus and add a fresh charge of toluene (250 ml – 350 ml) to the receiver and reflux flask.

WARNING: Open the chiller supply valves about 15 minutes before turning on the heating element and ensure that all of the condensors are cold before you turn the heating element on. Check all of the condensors about 15 minutes after starting the heating process to ensure that they are still cold and functioning properly. If this cleaning cycle is to be left unattended (e.g., overnight) the first chemist to arrive the next morning is to check all condensers to ensure that they are still cold and functioning properly.

11.6.7. Apply power to the heating mantle to begin refluxing. Adjust the reflux rate to match the rate of percolation of sand and silica beds until water removal lessens the restriction to toluene flow. Check the apparatus for foaming frequently during the first 2 hours of extraction. If foaming occurs, reduce the reflux rate until foaming subsides.

11.6.8. Drain the water from the receiver if the receiver fills with water. Check and drain when necessary.

Note: If the receiver holds 10 mL of liquid, and 20 g of a approximately 10% solid sample is being extracted, then approximately 9 mL of water will end up in the

receiver. In this case, the receiver will not need to be emptied (insufficient liquid to overflow), but it should be checked. If the sample amount is 50, and the percent solids is still 10%, then 45 mL of water will end up in the receiver. In this case, frequent checking is required, and the receiver will need to be emptied at least 5 times.

11.6.9. Reflux the sample for a minimum of 16 hours. Cool and disassemble the apparatus.

11.6.10. Remove the distilling flask. Drain the liquid from the Dean-Stark receiver.

11.6.10.1. The Soxtherm unit will drain all purged solvent and water into the waste solvent reservoir.

11.6.11. For solid samples, the extract must be concentrated to approximately 10 mL prior to additional cleanup. For the particulates filtered from an aqueous sample, the extract must be concentrated prior to combining with the extract of the filtrate. Therefore, add approximately 100 µL of C_{14} to the round bottom flask.

11.6.12. Proceed to Section 11.12, Extract Cleanup.

11.7. SoxTherm Extraction

11.7.1. Prior to loading samples, run the system through a cleaning cycle (approximately 1 hour).

11.7.2. After pre-extraction, cool and disassemble the apparatus.

11.7.3. Place the thimble into the SoxTherm apparatus.

11.7.4. Reassemble the pre-extracted apparatus and add a fresh charge (150 mL) of toluene to the apparatus.

11.7.5. Spike according to Section 11.7.5.1, 11.7.5.2, and 11.7.5.3.

11.7.6. Program the system to boil for 1 hour, and reduce the toluene volume by 70 – 90 mL (volume < volume of the thimble).

11.7.7. Continue the extraction for one hour fifteen minutes, reducing the toluene volume by another 15 mL. The SoxTherm unit will have between 5 mL and 20 mL of solvent following extraction. Transfer the extract from the extract beaker to a 35 mL culture tube and add approximately 100 uL C_{14} to each extract.

11.7.8. Proceed to Section 11.12, Extract Cleanup.

11.8. Microwave Assisted Extraction (MAE)

WARNING: Microwave ovens used for extraction or digestion of samples can create elevated pressure in the extraction/digestion containers. Follow procedures in the operator's manual to ensure that the vapor sensor(s) are functional and working properly prior to starting each extraction/digestion batch.

11.8.1. Prior to loading samples, run the system through a cleaning cycle (approximately 35 minutes) using approximately 30 mL 1:1 Toluene:Acetone and the following program (cleaning run.mpr):

Step	Time (min)	Power (W)	Temperature (℃)
1	10	1200	145
2	25	1200	145
3	10	0	ambient

11.8.2. After pre-extraction, cool and disassemble the apparatus.

11.8.3. Weigh 10 g (or required sample amount) of each field sample and MS/MSD if required into the cleaned Teflon chambers and mix with approximately 5 g of diatomaceous earth. MB, LCS and if required LCSD aliquots are made using 10g of Na_2SO_4 and 5g of diatomaceous earth.

11.8.4. Add 1 mL of deionized water to each sample and QC in Teflon sample chamber. The number of sample chambers should match the number of field samples and QC samples.

11.8.5. Spike according to Section 11.7.5.1, 11.7.5.2, and 11.7.5.3.

11.8.6. Add 30 mL of 1:1 Toluene:Acetone to each Teflon sample chamber.

11.8.7. Add the Teflon cap with the Teflon pressure release valve on top of each chamber and place the setup into the plastic pressure vessel and screw on the cap. Into one vessel (that has a special Teflon pressure release valve) carefully add the monitoring probe casing into the top of the chamber. Be careful to not force this probe casing as it is very fragile.

11.8.8. Place each extraction vessel into a pressure reactor. Screw on the pressure cap/safety lid. The pressure cap should be hand tightened until the sealing valve is flush with the top of the cap.

11.8.8.1. For the representative sample created in 11.9.7.2, add the protection foil and appropriate safety lid.

11.8.9. Add flared extraction vessel covers to each extraction vessel containing sample and QC aliquots.

11.8.9.1. It is important that each cover fits snugly to ensure a proper seal. The cover should not slide easily or loosely inside the extraction vessel, but should require some finger pressure to insert firmly. A cover flaring tool should be used.

11.8.9.2. For the visually wettest sample, add the thermowell liner into the extraction vessel cover to create the representative sample that the ATC temperature sensor can be inserted into.

11.8.10. Place all the extraction vessels into the rotor so that the pressure-release valves are facing outside of the rotor on the outside ring and inside toward the center on the inside ring.

WARNING: Microwave ovens used for extraction or digestion of samples can create elevated pressure in the extraction/digestion containers. Follow procedures in the operator's manual to ensure that the vapor sensor(s) are functional and working properly prior to starting each extraction/digestion batch.

11.8.10.1. Place the rotor in the microwave oven and insert the ATC temperature sensor into the representative sample (11.9.7.2 and 11.9.8.1).

11.8.10.2. Close the microwave oven and start the appropriate extraction profile

11.8.10.3. A 10 minute ramp from 25°C to 115° C, hold for 30 minutes at 115°C, followed by a 10 minute cool down to ambient temperature.

11.8.11. Run the following program for sample extraction:

Step	Time (min)	Power (W)	Temperature (^{o}C)
1	10	1200	Ramp to 115°
2	30	1200	115°
3	10	0	(ambient)

11.8.12. Following extraction filter the extract through Na_2SO_4 and collect in a round bottom flask, rinse twice more with approximately 5 mL to 10 mL of toluene for a total of about 50 mL.

11.8.13. Add approximately 100 uL C_{14} to each round bottom flask if % lipid is not needed.

11.8.14. Proceed to Section 11.16.

11.9. Tissue samples follow the same solid extraction procedure described in Section 11.3, except where % Lipids are needed, C_{14} is not added to the sample extract (only to the

method blank and LCS). The matrix for the method blank consists of 9 g of pre-cleaned sodium sulfate and 1 g of a suitable oil.

11.9.1. Weigh the concentration vessel with label and boiling chips prior to extraction and record on the benchsheet.

Note: If Soxtherm was used, weigh the 35 mL culture tube.

11.9.2. Weigh the appropriate sample size into the glass fiber thimble and mix thoroughly with sodium sulfate. Extract according to Section 6. Concentrate the sample using a rotary evaporator until toluene has been completely removed. Add approximately 25 mL hexane and repeat the concentration via rotary-evaporation to ensure only lipids are present.

Note: If Soxtherm was used, transfer the sample to 35 mL culture tubes and turbo-evaporate to near dryness. Add approximately 5 mL hexane and repeat the concentration via turbo-evaporation until only lipids are present.

11.9.3. Dry the concentration vessel and let stand at room temperature. Re-weigh the concentration vessel and record on the benchsheet.

11.9.4. %Lipids is calculated by the following:

$$\%Lipids = \frac{Final\ Vessel\ Weight - Initial\ Vessel\ Weight}{Sample\ Size} \times 100$$

11.9.5. Proceed to Section 11.10, Wipe Extractions, Soxhlet Method.

11.10. Wipe Extractions - Jar Shake Method

11.10.1. Place a pre-cleaned wipe in a French Square jar. This will be the method blank aliquot. Place an additional pre-cleaned wipe in yet another French Square. This will be the LCS aliquot.

11.10.2. Transfer each wipe sample and all accompanying liquid into separate French Square jars or appropriate sized containers

Note: If the container used to deliver the wipe to the laboratory can contain 100 ml of solvent, then the container can be used for the extraction.

11.10.3. Spike all samples, method blank and LCS with an appropriate amount of daily IDA standard. Additionally, spike the LCS with the appropriate amount of native standard.

11.10.4. Add 100 mL of toluene to each jar and secure with a Teflon-lined cap. Place the closed container onto the flatbed shaker and secure in place. Turn the shaker on such that the shaking motion is aggressive enough to move the liquid through the wipe. Shake for 4 hours.

11.10.5. Filter each sample through a filter funnel with a glasswool plug. Capture the sample into a 500 ml round bottom flask. Add approximately 100 µL of tetradecane and concentrate to approximately 100 µL on a rotary evaporator or TurboVap.

11.10.6. Add approximately 1 mL of hexane or toluene to rinse the sides of the round bottom flask. Using a pipette, withdraw the sample from the round bottom flask and transfer the liquid into a test-tube. Use additional amounts of solvents to rinse the flask. Transfer all the liquid into the test-tube. Insure that all traces of sample in the round bottom flask have been thoroughly rinsed from all surfaces. Bring the sample volume to 8.0 mL or 10.0 mL (or appropriate volume) with the addition of rinse solvent.

11.10.7. Upon completion of the rinsing, cap the test tube and shake vigorously. Take ½ of each sample (or an appropriate amount deemed from the client or other method) and transfer to a culture tube. Archive the remaining sample for future use.

11.10.7.1. If only one analysis is required, then ½ of the sample is archived and the other half is analyzed.

11.10.7.2. If two analyses are required, then 1/3 is archived, 1/3 is used for one test, and 1/3 is used for the second test. For additional analyses, adjust the fractions accordingly.

11.10.8. Proceed to section 11.12.

11.11. Wipe Extractions – Soxhlet/Soxtherms Method

11.11.1. Place a pre-cleaned wipe in a pre-cleaned Soxhlet extraction apparatus. This will be the method blank aliquot. Place an additional pre-cleaned wipe in yet another pre-cleaned Soxhlet/Soxtherm extraction apparatus. This will be the LCS aliquot.

11.11.2. Transfer each wipe sample and all accompanying liquid into separate precleaned Soxhlet/Soxtherm extraction apparatus.

11.11.3. Spike all samples, method blank and LCS with an appropriate amount of internal standard. Additionally, spike the LCS with the appropriate amount of native standard.

11.11.4. Charge the Soxhlet with approximately 300 mL toluene or 150 mL toluene for Soxtherms, and completely assemble the Soxhlet/Soxtherm apparatus. For Soxhlet, follow procedure in Section 11.6.6 to 11.6.11. For Soxtherm follow procedure outlined in Section 11.7.

WARNING: Open the chiller supply valves about 15 minutes before turning on the heating element and ensure that all of the condensors are cold before you turn the heating element on. Check all of the condensors about 15 minutes after starting the heating process to ensure that they are still cold and functioning properly. If this cleaning cycle is to be left unattended (e.g., overnight) the first chemist to arrive the next morning is to check all condensers to ensure that they are still cold and functioning properly.

11.11.5. After cycling is complete, turn the heat off and allow the glassware to cool. Remove the round bottom flask. Add approximately 100 µL of tetradecane and concentrate to approximately 100 µL on a rotary evaporator or TurboVap.

11.11.6. Add approximately 1 mL of hexane or toluene to rinse the sides of the round bottom flask. Using a pipette, withdraw the sample from the round bottom flask and transfer the liquid into a test tube. Use additional amounts of solvents to rinse the flask. Transfer all the liquid into the test tube. Insure that all traces of sample in the round bottom flask have been thoroughly rinsed from all surfaces. Bring the sample volume to 8.0 mL or 10.0 mL (or appropriate volume) with the addition of rinse solvent.

11.11.7. Upon completion of the rinsing, cap the test tube and shake vigorously. Take ½ of each sample (or an appropriate amount deemed from the client or other method) and transfer to a culture tube. Archive the remaining sample for future use.

11.11.7.1. If only one analysis is required, then ½ of the sample is archived and the other half is analyzed.

11.11.7.2. If two analyses are required, then 1/3 is archived, 1/3 is used for one test, and 1/3 is used for the second test. For additional analyses, adjust the fractions accordingly.

11.11.8. Proceed to section 11.12, Extract cleanup..

11.12. Extract cleanup

11.12.1. Cleanup may not be necessary for relatively clean samples (e.g., drinking water). If a cleanup procedure is required, the analyst may use any or all of the procedures below or any other appropriate procedure. Before using a cleanup procedure, the analyst must demonstrate that the requirements of Section 13 can be met using the cleanup procedure. Cleanup may include: back extraction with H_2S0_4 and H_2O, acid and neutral silica gel and alumina are used to remove nonpolar and polar interferences, and/or 5% Carbon/Silica is used to remove nonpolar interferences.

11.12.2. Spike 1.0 mL of the surrogate (SU)) prior to any cleanup into the round bottom flasks containing the samples and QC extracts. Concentrate samples according to Section 11.17 for the macro-concentration of round bottom flasks or Section 11.18 for the micro-concentration of culture tubes.

11.13. Optional Acid Cleanup (if required). This extract clean-up procedure is employed if the extract is dark in color, smells of hydrocarbons or is known to contain high levels of organic materials.

WARNING: Do not deliberately smell the extract. If there is a strong odor, close the fume hood sash to a smaller opening, move the work further back into the hood and ensure that the hood is operating properly.

11.13.1. Partition the extract against approximately 50 mL of concentrated sulfuric acid. Shake for 2 minutes, periodically venting into a hood. Remove and discard the acid layer. Repeat the acid washing until no color is visible in the aqueous layer, to a maximum of 4 washings. (Minimize contact time between the extract and the acid to prevent dehydration of the PCDDs and PCDFs).

WARNING: The use of separatory funnels during these cleanups is a high-risk activity. A face shield must be worn over safety glasses or goggles.

11.13.2. Repeat the partitioning against DI water and discard the aqueous layer. Most samples will not need the base partition and Sections 11.13.3 and 11.13.4 can be skipped. Consult with department manager or lead analyst if unsure.

11.13.3. Partition the extract against 50 mL of sodium hydroxide in the same way as with the acid. Repeat with base washing until no color is visible in the aqueous layer, to a maximum of four washings.

11.13.4. Repeat the partitioning against DI water and discard the aqueous layer.

11.13.5. Pour each extract through a filling funnel column containing 7 to 10 cm of

anhydrous sodium sulfate. Rinse the separatory funnel with 30-50 mL of hexane and pour through the filling funnel. Collect and cleanup the samples and QC aliquots.

11.13.6. See Section 11.16 for macro-concentration procedure.

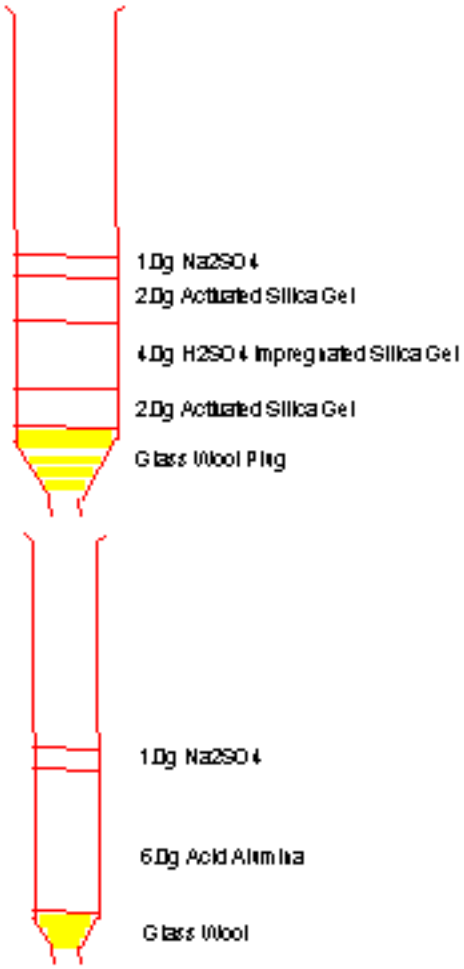
11.14. IFB Column Cleanup

The most frequently used clean-up procedure is the IFB column clean-up.

Exhibit A

IFB COLUMN CLEANUP

Use 20 mm column for upper column
Use 15 mm column for lower column



11.14.1. Pre-rinse both columns with hexane - 40 mL Top and 20 mL Bottom.

11.14.2. Put one column above the other.

11.14.3. Add extract to the top column - rinse the extract vessel 2 times with 1 mL each of hexane and add to column.

11.14.4. Elute the top column directly onto the bottom column with 50 mL hexane.

11.14.5. Disconnect the upper column from the lower column and elute the lower column with 10 mL of 20/80% DCM/hexane. Discard the 20/80% DCM/hexane.

11.14.6. Elute the lower column with 30 mL of 65/35% DCM/hexane into a 35 mL culture tube and save as the final extract eluate.

11.15. If further clean-up is necessary then use this procedure. Carbon Column Cleanup - Prepare an Activated Carbon on Silica Gel column as described below. The carbon column is used to remove non-polar interferences, DPE and should be used on sediment samples.

11.15.1. Use a pre-cut D2 column made from vendor

11.15.2. Push a glass wool plug down to the 3 inch mark.

11.15.3. Add 1 g of 5% Activated Carbon/silica and top with another glasswool plug.

11.15.4. Pre-elute with 5 mL 1:1 DCM: Cyclohexane. Direction "A" (carbon on lower end of the column)

11.15.5. Turn column over to the “B” direction (carbon on the top end of the column) and pre-elute with 5 mL 1:1 DCM: Cyclohexane.

11.15.6. Discard pre-eluates.

11.15.7. Dilute extract to 1 mL with hexane and transfer to the column.

11.15.8. Rinse sample vial onto the column with 2 x 2 mL 1:1 DCM: Cyclohexane.

11.15.9. Elute with: 6 mL 1:1 DCM:Cyclohexane then with 5 mL 75:20:5 DCM:MeOH:Benzene.

11.15.10. Discard eluates.

11.15.11. Turn the column over to the “A” Direction and elute with 25 mL toluene.

11.15.12. See Section 11.17 for micro-concentration procedure.

11.15.13. See Section 11.19 for N_2 concentration procedure.

11.15.14. Collect the sample and proceed to concentration step. 100 µl of tetradecane can be used as keeper if proceeding to IFB cleanup (Section 11.14).

11.16. Macro-concentration

Concentrate the extracts in separate 500 mL round bottom flasks on rotary evaporator.

11.16.1. Assemble the rotary evaporator according to manufacture’s instructions, and warm the water bath (60^{o} C for DCM and 75^{o} C for toluene and hexane). On a daily basis, preclean the rotary evaporator by solvent rinsing. Between samples, 2-3 rinses of toluene followed by a 2-3 mL rinse of hexane should be rinsed down the feed tube into a waste beaker.

11.16.2. Attach the round bottom flask containing the sample extract to the rotary evaporator. Slowly apply vacuum to the system, and begin rotating the sample flask.

11.16.3. Lower the flask into the water bath and adjust the speed of rotation and the temperature as required. At the proper rate of concentration, the flow of solvent into the receiving flask will be steady, but no bumping or visible boiling of the extract will occur.

NOTE: If the rate of concentration is too fast, analyte loss may occur.

11.17. Micro-concentration

Concentrate the extracts in 35 mL culture tubes in a turbo-evaporator. The turbo-evaporator model that the laboratory uses can hold up to 50-35 mL culture tubes. Other turbo-evaporator models can be used that may or may not have the same culture tube sizes and/or capacity. Adjust temperature according to solvent (65ºC for toluene and 45ºC for hexane or hexane/DCM mixtures)

11.17.1. The evaporating times are dependent on sample volume and solvent. The following are examples and can change from sample to sample. Each sample should be checked in intermittent intervals to make sure samples do not go dry.

11.17.2. When evaporating 30 mL toluene, it will normally take approximately 30-50 minutes with the temperature setting described above.

11.17.3. When evaporating 30 mL hexane/DCM, it will normally take approximately 10-20 minutes with the temperature setting described above.

11.18. N_2 concentration

11.18.1. Transfer extracts to a micro concentration vial (test tubes and other small vessels may also be used), and spike extract with 20 µL of internal standard

11.18.1.1. If the sample is a drinking water, spike 10 µL of internal standard.

11.18.2. Fit a glass concentration apparatus (or equivalent – apparatus can also be made of alternative material free from interferences) with multiple ports.

11.18.2.1. If the concentration apparatus has large orifices for each port, an interference free tube can be placed on each tube fitted with a Pasteur pipette to reduce the size of each port.

11.18.3. Apply a gentle stream of N_2 to the samples making sure turbulence is kept to a minimum.

11.18.4. Concentrate the sample to the necessary volume. Standard volume is 20 µL. Standard volume for drinking water is 10 µL. Alternative final volumes may be used with client request and/or approval.

11.18.5. Transfer extract to 2 mL vial with inserts.

12. CALCULATIONS/DATA REDUCTION

12.1. Not applicable

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate, who has been properly trained in its use and has the required experience.

13.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QC files.

14. POLLUTION CONTROL

It is TestAmerica 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 Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.1. Standards should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards that must be discarded.

14.2. The use of Roto-vaps and Turbo-vaps for some concentration steps, rather than Kuderna-Danish reduction, allows extraction solvents to be collected and disposed of rather than released to the atmosphere.

14.3. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.

14.4. Do not allow waste solvent to vent into the hoods. All solvent waste is stored in capped containers unless they are being filled.

14.5. Transfer waste solvent from collection cups (tri-pour and similar containers) to jugs and/or carboys as quickly as possible to minimize evaporation.

15. 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 SOP WS-EHS-0001.

The following waste streams are produced when this method is carried out.

15.1. Extracted aqueous/leachate samples contaminated with methylene chloride are collected at the fume hood in a 5-gallon or smaller carboy. If the samples are not at a neutral pH, add small quantities of sodium bicarbonate to bring the waste to neutral. Stir well. Once neutralized, immediately pour the carboy contents into a blue plastic LLE drum in the H3 closet. When full to between two and six inches of the top, or after no more than 75 days, move the LLE drum to the waste collection area for shipment.

15.2. Miscellaneous disposable glassware, test tubes, syringes, filter disks, chemical rsistant gloves, bench paper and simiilar materials that may or may not be contaminated hazardous. Place contaminated materials into a yellow contaminated lab trash bucket. When the bucket is full or after no more than one year, tie the plastic bab liner shut and put the lab trash into the appropriate steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.3. Extracted soil samples, used sodium sulfate, paper funnel tubes, glass wool, thimbles, and extracted solids contaminated with solvents. Dump these materials into an orange contaminated lab trash bucket. When the bucket is full, or after no more than one year, tie the plastic bag liner shut and put the lab trash into the appropriate steel collection drum in the H3 closet. When the drum if full or after no more than 75 days, move it to the waste collection area for shipment.

15.4. Assorted solvent and methylene chloride waste generated during glassware and sodium sulfate cleaning. Solvent collected during roto-vap/turbo-vap reduction of extracted samples. Collect the waste solvents in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel solvent drum in the H3 closet. When full to between two and six inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment.

15.5. Contaminated sulfuric acid used during extract cleanup. Collect the used sulfuric acid in empty, 2.5-liter, plastic coated jars. When full or after one year, whichever comes first, transfer these jars to the waste collection area for shipment.

15.6. Contaminated sodium hydroxide used during extract cleanup. Collect the used sodium hydroxide in empty, 2.5-liter, plastic coated jars. When full or after one year, whichever comes first, transfer these jars to the waste collection area for shipment.

15.7. Contaminated distilled water used during extract cleanup. Collect the contaminated water in a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the plastic LLE drum in the H3 closet. When full to between two and six inches of the top, or after no more than 75 days, move the plastic drum to the waste collection area for shipment.

16. REFERENCES/CROSS REFERENCES

16.1. EPA Method 1613, Revision B, October 1994.

16.2. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update IV. Method 3546, Microwave Extraction. February 2007.

16.3. Federal Register, 40 CFR Part 136 App. A (July 2005).

16.4. Dioxins SOW, Exhibits A-C (Rev 2.2) Summary of Requirements, Reporting and Deliverables Requirements, and Target Compound List with Contract Required Quantitation Limits

16.5. Dioxins SOW, Exhibit D (Rev 2.2) Analytical Method for Dioxins/Furans CLPs

17. METHOD MODIFICATIONS

17.1. Deviations from EPA 1613B

17.1.1. Tetradecane instead of nonane is used as the final solvent to increase the stability of extracts and standards. Tetradecane is less volatile than nonane. Loss of analyte as a result of solvent incompatibility is monitored through recovery checks and calibration acceptance criteria

17.1.2. Microwave Assisted Extraction (MAE) and Soxtherm extraction increase the efficiency of the extraction by decreasing the amount of sample required for analysis and reducing the time required for extraction of CDDS/CDDFs.

17.1.3. The silica gel clean up (IFB) does not incorporate basic silica gel.

17.1.4. Solvents and volumes used for some of the clean up procedures are adjusted to TestAmerica specifications.

17.1.5. Table 3 of this SOP differs from Table 10 of the reference method for some matrices due to historical experience with such matrices.

17.1.6. Water sample extractions use 100mL of DCM instead of the referenced 60mL.

18. ATTACHMENTS

18.1. Table 1 - Analyte List

18.2. Table 2 - Concentration of Stock and Spiking Solutions Containing CDDS/CDDFs and Labeled Compounds

18.3. Table 3 - Suggested Sample Quantities to be Extracted for Various Matrices

19. REVISON HISTORY

19.1. WS-IDP-0007, Revision 3.3, Effective 08/16/2017

19.1.1. Section 7.7, changed “isooctane (or toluene)” to “acetone”.

19.1.2. Section 11.1.2.1, removed “dissolved into approximately 2mL of acetone.”

19.1.3. Section 11.1.2.2, removed “into 2mL of acetone.”

19.1.4. Editorial changes.

19.2. WS-IDP-0007, Revision 3.2, Effective 03/06/2017

19.2.1. Added Section 11.1.2.3, “If the sample is a drinking water then all spiking volumes listed in 11.1.2.1 and 11.1.2.2 are reduced by ½.”

19.2.2. Added Section 11.18.1.1, “If the sample is a drinking water, spike 10µL of internal standard.”

19.2.3. Section 11.18.4, added “Standard volume for drinking water is 10 µL.”

19.3. WS-IDP-0007, Revision 3.1, Effective 02/07/2016

19.3.1. Section 6.33, first sentence revised to read, “Hach 2100Q Portable Turbidimeter, or equivalent.”

19.3.2. Sections 7.2.7, 7.2.8, and 7.2.9 combined, and revised to read, “Turbidimeter calibration and verification standards: 800 NTU, 0.1 NUT, ICV (5 NTU) and ICB (0 NTU), suitable for use with the Hach Turbidimeter (Section 6.33).”

19.3.3. First note following Section 11.2.4.9 moved to immediately following Section 11.2.4. Second note removed.

19.3.4. Section 11.2.4.1, revised to read, "Press **<VERIFY CAL>**. Initial value for calibration is set at 800 NTU with an acceptance criteria of 10%."

19.3.5. Section 11.2.4.2, revised to read, "Place 800 NTU blank into machine and press **<READ>**. "

19.3.6. Section 11.2.4.3, revised to read, After reading, press **<DONE>**.

19.3.7. Section 11.2.4.4, revised to read, "Press **<CANCEL>**. This will take you back to the initial screen."

19.3.8. Section 11.2.4.5, revised to read, "Since the machine does not allow you to set the next calibration value to 0.1 NTU, you can place the 0.1 NTU blank into the machine and press **<READ>**. Reading must be between 90%-110."

19.3.9. Section 11.2.4.6, revised to read, "Press **<DONE>**. Place ICV into machine and take reading. Reading must be less than 5.5 NTU."

19.3.10. Section 11.2.4.7, revised to read, "Take ICB reading after. Reading must be less than 0.5 NTU."

19.3.11. Section 11.2.4.8, removed.

19.3.12. Section 11.2.4.9, renumbered as 11.2.5, and revised to read, "Mix the samples thoroughly to disperse solids. Wait for air bubbles to disappear. Pour samples into clean turbidimeter vials and record the readings."

19.3.13. Section 11.2.4.10, renumbered as 11.2.6, and revised to read, "Following every 10 samples, analyze a 5.0 NTU ICV. Reading must be less than 5.5 NTU."

19.3.14. Section 11.2.4.11, renumbered as 11.2.7.

19.3.15. Added Section 11.1.1.1, "For leachate samples (including an aliquot of the leachate blank), measure 200 mL of leachate into a clean 1 L sample bottle. Add approximately 800 mL of reagent water to the leachate. The sample volume in TALS for leachates is 200 mL."

19.3.16. Editorial Changes.

19.4. WS-IDP-0007, Revision 3.0 Effective 02/26/2016

19.4.1. All references to SPE apparatus and equipment for aqueous samples were removed.

19.4.2. Section 8 for holding times had clarification added and storage conditions

updated.

19.4.3. Editorial changes.

19.5. WS-IDP-0007, Revision 2.0 Effective 05/29/2015

19.5.1. Added Section 5.1.13 – "Microwave ovens used for extraction or digestion of samples can create elevated pressure in the extraction/digestion containers. Microwaves used for these processes must be equipped with an automated vapor sensor and shutoff system that automatically shuts down the digestion/extraction process when vapor pressure reaches pre-specific levels. Users must follow procedures in the microwave operator's manual to ensure that the vapor sensors are functional and working properly prior to starting each extraction/digestion batch."

19.5.2. Added reagents specific for MAE extraction.

19.5.3. Inserted Section 11.9, Microwave Extraction.

19.5.4. Editorial changes.

19.6. WS-IDP-0007, Revision 1.9, Effective 04/11/2014

19.6.1. Inserted Section 11.16.14 - Collect the sample and proceed to concentration step. 100 µl of tetradecane can be used as keeper if proceeding to IFB cleanup (Section 11.15).

19.6.2. Editorial changes.

19.7. WS-IDP-0007, Revision 1.8, Effective 03/07/2014

19.7.1. Inserted Section 11.2 –Turbidity

19.7.2. Editorial changes.

19.8. WS-IDP-0007, Revision 1.7, Effective 8/26/2013

19.8.1. Updated naming convention for IDA, IS, and native spike.

19.8.2. Modified Section 11.2.5 to reflect current laboratory practice regarding sample size, extraction volume, and spiking concentrations and amounts..

19.8.3. Editorial changes.

19.9. WS-IDP-0007, Revision 1.6, Effective 03/20/2012

19.9.1. Appended to Section 2.1: “This method can also use solid phase extraction (SPE) however, TestAmerica Sacramento is in the developmental stages for this extraction type and is not currently certified for its use.

19.9.2. Editorial changes.

19.10. WS-IDP-0007, Revision 1.5, Effective 06/10/2011

19.10.1. Modified Section 6.17: Whatman GF-D, GF-F, GMF150, or equivalent.

19.10.2. Inserted Section 6.18: Solid phase extraction discs, 3M 90mm C18, or equivalent.

19.10.3. Inserted Section 6.23: Vacuum extraction device for solid phase extraction, 1 Liter glass funnel with 90mm filter disc holder with a vacuum source, Kontes or equivalent.

19.10.4. Inserted Section 11.3: Aqueous Samples (solid phase extraction).

19.10.5. Inserted new Section 5.1.1 dealing with microwave oven safety.

19.10.6. Added new safety warning at Section 11.8

19.10.7. Editorial revisions.

Table 1
Analyte List

CDD/CDF
2,3,7,8-TCDF
2,3,7,8-TCDD
1,2,3,7,8-PeCDF
2,3,4,7,8-PeCDF
1,2,3,7,8-PeCDD
1,2,3,4,7,8-HxCDF
1,2,3,6,7,8-HxCDF
1,2,3,7,8,9-HxCDF
2,3,4,6,7,8-HxCDF
1,2,3,4,7,8-HxCDD
1,2,3,6,7,8-HxCDD
1,2,3,7,8,9-HxCDD
1,2,3,4,6,7,8-HpCDF
1,2,3,4,7,8,9-HpCDF
1,2,3,4,6,7,8-HpCDD
OCDF
OCDD

Table 2
Concentration of Stock and Spiking Solutions Containing CDDS/CDDFs and Labeled Compounds

Analyte	Type	Labeled Compound Stock Solution (ng/mL=pg/uL)	Labeled Compound Spiking Solution[a] (ng/mL=pg/uL)	PAR Stock Solution (ng/mL=pg/uL)	PAR Spiking Solution[b] (ng/mL=pg/uL)
Isotope Dilution Analyte (IDA)					
$^{13}C_{12}$-2,3,7,8-TCDD	IDA	100	2	--	--
$^{13}C_{12}$-1,2,3,7,8-PeCDD	IDA	100	2	--	--
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	IDA	100	2	--	--
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	IDA	100	2	--	--
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	IDA	100	2	--	--
$^{13}C_{12}$-OCDD	IDA	100	4	--	--
$^{13}C_{12}$-2,3,7,8-TCDF	IDA	100	2	--	--
$^{13}C_{12}$-1,2,3,7,8-PeCDF	IDA	100	2	--	--
$^{13}C_{12}$-2,3,4,7,8-PeCDF	IDA	100	2	--	--
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	IDA	100	2	--	--
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	IDA	100	2	--	--
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	IDA	100	2	--	--
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	IDA	100	2	--	--
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	IDA	100	2	--	--
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	IDA	100	2	--	--
Internal Standard (IS)					
$^{13}C_{12}$-1,2,3,4-TCDD	IS	200	100[d]	--	--
$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	IS	200	100[d]	--	--
Surrogate (SU)					
$^{37}Cl_4$-2,3,7,8-TCDD	SU	200	0.8[c]	--	--
Target Analyte (TA)					
2,3,7,8-TCDD	TA	--	--	40	4
1,2,3,7,8-PeCDD	TA	--	--	200	20
1,2,3,4,7,8-HxCDD	TA	--	--	200	20
1,2,3,6,7,8-HxCDD	TA	--	--	200	20
1,2,3,7,8,9-HxCDD	TA	--	--	200	20
1,2,3,4,6,7,8-HpCDD	TA	--	--	200	20
OCDD	TA	--	--	400	40
2,3,7,8-TCDF	TA	--	--	40	4
1,2,3,7,8-PeCDF	TA	--	--	200	20
2,3,4,7,8-PeCDF	TA	--	--	200	20
1,2,3,4,7,8-HxCDF	TA	--	--	200	20
1,2,3,6,7,8-HxCDF	TA	--	--	200	20
2,3,4,6,7,8-HxCDF	TA	--	--	200	20
1,2,3,7,8,9-HxCDF	TA	--	--	200	20
1,2,3,4,6,7,8-HpCDF	TA	--	--	200	20
1,2,3,4,7,8,9-HpCDF	TA	--	--	200	20
OCDF	TA	--	--	400	40

(a) typical spike amount is 1.0 mL
(b) typical spike amount is 50 uL
(c) typical spike amount is 1.0 mL
(d) typical spike amount is 20 uL

Table 3
Suggested Sample Quantities to be Extracted for Various Matrices [1]

Sample Matrix [1]		Example	Percent Solids	Phase	Quantity
Single-Phase					
	Aqueous	Drinking water	<1% Solid	[3]	1000mL
		Ground water	<1% Solid	[2]	1000mL
		Treated wastewater	<1% Solid	[2]	1000mL
	Solid	Dry Soil	>20% Solid	Solid	10g
		Compost	>20% Solid	Solid	10g
		Ash	>20% Solid	Solid	10g
	Organic	Waste solvent	<1% Solid	Organic	0.1g
		Waste oil	<1% Solid	Organic	0.1g
		Organic polymer	<1% Solid	Organic	0.1g
	Tissue	Fish & Wildlife		Organic	10g
		Food stuff		Organic	10g
		Human adipose		Organic	10g
Multi-Phase					
Liquid/Solid	Aqueous/Solid	Wet soil	1% Solid<x<30% Solid	Solid	10g
		Untreated effluent	1% Solid<x<30% Solid	Solid	10g
		Digested municipal sludge	1% Solid<x<30% Solid	Solid	5g
		Paper pulp	Dry and homogenize	Solid	10g
		Paper pulp-Sludge	Dry and homogenize	Solid	5g
	Organic/Solid	Industrial sludge	All	All	5g
		Oily waste	All	All	1g
Liquid/Liquid	Aqueous/Organic	In-process effluent	<1% Solid	Organic	1g
		Untreated effluent	<1% Solid	Organic	1g
		Drum waste	<1% Solid	Organic	1g
	Aqueous/Organic /Solid	Untreated effluent	>1% Solid	Organic/Solid	1g
		Drum waste	>1% Solid	Organic/Solid	1g

(1) The sample matrix may be amorphous for some samples. In general, when the CDDs/CDFs are in contact with a multiphase system in which one of the phases is water, they will be preferentially dispersed in or adsorbed on the alternate phase because of their low solubility in water.
(2) Aqueous samples are filtered after spiking with the labeled compounds. The filtrate and the solid phase trapped on the filter are extracted separately, and the extracts are combined for cleanup and analysis.
(3) Drinking water is filtered only if there are particulates and or have color.

Title: Determination of Nitroaromatics, Nitramines, and Specialty Explosives Based on Method 8330, SW-846 [Method 8330, 8330A, and 8330B]

Approvals (Signature/Date):

Koroush Vaziri Department Manager 7/5/2016 Date	Joe Schairer Health & Safety Manager / Coordinator 7/6/16 Date
Lisa Stafford Quality Assurance Manager 7/6/2016 Date	Crystal Pollock Laboratory Director 7.11.16 Date

1. SCOPE AND APPLICATION

1.1. This SOP describes the determination of nitroaromatic, nitramine, and specialty explosives or explosive residues based on Methods 8330, 8330A, 8330B. This method is applicable for compounds listed in Table 1 in aqueous, soil, solid, tissue, air, and wipe matrices. Analysis is performed utilizing an HPLC, coupled with an ultra-violet (UV) detection system. Confirmation is performed using a second, dissimilar column.

1.2. This method is NOT applicable for high concentrations of explosives in soil or waste samples.

1.3. The calibration range for the 8330 compounds is from 5.0 ng/mL to 1000 ng/mL on the instrument.

1.4. When undertaking projects for Department of Defense (DoD) and/or Department of Energy (DOE) the relevant criteria in QA Policy WS-PQA-021, "Federal Program Requirements" must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. Aqueous samples are prepared in one of three manners: Direct Aqueous Injection (DAI), low-level SPE, or high-level SPE per Method 8330A or 8330B.

2.1.1. Aqueous samples for the high level method can be performed by Direct Aqueous Injection (DAI), in which case the samples must be analyzed within 7 days of sampling. This method may be used as a screening analysis prior to utilizing SPE in order to prevent contaminating the SPE workstation.

2.1.2. In the low level SPE method, aqueous samples for the SPE method are extracted by passing water samples through Porapak™ RDX cartridges, where the components are absorbed. Analytes are eluted from the cartridges and volume adjusted.

2.1.2.1. The high level SPE method is similar to the low level method where the volume of the samples and reagents are scaled down by a factor of 10 with higher fortification levels.

2.2. Soil and sediment samples that are not suspected of high analyte levels are air dried, ground to a finer texture, and sieved. Samples for 8330 and 8330A are sieved through a number 30 mesh, and samples for 8330B are sieved through a number 10 mesh.

2.2.1. A sub-sample is extracted with 0.1% acetic acid in acetonitrile in a cooled ultrasonic water bath for 18 hours. An aliquot of the extract is diluted and filtered.

2.2.1.1. Tissue samples are not air-dried, ground, and sieved. Tissue samples are dried by mixing with anhydrous sodium sulfate prior to extraction with 0.1% acetic acid in acetonitrile.

2.3. Wipe samples are air dried and extracted with 0.1% acetic acid in acetonitrile in a cooled ultrasonic water bath for 18 hours. An aliquot of the extract is diluted and filtered.

2.4. Samples collected from stationary emission sources (Modified Method 5 sampling train for explosive materials) are prepared according to a separate procedure as outlined in the appropriate laboratory work instruction (WS-WI-0015).

2.5. Analysis is conducted using reverse phase High Performance Liquid Chromatography with ultraviolet (HPLC/UV) detection.

2.6. Confirmation is performed using a dissimilar column (typically cyano phase), HPLC/APCI/MS, or a second wavelength (Picric Acid only).

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Laboratory Quality Assurance Manual (QAM).

3.2. Data Qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. Coelution of target analytes can occur, resulting in false positives or biased high results. See the appendices for interferences specific to individual tests and suggested corrective actions.

4.2. The solvents, reagents, glassware, and other sample process hardware used in the extraction and analysis of nitroaromatics, nitramines, and specialty explosives must be free of interferences. All of these materials must be routinely demonstrated to be free from interference under the conditions of the analysis by running method blanks.

4.3. Glassware must be cleaned, dried, and solvent rinsed to maximize cleanliness and minimize interference.

4.4. HPLC and pesticide grade reagents must be used to minimize interference problems.

4.5. With the addition of acetic acid in the extracting solvent and in standard solutions, TETRYL has been stable for months without affecting the other nitroaromatics and

nitramines. If requested, acetic acid in the extraction solvent and in the standards may be omitted. The control limits for the 8330 analytes must be re-established in this case.

4.6. Nitroglycerin and PETN may be analyzed on the HPLC with a UV detector at 200 nm. The HPLC is best run under isocratic conditions for a stable baseline.

4.7. 3,5-Dinitroaniline may coelute with TNT on a C8 column or with Tetryl and nitrobenzene on a C18 column. If resolution is not sufficient on the primary column, 3,5-DNA must be identified and quantified on a Cyano column.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) 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-toed, nonabsorbent shoes are a minimum.

5.1. Specific safety concerns or requirements

5.1.1. The compound, 2,4,6-TNT is the analyte most often detected in high concentrations in soil samples. Soil samples as high as 2% 2,4,6-TNT can be safely ground. Samples containing higher concentrations should not be ground. The project manager or client must provide information as to whether the samples are suspected to contain explosives at a level greater than 2%. Visual observation of soil samples taken from a site expected to contain explosives is also important. Lumps of material that have a chemical appearance should be suspect and not ground. Explosives are generally a very finely ground grayish-white material.

5.1.2. The sources of many samples analyzed for explosives may also be contaminated with phosphorus. Exercise caution when allowing soil samples, filter papers, filter discs and similar materials to dry. As phosphorus dries in air, it may spontaneously ignite or begin to smoke. Should this happen, immerse the filter or material in clean water, activate the Emergency Response Team and contact the EH&S Coordinator or Hazardous Waste Specialist for additional guidance.

5.1.3. Hearing protection is recommended when ultrasonic digestion is carried out.

5.1.4. Hearing protection is required for everyone in the laboratory any time the ball mill, puck mill, or benchtop grinder are grinding.

5.1.5. Explosive standards used in this method, if purchased as neat, are by their nature unstable and must be stored according to the manufacturer's directions, preferably wet. If standards are purchased as solutions at low concentrations in organic solvents, safety hazards are minimized.

5.1.6. Exercise caution when using syringes with attached filter disc assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

5.1.7. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Nitrile gloves provide satisfactory protection against acetone and acetonitrile. Latex gloves provide suitable protection against methanol. Any of these gloves are suitable for acetic acid.

5.1.8. Exposure to chemicals must be maintained as low as reasonably achievable; therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.9. Laboratory procedures such as repetitive use of pipettes, repetitive transferring of extracts and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Materials (1)	Hazards	Exposure Limits(2)	Signs and symptoms of exposure
Acetic Acid	Corrosive Poison Flammable	10 ppm-TWA	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.

Materials (1)	Hazards	Exposure Limits(2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Acetonitrile	Flammable Poison	40 ppm-TWA	Early symptoms may include nose and throat irritation, flushing of the face, and chest tightness. Prolonged exposure to high levels of vapors may cause formation of cyanide anions in the body.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Phosphoric Acid (Orthophosphoric Acid)	Corrosive	1 mg/m^3 TWA	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Recommended preventive and routine maintenance is described in below.

On an as-needed basis, perform the following: Replace columns when peak shape and resolution indicate that chromatographic performance of column is below method requirements. Rinse flow cell with 1N nitric acid if dirty flow cell. Change pump seals when flow becomes inconsistent. Backflush column if applicable. Change in-line filters for solvents.
Daily, when the instrument is in use, perform the following: Check level of solution in reservoirs. If adding, verify that solvent is from the same source. If changing, rinse delivery lines to prevent contamination of the new solvent. Check gas supply if applicable. Flush with an appropriate solvent to remove all bubbles. Pre-filter all samples.
Every 6-9 months, perform the following: Change pump seals.

6.2. Balances - Top loading, capable of accurately weighing to the nearest 0.01 gram. Analytical, capable of accurately weighing to the nearest 0.0001 gram.

6.3. Centrifuge.

6.4. Filter assembly, 25-mm, 0.45 um pore size PTFE filters, such as Millipore Millex-LCR filters. Other manufacturer's filters may be substituted.

6.5. Liquid Chromatograph (HPLC) – Agilent 1100 Series or equivalent, equipped with:

- Autosampler
- Temperature Controlled Column Compartment
- Solvent Degasser
- Multiwavelength UV Detector
- Quaternary Pump
- Data system – Agilent Chemstation B.01.03 or higher version, or equivalent
- Data Processing Software: Target Revision 4.12 and Chrom version 2.x or equivalent

6.6. HPLC Columns – Columns specified below may be replaced with equivalent columns from alternate vendors, provided they perform in a similar manner.

- C18 Column: Phenomenex Synergi Hydro-RP, 250 x 4.6 mm, 5um
- CN Column: Restek Zorbax Cyano, 250 x 4.6 mm, 5um
- Short C-18: Agilent Extend C-18 100 x 3.0 mm, 3.5um
- PFP: Phenomenex PFP, 150 x 4.6mm, 2um

6.7. Mesh sieve and pan, brass or stainless steel, mesh size # 30 and #10.

6.8. Mortar and pestle, ceramic.

6.8.1. Blender may be used, glass or stainless steel.

6.8.2. Grinder, Retch model RM 100, with ceramic bowl and pestle, or equivalent.

6.9. Pulverizing Mill (ring and puck), ESSA model LM2-P or equivalent - for the grinding of soils per method 8330B

6.9.1. The grinding bowl and puck are cleaned after each use by washing with soap and water, rinsing with hot tap water, rinsing with DI water, and then rinsing with acetonitrile. A final wipe down of the bowl and puck while still wet with acetonitrile is done with a Kimwipe (TNT in particular is reported to be prone to adhering to steel surface). In addition, sand blanks are used to monitor potential carry-over for each batch of samples (see Section 9.11 for details).

6.10. Recirculating cooler.

6.11. Spatula, stainless steel, or equivalent.

6.12. Syringe, 10mL disposable.

6.13. Temperature controlled ultrasonic water bath.

6.14. Test tubes, 8-mL and 16-mL, glass with teflon-lined screw cap.

6.15. Vials, 40-mL, glass with teflon-lined screw cap.

6.16. Workstation, Zymark SPE Auto-Trace, an automated workstation to perform solid phase extraction on aqueous samples.

6.17. Reciprocating shaker.

7. REAGENTS AND STANDARDS

All reagents must be ACS reagent grade or better unless otherwise specified.

7.1. The preparation of reagents and solutions is recorded in the Reagent Prep Module of TALS, or in a reagent preparation notebook.

7.2. Acetic acid, glacial, reagent grade.

7.3. Acetic acid in acetonitrile, 0.1% v/v.

7.4. Phosphoric acid, H_3PO_4, reagent grade

7.5. Acetone, pesticide quality.

7.6. Acetonitrile, HPLC grade.

7.7. Sodium chloride, NaCl

7.8. Ammonium Formate, reagent grade; 5 mM in water.

7.9. Calcium chloride, $CaCl_2$, aqueous solution; 1.3 grams/liter in distilled water.

7.10. Methanol, HPLC grade.

7.11. Water, de-ionized and HPLC grade.

7.12. Standards may be prepared from neat or purchased as certified solutions. All standard preparations must be entered in the standards prep log. Whenever possible, standard materials are obtained from ISO 17025 accredited firms and are manufactured consistent with Guide 34 ISO 17025.

7.13. Standards must be reviewed for expiration dates prior to each use. All expired standards must be rotated out of the laboratory to the HazWaste storage area for disposal per the Corporate Safety Manual.

7.14. 8330 Mix: A prepared mixture of the 8330 components (Table 2) at 1.0 mg/mL in acetonitrile/methanol (1:1) is purchased for the preparation of fortification and analytical solutions. They are obtained from EM Science, Accustandard, Ultra Scientific, Radian International, Cerilliant, Restek, or other reputable source. The concentration of the stock mix solution is 1.0 mg/mL. The expiration date is either the manufacturer's expiration date, or one year from date of opening, whichever is earlier, provided it is properly stored. Once the ampoule is broken, the unused portion is transferred to an amber vial, sealed with teflon-lined screw cap, and refrigerated at 2-6°C.

7.14.1. Prepare primary mix solutions of the 8330 mix at 50 µg/mL and 5.0 µg/mL, each in 0.1% acetic acid in acetonitrile from the stock mix. Refrigerate at 2-6°C. This standard is valid for 6 months, or until the stock expires, whichever is earlier. These solutions are used for fortification of samples, and the preparation of the analytical standards.

7.15. 3,5-Dinitroaniline (3,5-DNA) at 1.0 mg/mL in acetonitrile is prepared from neat (Aldrich or ChemService) or purchased as solution (Accustandard). Store in the refrigerator at 2-6°C and replace after 1 year.

7.15.1. Prepare a solution of 3,5-DNA at 50 µg/mL in acetonitrile from the 1.0 mg/mL stock. Store in the refrigerator and replace after six months. The solution is also used in the fortification of samples.

7.15.2. 3,5-DNA may be added with the 8330 spike mix at 50 ug/mL.

7.16. Nitroglycerin, PETN, and EGDN are received as solutions at 0.10 mg/mL or at 1.0 mg/mL in ethanol, methanol, or acetonitrile (Radian International, EM Science, Accustandard, Ultra Scientific).

7.16.1. Prepare a solution of nitroglycerin and PETN (NG/PETN) at 50 µg/mL each in acetonitrile. Refrigerate at 2-6°C, and replace after 6 months. This solution is also used in the fortification of samples. EGDN may be added to the NG/PETN mix at the same concentration, or a separate standard may be prepared.

7.17. Picric Acid is received as a solution at 0.10 mg/mL or at 1.0 mg/mL in ethanol, methanol, or acetonitrile (Radian International, EM Science, Accustandard, Ultra Scientific).

7.17.1. Prepare a solution of picric acid at 50 µg/mL in acetonitrile. Refrigerate at 2-

6°C, and replace after 6 months. This solution is also used in the fortification of samples.

7.18. 3,4-Dinitrotoluene, is used as a surrogate compound.

NOTE: 3,4-DNT co-elutes with 2,4-DNT on the C8 analytical column, but resolves on the cyano column. A C18 analytical column with high carbon loading such as Carbosorb is needed to resolve 3,4-DNT from the 8330 analytes.

7.18.1. Prepare stock solution from neat (Aldrich or other reputable source) at approximately 1.0 mg/mL in acetonitrile. Refrigerate at 2-6°C, and replace after 1 year.

7.18.2. Prepare surrogate solutions from the stock solution at 50 µg/mL in acetonitrile. Refrigerate at 2-6°C, and replace after 6 months.

7.19. Dilute the stock 8330 (5.0 and 50ppm), surrogate, picric acid, and NG/PETN solutions in 25:75 0.1% Acetic acid in acetonitrile: HPLC grade water to make the calibration standards listed in Table 3.

7.19.1. Store the solutions in the refrigerator. These solutions expire after 6 months, or when the stock solution expires, whichever is earlier. Tetryl is stable when properly stored with acetic acid.

7.19.2. If there is chromatographic coelution of 3,5-DNA with other 8330 analytes such as Tetryl on the columns used for primary and confirmation analysis, then 3,5-DNA prepared as a separate calibration and spiking mix. Otherwise, it is included in the 8330 mix.

7.20. Occasionally, compounds such as metabolites of explosive materials may be added to the analysis. Stock solutions and spiking solutions are prepared as above. Refer to Tables 1 through 4 for information regarding compounds infrequently analyzed.

7.21. Certified Reference Material (CRM) (AKA Grinding LCS)

This is solid matrix spiked with known concentrations of each explosive, for use when grinding and incremental subsampling is performed. It may be purchased from either Environmental Resource Associates (USACE Explosives in Soil Standard) or Phenova (Custom Explosives in Soil by 8330B). Because of the nature of the process, it is not recommended to attempt in-house preparation of this sample.

7.22. Reference (Second Source) Standards.

7.22.1. Prepare reference standards to verify the quality of the fortification solutions and analytical standards.

7.22.2. A reference standard is any standard solution made from a source other than

the stock standard. Reference standards may be from the EPA, the manufacturer, or from another reliable source.

7.22.3. If a secondary source is not available, a separate intermediate stock solution will be made from the same neat by another chemist or from a neat from a different vendor and different lot number.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Aqueous samples are collected in glass bottles, sealed with teflon-lined screw caps and iced or refrigerated at 4 ± 2ºC and protected from sunlight from time of collection until extraction.

8.2. Soil samples are collected in glass jars with teflon-lined screw caps and iced or refrigerated at 4 ± 2°C and covered from sunlight from time of collection until extraction.

8.3. Samples collected from stationary emission sources consist of solid, solvent, and aqueous matrices. Sample collection and preservation requirement are described in the appropriate laboratory work instruction

8.4. Soil and sediment samples should be air dried until there is no visible appearance of moisture at room temperature or cooler after collection. Successful grinding and sifting verifies dryness. While it is possible to analyze wet soil samples, it is much more difficult to obtain a homogeneous sample on a wet sample. If wet soil samples are to be analyzed, a moisture determination must be made on a separate portion (refer to SOP WS-OP-0013, “Determination of Percent Moisture”).

8.5. The extraction hold time for aqueous samples is 7 days from the time of sampling to extraction and the analytical hold time is 40 days from extraction to analysis. The holding times for samples collected from stationary emission sources are described in the appropriate laboratory work instruction.

8.5.1. Aqueous samples for the high level method by DAI must be aliquotted, filtered, analyzed and confirmed within 7 days of sampling. If aqueous samples are to be extracted by solid-phase extraction procedure, they must be extracted within 7 days and analyzed within 40 days from extraction.

8.6. Soil samples are to be air dried, finely ground, sifted, and extracted within 14 days of sampling and analyzed within 40 days of extraction.

8.6.1. Tissue samples may have an extended extraction holding of up to 1 year if stored frozen. Samples should be analyzed within 40 days of extraction.

9. QUALITY CONTROL

9.1. Specific Quality Control requirements are associated with DoD/DOE projects. These specific requirements can be found in WS-PQA-021 "Federal Program Requirements".

9.2. Initial Demonstration of Capability.

The initial demonstration and method detection limit (MDL) studies described in Section 13 must be acceptable before analysis of samples may begin. MDLs are filed in the Quality Assurance Department.

9.3. Batches are defined at the sample preparation state. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the QC program document (WS-PQA-0003) for further details of the batch definition.

9.3.1. The quality control batch is a set up of up to 20 samples of the same matrix processed using the same procedure and reagents within the same time period. The quality control batch must contain a matrix spike/spike duplicate (MS/MSD), a Laboratory Control Sample (LCS) and a method blank. Laboratory generated QC samples (Blank, LCS, MS/MSD) do not count toward the maximum 20 samples in a batch. Field QC samples are included in the batch count. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. If insufficient sample is available for an MS/MSD a LCSD must be substituted. In the event that multiple MS/MSD are run with a batch due to client requirements, the additional MS/MSD do not count toward the maximum 20 samples in a batch.

9.4. In-house control limits must be determined for surrogates, matrix spikes, and laboratory control samples. These limits must be determined at least annually. The recovery limits are mean recovery ± 3 standard deviations, unless that limit is tighter than the calibration criteria, in which case limits may be widened. Refer to policy WS-PQA-0003 for details.

9.5. Surrogate recoveries in samples and QC samples must be assessed to ensure that recoveries are within established limits. If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze the extract if either of the above checks reveals a problem.

- Reprepare and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem. Repreparation is not necessary if there is obvious chromatographic interference.
- The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to reprepare/reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

9.5.1. If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and repreparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then repreparation or flagging of the data is required.

9.5.2. Refer to WS-PQA-003 for further details of the corrective actions.

9.6. One method blank must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. For aqueous samples, the method blank is an aliquot of laboratory reagent water. For soil samples, the method blank is an aliquot of control soil (such as Ottawa sand). For tissue samples, the method blank is an aliquot of control soil (such as Ottawa sand) mixed with anhydrous sodium sulfate.

9.7. The method blank is processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when target analytes are detected in the method blank above the reporting limit or when surrogate recoveries are outside of the control limits. Re-extraction of the blank, other batch QC, and the affected samples are required when the method blank is deemed unacceptable. See WS-PQA-0003 for specific acceptance criteria.

9.7.1. The method blank must not contain any analyte at or above the reporting limit or at or above 10% of the measured concentration of that analyte in the associated samples, whichever is higher.

9.7.2. If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.

9.7.3. Re-extraction and reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

9.7.4. Projects performed under the auspices of the DOD/DOE QSM must meet QSM specific criteria for method blanks. Results are acceptable if the blank contamination is less than ½ of the reporting limit for each analyte, or less

than 1/10 of the regulatory limit, or less than 1/10 of the sample result for the same analyte, whichever is greater. If the method blank does not meet the acceptance criteria, the source of contamination must be investigated and measures taken to correct, minimize or eliminate the problem. If contamination remains, the contaminated samples should be re-prepared and reanalyzed with a new MB and batch-specific QC samples.

9.7.5. Refer to WS-PQA-0003 for further details of the corrective actions.

9.8. A Laboratory Control Sample (LCS) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The LCS is an aliquot of laboratory matrix (e.g. water, Ottawa sand, sodium sulfate, etc.) spiked with analytes of known the identity and concentration. The LCS must be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside of the control limits provided by the LIMS or by the client. Re-extraction of the blank, other batch QC and all associated samples are required if the LCS is deemed unacceptable. See WS-PQA-0003 for specific acceptance criteria.

9.9. A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. An MS/MSD pair is aliquots of a selected field sample spiked with analytes of known identity and concentration. The MS/MSD pair must be processed in the same manner and at the same time as the associated samples. Spiked analytes with recoveries or precision outside of the control limits must be within the control limits in the LCS. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte are outside of the control limits provided by the LIMS or by the client. Re-extraction of the blank, the LCS, the selected field samples, and the MS/MSD may be required after evaluation and review. MS/SD samples are not typically prepared for samples collected from stationary emission sources because the entire sample is consumed in the initial extraction procedure.

9.10. A duplicate control sample (LCSD) must be substituted when insufficient sample volume is provided to process an MS/MSD pair and batch precision is required by client or program. Otherwise, precision may be evaluated between sequential LCS batches. The LCSD is evaluated in the same manner as the LCS. See WS-PQA-0003 for specific acceptance criteria.

9.11. Method 8330B Grinding LCS

When samples for Method 8330B must be ground and subsampled, an additional LCS is required. This sample is a pre-made reference sample from an outside supplier, such as the USACE Explosives in Soil Standard from Environmental Resource Associates, or the Custom Explosives in Soil by 8330B from Phenova.

9.11.1. To process the sample, 100-200g Certified Reference Material (CRM) must be ground using the Ring and Puck Mill in the same manner as the grinding blanks referenced in section 9.11 and samples referenced in section 11.5.7. After grinding, the sample is sub sampled by incremental subsampling techniques and a 10g sample is created. The remaining sample may be stored in the freezer and re-used until empty or until the manufacturer's expiration date has been reached (whichever comes first). It is recommended the CRM may be good for up to 30 days, but the manufacturer may extend the expiration date to 90 days.

9.12. Method 8330B Grinding/Subsampling Triplicate

For method 8330B, after grinding, three 10 gram multi-incremental sub samples must be taken for one of the samples in the batch. All three sub samples are to be extracted and analyzed with the analytical batch. Control limits for target analytes detected above the reporting limit in the triplicate samples will be established as $\leq$ 20% RSD. These may be updated after sufficient data has been collected. Refer to corrective action in WS-PQA-003 (duplicates) if criteria are not met.

9.13. Grinding Blanks

Before each sample is processed through the ring and puck mill, the ring and puck is cleaned per section 6.8. Then approximately 200g of Ottawa Sand is ground. This ground sand is saved and labeled with the sample ID of the next sample ground with the suffix "blank". After a batch of samples has been processed through the ring and puck, generate a composite using sub-aliquots from all blanks ground before the samples. These aliquots are equal portions from each blank, combined to create a 10 g composite sample. This composite is extracted and analyzed in the same manner as the field samples.

9.13.1. Acceptance Criteria:

The grinding blank must not contain any analyte of interest at or above the RL or one-half of the RL for DoD projects or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

9.13.2. Corrective Action:

If the composite grinding blank results are greater than the acceptance limits, then the individual grinding blanks will be extracted and analyzed to determine when the contamination occurred and exactly which samples were affected. Samples associated with a contaminated grinding blank producing positive results for the same contaminant must be reprocessed and reanalyzed. If unground sample is not available, then the potential carry-over between samples must be described in a non-conformance memo and discussed in the final report case narrative.

10. CALIBRATION

For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to Policy CA-Q-P-003, "Calibration Curves and Selection of Calibration Points".

10.1. External calibration is used as described below. Prepare standards containing each analyte of interest at a minimum of five concentration levels. The low level standard should be at or below the reporting limit. The other standards define the working range of the detector. The calibration criteria apply to both the primary and confirmation columns.

10.2. Retention Time Windows

10.2.1. Retention time windows must be determined for all analytes. Make an injection of all analytes of interest each day over a three-day period. Calculate the standard deviation of the three retention times for each analyte. The retention time window is defined as plus or minus three times the standard deviation of the retention times of each analyte.

10.2.2. The center of the retention time window is the retention time from the last of the three standards. The centers of the windows are updated with the mid point of the initial calibration and each 12 hour calibration. The widths of the windows will remain the same until new windows are generated following the installation of a new column.

10.2.3. If the retention time window as calculated above is less than ± 0.15 minutes, use ± 0.15 minutes as the retention time window. This allows for slight variations in retention times caused by sample matrix.

10.2.4. The laboratory must calculate new retention time windows each time a new column is installed. The new windows must be generated within one week of the installation of the new column. Until these standards have been run on the new column, the retention time windows from the old column may be used, updated with the retention times from the new initial calibration.

10.2.5. Corrective Action for Retention Times

The retention times of all compounds in each continuing calibration must be within the retention time windows established by the 12 hour calibration. Retention time shifts due to matrix should be narrated.

10.3. Initial Calibration

10.3.1. A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes

include, but are not limited to: new columns or detector lamps. A new calibration is not required after minor maintenance.

10.3.2. With the exception of the circumstances delineated in policy CA-Q-P-003, it is not acceptable to remove points from a calibration curve. In any event, at least five points must be included in the calibration curve. Quadratic (second order) calibrations require at least six points.

10.3.3. In general, for environmental analysis, average response factors are the most appropriate calibration model. Linear or curved regression fits should only be used if the analyst has reason to believe that the average RF model does not fit the normal concentration/response behavior of the detector. For details of linear or curved regression fit equations, refer to policy CA-Q-P-003.

10.3.4. The following requirements must be met for any calibration to be used.

- Response must increase with increasing concentration.
- If a curve fit (linear or otherwise) is used, the intercept of the curve must be less than ± the reporting limit for the analyte.
- For the linear case, the correlation coefficient (r) must be greater than or equal to 0.995. For the quadratic case, the coefficient of determination (r2) may be used, and must be greater or equal to 0.990.

10.3.5. The same injection volume is used for both samples and standards.

10.3.6. All units used in the calculations must be consistently uniform, such as concentration in ng/mL and response in area or height counts.

10.3.7. A minimum of five analytical standards of different analyte concentrations (refer to Table 3 for composition of standards) is used to generate the curve.

10.3.8. Each standard is injected once to obtain the peak height for each analyte at each concentration, and the response factor (Rf) is calculated as follows

$$Rf = \frac{PeakHeight}{analyteconcentration}$$

Equation 1

10.3.9. Determine the average response factor ($\overline{Rf}$) and the percent relative standard deviation (% RSD) for each analyte:

$$\overline{Rf} = \frac{\sum Rf}{n}$$

Equation 2

Where n is the number of data points (n = 5 for five levels).

10.3.10. The initial calibration is valid for each analyte when the %RSD is less than

or equal to 20%. If the %RSD is ≤ 20%, linearity can be assumed and the average response factor can be used in place of a calibration curve.

10.3.10.1. If the % RSD is greater than 20%, then the cause must be determined to bring the system back in control before the samples are analyzed, and the system must be recalibrated.

10.3.10.2. If a linear or regression line is used, the coefficient of determination (r2) shall be greater than or equal to 0.990, or the correlation coefficient (r) shall be greater than or equal to 0.995, whichever is appropriate to the regression fit used. If the regression lines do not meet criteria, evaluate the calibration, then perform any required instrument maintenance prior to repeating the initial calibration process.

10.3.11. Minimally, if the daily CCV criterion (15%D for Methods 8330 and 8330A, 20%D for Method 8330B) is met, a five point curve is valid for 12 months. However, the typical life of the column varies and can last two to three months of continuous use. New columns require a new initial calibration.

10.4. Initial Calibration Verification (ICV): A second source standard must be analyzed with the initial calibration curve. Each compound of the second source calibration must meet ≤15%D for Methods 8330 and 8330A and ≤20%D for Method 8330B when calculated against the initial calibration.

10.4.1. Corrective actions for the ICV include:

- Rerun the ICV
- Remake or acquire a new ICV
- Evaluate the instrument conditions
- Evaluate the Initial Calibration Standards
- Re-calibrate as needed

Equation 3

$$\%D = \frac{|R_1 - R_2|}{R_1} \times 100$$

Where R1 is the expected result, and R2 is the calculated result based on the initial curve.
Refer to Table 3 for the concentration of the ICV.

10.5. Calibration Verification

10.5.1. At a minimum, the working calibration curve or RF must be verified by the analysis of a mid-level calibration standard at the beginning, following every 10 samples, and at the end of the analysis sequence.

10.5.2. The center of each retention time window is updated at the start of the analytical shift.

10.5.3. The initial calibration verification standard used at the start of the analysis sequence must be a different concentration than the mid-level calibration standard used for subsequent calibration verifications. Recommended concentrations are given in Table 3, with Level 5 suggested for the first calibration verification, and Level 4 suggested for subsequent calibration verifications.

10.5.4. Any individual compounds with %D ≤ 15% meet the calibration criteria. For Method 8330B, the criteria is %D ≤ 20%.

10.5.5. It is not necessary to run a daily continuing calibration verification standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration, i.e., the calibration curve and second source standard (initial calibration verification).

10.5.6. Samples must be bracketed by calibration verification standards that meet the criteria listed above.

10.5.7. If the analyst notes that a CCV has failed and can document the reason for failure (e.g. no purge, broken vial, carryover from the previous sample etc.) then a second CCV may be analyzed without any adjustments to the instrument. If this CCV meets criteria and the preceding samples show no evidence of similar failure, the preceding samples have been successfully bracketed and no further action is required. If this CCV fails or adjustments to the instrument are performed before the repeat CCV then the preceding samples have not been successfully bracketed but analysis may continue following a passing CCV.

10.5.8. It is not allowed to analyze repeat CCVs on unattended runs.

10.5.9. If highly contaminated samples are expected it is acceptable to analyze blanks or primers at any point in the run to eliminate carryover.

10.5.10. % Difference is used to compare the continuing calibration verification to the initial calibration when the average response factor is used.

Equation 4

$$\%Difference = \frac{\left|x - \bar{x}\right|}{x} \times 100 \quad \%$$

Where X = Calibration or response factor of the CCV
$\bar{X}$ = Average Calibration or response factor.

10.5.11. Corrective Actions for Continuing Calibration

If the continuing calibration fails the %D criteria for any analyte, corrective action must be taken. This may include replacing the guard column, replacing the in-line frit, or other minor instrument adjustments, followed by reanalyzing the standard. If the overall average %D still varies by more than ± 15%, there may be a problem with the standards, the instrument, or the column. If the column is replaced, a new initial calibration must be performed.

10.5.11.1. Any samples injected after the last good continuing calibration standard must be reinjected, unless one of the following conditions is satisfied:

10.5.11.2. If the CCV response is elevated and the preceding samples are non-detect for all analytes of concern, an NCM may be filed and the data reported.

10.5.11.3. If the samples have already been analyzed twice, and it is apparent that matrix effects are causing the out of control event, an NCM may be filed and the data reported. Client consultation or dilution of samples may be necessary.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Direct Aqueous Injection (DAI) (A HIGH LEVEL method):

NOTE: If Direct Aqueous Injection cannot be performed within the 7 day holding time, then the hold times can be extended by performing the SPE method within 7 days. Analysis must then be performed within 40 days of extraction.

11.2.1. For samples received as acetone (STEM analysis), dilute the sample 10x with HPLC water prior to proceeding as directed below.

11.2.2. Allow the aqueous samples to equilibrate to room temperature.

11.2.3. Pipet 10 mL aqueous sample into a 16-mL screw cap test tube. For control samples (MB and LCS), use HPLC grade water.

11.2.4. Add the surrogate to the samples, including the method blank, DCS, LCS, and MS/MSD. Add 20 uL of the 50 ug/mL 3,4-DNT surrogate to the 10 mL sample aliquots to yield 100 ppb.

11.2.5. Add the spiking solution to the LCS and MS/MSD.

LCS/LCSD/MS/MSD Spiking – DAI Method			
Requested Analyte	**Amount Added**	**Solution Concentration**	**Sample Concentration**
8330 + DNA	100 uL	5.0 ug/mL 8330	50 ug/L
NG/PETN	20 uL	50 ug/mL NG/PETN	100 ug/L
Additional analytes	20 uL	50 ug/mL spiking solution	100 ug/L

11.2.6. Mix the contents well, and filter the samples and QC: Transfer the aqueous sample into a 10-mL disposable syringe fitted with a 25-mm PTFE 0.45 µm filter on the syringe tip and press the plunger. Collect the sample in a 16-mL screw cap test tube.

WARNING: Exercise caution when using syringes with attached filter assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

11.2.7. If the extract is to be split for another test, then aliquot the required amount of the extract into an 8-mL test tube.

11.2.8. Extracts are ready for analysis. The final sample concentration is 10mL/10mL. Store the extracts in the refrigerator at 2-6°C.

11.3. Solid Phase Extraction (SPE) on Aqueous Samples using the Zymark Autotrace Workstation.
Below is an overview of the program to run the workstation. Whenever the method is modified, edit and update the method on a 3.5 inch disk.

Step 1: Process 6 samples using the following procedure:
Step 2: Condition column with 10 mL of acetonitrile into SOLVENT WASTE
Step 3: Condition column with 5 mL of acetonitrile into SOLVENT WASTE
Step 4: Condition column with 5 mL of water into AQUEOUS WASTE
Step 5: Condition column with 5 mL of HOAc/water 0.1% into AQUEOUS WASTE
Step 6: Load 100 or 1050 mL of sample onto column
Step 7: Rinse column with 5 mL of HOAc/Water 0.1% into AQUEOUS WASTE
Step 8: Dry Column with gas for 2.0 minutes
Step 9: Rinse column with 0.5 mL of HOAc/MeCN 0.1%

Step 10: Collect 5 mL fraction into sample tube using HOAc/MeCN 0.1%
Step 11: END

FLOW RATES	mL/min
Conditioning flow	15.0
Load flow	15.0
Rinse flow	5.0
Elute flow	2.0
Conditioning Air Push	15.0
Rinse Air Push	20.0
Elute Air Push	5.0
SPE PARAMETERS	
Push Delay	5 sec
Air Factor	1.0
Autowash Volume	1.00 mL
WORKSTATION PARAMETERS	
Maximum Elution Volume	12.0 mL
Exhaust Fan on	Y (Y=Yes N=No)
Beeper on	Y (Y=Yes N=No)
NAME SOLVENTS	
Solvent 1	water
Solvent 2	methanol
Solvent 3	acetonitrile
Solvent 4	HOAc/Water 0.1%
Solvent 5	HOAc/MeCN 0.1%

where HOAc/Water is acetic acid in water, 0.1% v/v, and HOAc/MeCN is acetic acid in acetonitrile, 0.1% v/v. gas = compressed air or nitrogen.

CAUTION: NEVER USE ACETONE ON THE ZYMARK AUTOTRACE SPE.

Per manufacturer's comment, acetone will dissolve the seals within the unit.

11.3.1. Allow the samples to equilibrate to room temperature.

11.3.2. Measure the amount of aqueous sample needed into a glass bottle or flask.

11.3.3. Use HPLC water for MB, LCS, and LCSD (if performed).

11.3.4. If leachate samples are being prepared, measure out one 200 mL aliquot of the blank leachate for the leachate blank (LMB).

Sample Volumes (Approximate) – SPE Preparation	
Method	**Volume Required**
High Level	100 mL water sample
Low Level	1000 mL water sample
TCLP	200 mL leachate, diluted to 1L with HPLC water

11.3.5. Add the surrogates to all samples, including the MB, LCS and MS/MSD.

Surrogate Spiking – SPE Preparation			
Method	**Amount Added**	**Solution Concentration**	**Sample Concentration**
High Level	200 uL	50 ug/mL 3.4-DNT	100 ug/L
Low Level	50 uL	50 ug/mL 3.4-DNT	2.5 ug/L
TCLP	50 uL	50 ug/mL 3.4-DNT	12.5 ug/L

11.3.6. If applicable, fortify the LCS, LCSD, and MS/MSD.

LCS/LCSD/MS/MSD Spiking – SPE Preparation				
Method	**Spike**	**Amount Added**	**Solution Concentration**	**Sample Concentration**
High Level	8330	100 uL	50 ug/mL	50 ug/L
	NG/PETN	200 uL	50 ug/mL	100 ug/L
	Additional Analytes	200 uL	50 ug/mL	100 ug/L
Low Level	8330	20 uL	50 ug/mL	1.0 ug/L
	NG/PETN	100 uL	50 ug/mL	5.0 ug/L
	TNX, DNX, MNX	80 uL	50 ug/mL	4.0 ug/L
	Picric Acid	100 uL	50 ug/mL	5.0 ug/L
	Additional Analytes	20 uL	50 ug/mL	1.0 ug/L
TCLP	8330	20 uL	50 ug/mL	5.0 ug/L
	NG/PETN	100 uL	50 ug/mL	25 ug/L
	Picric Acid	100 uL	50 ug/mL	25 ug/L

11.3.7. Weigh and record the initial mass of each sample bottle, including the QC's (with the screw caps on).

Note: If water samples are cloudy or turbid, and contain sediments or particulates, they should be pre-filtered before proceeding with the extraction: Add the surrogate, mix the sample well, and filter with vacuum. Proceed with the extraction using the filtered sample. If filtering is performed, a separate method blank should undergo the filtering procedure as well.

11.3.8. If the samples are to be analyzed for picric acid, add 50g NaCl to each sample and mix thoroughly. Fortify the sample .

11.3.8.1. If there is not enough room in the bottle for the NaCl addition, remove the excess water, cap the bottle, and reweigh (and record the mass) before proceeding with the NaCl addition, mixing, and fortification.

11.3.9. The samples are ready for extraction by the Zymark Autotrace SPE workstation.

11.3.10. The program for the Autotrace SPE workstation must be set up using the PC computer on a 3.5" disk. The overview of the program and the parameters are shown at the beginning of the section above, and the correct values must be entered for the appropriate method; the high level or the low level.

11.3.11. For the High Level method, edit the volume to load **100** mL on step 6.

11.3.12. For the Low Level method, edit the volume to load **1050** mL on step 6.

11.3.13. Before starting for the day, check the solvents and replace the solvents if necessary. If necessary, solvent lines should be purged by loading the solvent purging program.

11.3.14. The sample lines should have been in a plastic bag or in a glass jar, indicating the lines have been cleaned with methanol followed by de-ionized water from the Cleaning Sample Lines program.

11.3.15. Set the SPE columns in place and depress the plunger. Once in place the green light should be on. Use 6 mL, 500 mg Porapak™ RDX Cartridges, Waters Catalog number WAT047220, or equivalent.

11.3.16. Set the receiving vessels (test tubes) in place. Be sure the first sample is on your left and the sixth sample on your right.

11.3.17. Check the level and identity of the solvents - replace if necessary, and set the samples in its place.

11.3.18. Check the waste containers (Aqueous and Organic) and replace if necessary. Do NOT allow waste containers to overflow.

11.3.19. Load the program '8330-A' in the disk drive of the Zymark Autotrace, and press Load.

11.3.20. When the disk has been loaded, the Zymark Autotrace is ready to start. Follow instructions displayed on the small screen "Press CONT to start" if ready to start.

11.3.21. After the system is completed, the screen displays "program completed".

Move the extracts aside and seal them with teflon-lined screw caps. Remove the SPE columns and discard them, and move the sample bottles and caps to **measure and record the final mass**.

11.3.22. Set up to clean the sample lines, place the sample lines in a container containing methanol, and run the 'Cleaning Sample Lines program' using emptied columns (or rinse the plunger with methanol and press the plunger without columns).

11.3.23. After the lines are cleaned, continue with next set of samples or turn off the Zymark - place the sample lines in a plastic bag.

11.3.24. The acetonitrile extracts are collected in 8-mL test tubes. Adjust the final volume to 5.0 mL with acetonitrile containing 0.1% acetic acid. Mix the contents well.

11.3.25. Filter the acetonitrile extract through an Acrodisc LC25 filter assembly (or equivalent filter with 0.45 um pore size) using a 10mL disposable syringe into a new test tube. DO NOT READJUST THE VOLUME.

WARNING: Exercise caution when using syringes with attached filter assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

11.3.26. The acetonitrile extracts are refrigerated at 4 ± 2ºC. Dilute 4x with water prior to analysis. Be sure to mix the dilution well before transferring to vials for analysis.

11.3.27. The extracts are stored until ready for analysis.

11.4. Soil/Sediment Samples - Method 8330 / 8330A;

NOTE: Do not dry and grind tissue samples. Tissue samples are dried by mixing with anhydrous sodium sulfate.

WARNING: Do NOT grind samples containing high concentrations of explosives. Lumps of material that have a chemical appearance should be considered suspect and not ground. Explosives are generally a very finely ground grayish-white material.

11.4.1. **For Drying and Initial Sieving**; Sub-sample approximately 20 to 80 grams of soil into a disposable weighing boat for drying. Other types of containers may be used.

11.4.2. If the samples do not appear homogeneous and are not suspicious, dry the samples in a cool, ventilated area and away from direct light. **DO NOT HEAT** the samples.

11.4.3. Drying time of soil samples depends on how wet the samples are when received. A typical time for a damp soil to dry is approximately 8 to 24 hours, or less if the sample is spread out to increase surface area.

11.4.4. If samples do appear suspicious, stop work and consult with the project manager and client regarding possible high levels of explosives.

11.4.5. After the samples are dried, grind thoroughly in an appropriate apparatus (previously rinsed with acetonitrile and dried).

11.4.6. Sift the sample through a sieve size 30 mesh. If samples are not to be extracted on the same day, store the sifted soil in the freezer and protect from light.

11.4.7. **Sub-sampling and Extraction for Method 8330 / 8330A;** Weigh the dried, homogeneous sample into 40-mL vials. Weigh 2.0 grams into the 40-mL vials. For tissue samples weigh out a 2g aliquot of the homogenized tissue sample into a 40 mL vial. Add 5g of cleaned anhydrous sodium sulfate and mix thoroughly to dry the tissue.

11.4.7.1. For control, such as the MB and LCS or DCS, use Ottawa sand. For the MB and LCS aliquot for tissue extractions use Ottawa sand and sodium sulfate.

NOTE: If a larger sample size is desired, then the ratio of sample to solvent must be consistent to 1 gram/5 mL.

11.4.8. Surrogate is added to each sample, MB, and LCS and MS/MSD or DCS.

11.4.8.1. Add 80 uL of the 50 ug/mL 3,4-DNT surrogate solution to each 2.0 gram soil sample to yield 2.0 ppm.

11.4.9. If applicable, fortify the LCS, LCSD, and MS/MSD.

LCS/LCSD/MS/MSD Spiking – Solid Method 8330 / 8330A			
Requested Analyte	**Amount Added**	**Solution Concentration**	**Sample Concentration**
8330 + DNA	40 uL	50 ug/mL 8330	1.0 mg/kg
NG/PETN	200 uL	50 ug/mL NG/PETN	5.0 mg/kg
Picric Acid	200 uL	50 ug/mL PA	5.0 mg/kg

11.4.10. Add 10.0 mL of the 0.1% (v/v) acetic acid in acetonitrile solution for extraction, cap, and shake briefly to mix the contents well.

11.4.11. Place the samples in the ultrasonic bath, and sonicate for 18 hours, keeping the bath at 35 °C or cooler. Protect the samples from light by covering the sonication bath.

11.4.12. Allow the samples to settle for 30 minutes or longer, or centrifuge the samples at approximately 1,200 rpm for approximately 10 minutes.

11.4.13. Decant or filter the extracts (minimally 4 mL) into 8mL test tubes. The extracts may be worked up immediately or refrigerated at 4 ± 2ºC until ready for further prep.

11.4.14. If necessary, filter the soil extracts by transferring into 10mL disposable syringes fitted with 0.45 um PTFE 25-mm filters, and filter them with positive pressure using the plunger into a clean 8mL screw cap test tubes.

WARNING: Exercise caution when using syringes with attached filter assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

11.4.15. Dilute the extracts 4× by aliquotting 2.0 mL of the filtered extract in an 8mL screw cap test tube with 6.0 mL of the 1.3 g/L calcium chloride solution. Shake briefly to mix the contents.

11.4.16. Filter the diluted extracts with disposable syringes fitted with 0.45 um PTFE 25-mm filters, and filter them into test tubes and into 2 mL vials for analysis.

WARNING: Exercise caution when using syringes with attached filter assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

11.4.17. Refrigerate the extracts at 4 ± 2ºC and protect from light until analysis. The final sample concentration is 2.0g/40mL.

11.5. Method 8330B - Drying and Initial Sieving

11.5.1. Spread the entire sample evenly on a tray covered with fresh aluminum foil in a fume hood or in a well ventilated area to minimize exposure to dust. Moist samples should be placed on baker's racks or another location which allows for proper ventilation. Use a wooden spatula to spread the material out and to break up the soil into small pieces; this is critical for clay soils. Allow to air-dry until there are no signs of visible moisture. This typically takes 24 hours but may take more or less time depending on how wet the samples are when received.

11.5.2. When the sample is dry, remove any obvious organic materials such as leaves and twigs. Remove any non-representative material such as large rocks.

Document the removal of any material on the benchsheet and/or in an NCM.

11.5.3. Employ an appropriate disaggregation techniques (e.g., preliminary grinding in mortar and pestle) if needed to break up loose clumps. Mosses and other

types of fine vegetation should be physically shredded while sieving to release trapped soil and residues.

11.5.4. Carefully sieve the sample from the metal tray using a #10 sieve (2mm mesh). Clients may ask to weigh the individual portions for particle size analysis. Record all observations on the appropriate laboratory benchsheet.

11.5.5. Transfer the materials with particle size greater than the designated particle size back into the original sample container. The portion of the sample that has been prepared for grinding and sub-sampling is now ready for further processing.

11.5.6. Samples collected from ammunition plants and depots should be ground with an acetonitrile rinsed mortar & pestle until finely divided. Following grinding the samples are ready for incremental subsampling.

11.5.7. Samples from firing ranges and impact zones can contain particles of explosives at a variety of sizes, shapes, and compositions. Therefore the entire sample must be processed further prior to removal of the subsample for analysis. For these samples only the ring and puck mill should be used. Samples collected at the firing point can contain nitrocellulose fibers. These fibers present a special problem in the grinding step. In order to get the fibers to release the target analytes they must be very finely ground. Refer to SOP WS-QA-0028 for details of grinding with the ring and puck mill.

11.5.7.1. If the ring and puck mill is used for grinding, a grinding LCS (section 9.11) and a composite grinding blank (section 9.13) must be prepared. In addition, a grinding triplicate (section 9.12) is also required.

11.6. **Method 8330B Incremental Sub-Sampling**: Proceed with this step only if directed by QAS instructions, or by the associated TALS Method codes. Otherwise, proceed to the 'Extraction for 8330B Soil/Sediment' section. Refer also to SOP WS-QA-0028, "Incremental Sampling Methodology of Soils and Sediments".

11.6.1. **Incremental Sub-sampling**; Spread out the entire ground sample on a sheet of aluminum foil to approximately 1 cm thickness.

11.6.2. Collect about 30 increments from random locations through the entire thickness, top to bottom, of the layer of ground material using a square-ended spatula into an appropriate container. Record the sample weight on the benchsheet.

11.6.3. Routine subsample sizes:

Explosives = 10 g

Metals = 10 g

Other extractable organics = 30 g

Special project instructions can indicate amounts other than those listed.

11.6.4. Store the subsamples in the proper location for the test being conducted until they are ready for further preparation

11.7. Extraction for 8330B Soil/Sediment

11.7.1. Extraction is performed for 8330B on 10 gram sub-sampled dried and ground soils from the preceding sections in 40-mL glass vials.

11.7.1.1. For control, such as the MB and LCS, use Ottawa sand.

11.7.1.2. If the samples have been subjected to grinding and incremental subsampling, there should be ground CRM, a grinding blank composition, and a grinding triplicate associated with them. Be sure to include them in the batch preparation.

11.7.2. Surrogate is added to each sample, MB, and LCS and MS/MSD.

11.7.2.1. Add 100 uL of the 50 ug/mL 3,4-DNT surrogate solution to each10.0 gram soil sample to yield 0.50 ppm.

11.7.3. If applicable, fortify the LCS, LCSD, and MS/MSD.

LCS/LCSD/MS/MSD Spiking – Solid Method 8330B			
Requested Analyte	**Amount Added**	**Solution Concentration**	**Sample Concentration**
8330 + DNA	100 uL	50 ug/mL 8330	0.50 mg/kg
NG/PETN	200 uL	50 ug/mL NG/PETN	1.0 mg/kg
Picric Acid	200 uL	50 ug/mL PA	1.0 mg/kg

11.7.4. Add 20.0 mL of the 0.1% (v/v) acetic acid in acetonitrile solution for extraction, cap, and shake briefly to mix the contents well.

11.7.5. Place the samples in the ultrasonic bath, and sonicate for 18 hours, keeping the bath at 35 °C or cooler. Protect the samples from light by covering the sonication bath.

11.7.6. Allow the samples to settle for 30 minutes or longer, or centrifuge the samples at approximately 1,200 rpm for approximately 10 minutes.

11.7.7. Decant or filter the extracts (minimally 4 mL) into 8mL test tubes. The extracts may be worked up immediately or refrigerated at 4 ± 2ºC until ready for further prep.

11.7.8. If necessary, filter the soil extracts by transferring into 10mL disposable syringes fitted with 0.45 um PTFE 25-mm filters, and filter them with positive pressure using the plunger into a clean 8mL screw cap test tubes.

WARNING: Exercise caution when using syringes with attached filter assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

11.7.9. Dilute the extracts 4× by aliquotting 2.0 mL of the filtered extract in an 8mL screw cap test tube with 6.0 mL of the 1.3 g/L calcium chloride solution. Shake briefly to mix the contents.

11.7.10. Filter the diluted extracts with disposable syringes fitted with 0.45 um PTFE 25-mm filters, and filter them into test tubes.

WARNING: Exercise caution when using syringes with attached filter assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

11.7.11. Refrigerate the extracts at 4 ± 2ºC and protect from light until analysis. The final sample concentration is 10.0g/80mL.

11.8. Preparation of wipe samples

Assuming the wipe samples are collected specifically for each of the above tests and are not shared for other analysis, then proceed as follows:

11.8.1. Dry the wipe samples in the drying cabinet (at room temperature and protect from light).

11.8.2. If possible, transfer the wipe sample to 40-mL vials. Do not over-stuff the vials, and the amount of surrogate to add will depend how much of the extracting solvent is necessary to submerge the wipe sample.

11.8.3. If the wipe sample is to be extracted in its original container, then it will be necessary to determine several factors:

- How much extracting solvent is needed to submerge the wipe?
- Can the samples to be secured in the sonication bath for 18 hours, or
- can the samples be shaken on the platform shaker for 4 hours if the shake method technique is acceptable?

11.8.4. For controlled matrix on the wipe, use filter paper (the MB and LCS). Fold the paper and place it in the extraction vessel (40-mL vials). Also run a RB (reagent blank: without the filter paper) if data is not available on the background of the filter paper.

11.8.5. Add surrogate to the wipe samples, based on using 20 mL of extraction solvent. Add 400 µL of the 50 µg/mL 3,4-DNT surrogate to yield 20.0

µg/wipe.

11.8.6. If applicable, fortify the LCS, LCSD, and MS/MSD. The values below assume 20 mL of extraction solvent.

LCS/LCSD/MS/MSD Spiking – Solid Method (wipes)			
Requested Analyte	**Amount Added**	**Solution Concentration**	**Sample Concentration**
8330 + DNA	80 uL	50 ug/mL 8330	4.0 ug/wipe
NG/PETN	400 uL	50 ug/mL NG/PETN	20 ug/wipe
Picric Acid	400 uL	50 ug/mL PA	20 ug/wipe

11.8.7. If a larger amount of the extracting solvent is required for wipe samples, then stock solutions at higher levels may be used for fortification:

- The 8330 mix solution at 1.0 mg/mL.
- 3,4-Dinitrotoluene at 1.0 mg/mL.
- 3,5-Dinitroaniline at 1.0 mg/mL.
- Picric Acid at 1.0 mg/mL.
- PETN at 100 ug/mL.
- Nitroglycerin at 1.0 mg/mL.

11.8.8. Add 20 mL (or enough to submerge the wipe, whichever is greater) of 0.1% (v/v) acetic acid in acetonitrile, cap, and shake briefly to mix the contents well. (If adding more than 20 mL, be sure to adjust the spiking volume to maintain the final concentration of surrogate and matrix spike solution).

11.8.9. Sonicate for 18 hours (or shake on the reciprocating shaker for 4 hours).

11.8.10. Allow the extracts to settle and filter approximately 8 mL of the extracts into 8mL test tubes. For a wipe that was extracted with 20 mL, the extract concentration is 1.0 wipe/20mL.

11.8.10.1. The excess may be discarded according to waste disposal protocol.

11.8.11. Dilute the extracts 4 times with water. A 2.0mL aliquot of the 1 wipe/20mL extract is diluted with 6.0 mL of HPLC grade water, and mixed well. The final extract concentration is 1 wipe / 80 mL, or 0.10 wipe / 8.0mL, and refrigerated at 4 ± 2ºC until ready for analysis.

11.9. Analytical Conditions – Primary Analysis

11.9.1. The conditions listed below are the recommended analytical conditions for 8330 by HPLC/UV. If changes to the HPLC conditions are necessary, they will be noted in the maintenance log.

<table>
<tr><td>Column</td><td>Synergi Hydro-RP 250 x 4.6 mm, 5 um, or equivalent</td></tr>
<tr><td>Column Temperature</td><td>Approximately 32°C (The column may be kept to a constant temperature with a recirculating cooler).</td></tr>
<tr><td>Mobile Phase</td><td>
<table>
<tr><th>RESERVOIR</th><th>SOLVENT</th></tr>
<tr><td>A</td><td>Water</td></tr>
<tr><td>B</td><td>50:50 Water:Methanol + 0.4% phosphoric acid</td></tr>
<tr><td>C</td><td>Acetonitrile</td></tr>
</table>
</td></tr>
<tr><td>Gradient program (updated conditions are posted on the instrument or in the maintenance log, as they are subject to change based on the column lot.)</td><td>
<table>
<tr><th>Time, minutes</th><th>%A</th><th>%B</th><th>%C</th></tr>
<tr><td>0.0</td><td>8</td><td>85.5</td><td>6.5</td></tr>
<tr><td>29</td><td>85.5</td><td>7.5</td><td>7</td></tr>
<tr><td>31</td><td>0</td><td>100</td><td>0</td></tr>
</table>
</td></tr>
<tr><td>Flow Rate</td><td>1.0 mL/min</td></tr>
<tr><td>Injection Volume</td><td>500 µL</td></tr>
</table>

11.9.2. Elution order, approximate retention times, and detector wavelength used for quantitation under the conditions listed above:

Analytes	minutes	Wavelength (Channel)
HMX	5.21	250 nm (1)
RDX	7.46	250 nm (1)
Picric Acid	8.50	250 nm (1) / 358 nm (2) 358 nm used for quantitation
TNB	9.73	250 nm (1)
DNB	12.27	250 nm (1)
3,5-DNA	12.96	250 nm (1)
TETRYL	13.28	250 nm (1)
NB	13.93	250 nm (1)
Nitroglycerin	14.46	205 nm (2)
TNT	15.33	250 nm (1)
4-AM-2,6-DNT	15.80	250 nm (1)
3,4-DNT (surrogate)	16.25	250 nm (1) / 205 nm (2)
2-AM-4,6-DNT	16.74	250 nm (1)
2,6-DNT	18.25	250 nm (1)
2,4-DNT	18.88	250 nm (1)
2-NT	21.99	265 nm (1)
4-NT	23.58	265 nm (1)
3-NT	25.34	265 nm (1)
PETN	27.25	205 nm (2)

Notes:

- Nitroglycerin and PETN do not respond at 250nm detection.
- Picric Acid responds on both 250 nm and 358 nm. Picric Acid is

reported from 358 nm, confirmed on the second wavelength at 250 nm.

11.9.3. Preventative Maintenance:

- Change in-line frit as needed to retain proper pressure, peak shape and response.
- The use of guard column is highly recommended but can cause poor resolution and poor peak shape.
- On occasion, the 50:50 Water:Methanol + 0.4% phosphoric acid solution may stratify, causing retention time shifts and coelutions. Remake the solution, and mix well, then reprime the HPLC system with the new mobile phase prior to evaluating a reinjection of the CCV.

***Note**: Separation of TNT and 4-AM, 3-NT and PETN, and 3,4-DNT and nitroglycerin on the primary column may depend on the composition of methanol/water and acetonitrile in the mobile phase.*

***Note**: The elution order of 4-AM and TNT is in reverse order from the manufacturer's elution order when sufficient amount of acetonitrile is added to the mobile phase to get the separation between nitroglycerin and 3,4-DNT and between 3-NT and PETN.*

11.10. Confirmation Conditions

All samples for 8330 analysis (not including picric acid at this time) are subject to confirmation using the cyano column. At a minimum, field samples with positive detections above the reporting limit must be confirmed. Specific programs, such as DOD QSM or client QAPP may require confirmation of positives above the method detection limit.

The conditions listed below are the recommended analytical conditions for confirmation of 8330 by HPLC/UV. If changes to the HPLC conditions are necessary, they will be noted on the chromatogram in the maintenance log.

<table>
<tr><td>Column</td><td>Zorbax Cyano CN 250 x 4.6 mm, 5 um, or equivalent</td></tr>
<tr><td>Column Temperature</td><td>Approximately 15°C (The column may be kept to a constant temperature with a recirculating cooler).</td></tr>
<tr><td>Mobile Phase</td><td><table><tr><th>RESERVOIR</th><th>SOLVENT</th></tr><tr><td>A</td><td>Water</td></tr><tr><td>B</td><td>Acetonitrile</td></tr><tr><td>C</td><td>Methanol</td></tr></table></td></tr>
<tr><td>Gradient program (updated conditions are posted on the instrument or in the maintenance log, as they are subject to change based on the column lot.)</td><td><table><tr><th>Time, minutes</th><th>%A</th><th>%B</th><th>%C</th></tr><tr><td>0.0</td><td>74</td><td>13</td><td>13</td></tr><tr><td>25</td><td>60</td><td>30</td><td>10</td></tr><tr><td>52</td><td>30</td><td>70</td><td>0</td></tr></table></td></tr>
<tr><td>Flow Rate</td><td>0.85 mL/min</td></tr>
<tr><td>Injection Volume</td><td>500 µL</td></tr>
</table>

11.10.1. Elution order, approximate retention times, and detector wavelength used for quantitation under the conditions listed above:

Analyte	RT (minutes)	Wavelength (Channel)
NB	21.98	250 nm (1)
DNB	24.98	250 nm (1)
TNB	27.75	250 nm (1)
2-NT	29.12	250 nm (1)
4-NT	29.12	250 nm (1)
3-NT	29.71	250 nm (1)
3,5-DNA	31.10	250 nm (1)
RDX	31.71	250 nm (1)
2,4-DNT	32.83	250 nm (1)
2,6-DNT	33.86	250 nm (1)
2-AM-4,6-DNT	35.95	250 nm (1)
4-AM-2,6-DNT	36.61	250 nm (1)
3,4-DNT (surrogate)	38.28	250 nm (1)
TNT	40.41	250 nm (1)
HMX	42.12	250 nm (1)
Nitroglycerin	44.65	205 nm (2)
TETRYL	46.35	250 nm (1)
PETN	52.85	205 nm (2)

- 2-NT and 4-NT co-elute in this method.
- Depending on the CN column, column temperature, and the gradient conditions, 2-AM and 4-AM may co-elute.
- Nitroglycerin and PETN are confirmed under the sample HPLC conditions, with the UV absorbance at 205 nm.
- Picric acid is confirmed on a second wavelength (250 nm) on the primary column. Second column is not use unless requested.

11.11. Confirmation Conditions – PFP column

The PFP column is used when a confirmation analysis requiring resolution between 2-NT and 4-NT is necessary. The conditions below are recommended, and may require adjustment to improve peak resolution.

<table>
<tr><td>Column</td><td>Phenomenex PFP, 150 x 4.6mm, 2um, or equivalent</td></tr>
<tr><td>Column Temperature</td><td>Approximately 22°C (The column may be kept to a constant temperature with a recirculating cooler).</td></tr>
<tr><td>Mobile Phase</td><td><table>
<tr><th>RESERVOIR</th><th>SOLVENT</th></tr>
<tr><td>A</td><td>Water</td></tr>
<tr><td>B</td><td>1:1 Water:Methanol + 0.4% phosphoric acid</td></tr>
<tr><td>C</td><td>Acetonitrile</td></tr>
<tr><td>D</td><td>Methanol</td></tr>
</table></td></tr>
<tr><td>Gradient program (updated conditions are posted on the instrument or in the maintenance log, as they are subject to change based on the column lot.)</td><td><table>
<tr><th>Time, minutes</th><th>%A</th><th>%B</th><th>%C</th><th>%D</th><th>Flow (ml/min)</th></tr>
<tr><td>0.0</td><td>0</td><td>100</td><td>0</td><td>0</td><td>0.4</td></tr>
<tr><td>6.0</td><td>0</td><td>100</td><td>0</td><td>0</td><td>0.4</td></tr>
<tr><td>10</td><td>0</td><td>98</td><td>2</td><td></td><td>0.4</td></tr>
<tr><td>45</td><td>0</td><td>89</td><td>5</td><td>6</td><td>0.4</td></tr>
<tr><td>55</td><td>0</td><td>60</td><td>30</td><td>10</td><td>0.4</td></tr>
<tr><td>60</td><td>0</td><td>10</td><td>30</td><td>60</td><td>0.4</td></tr>
<tr><td>62</td><td>0</td><td>0</td><td>0</td><td>100</td><td>0.5</td></tr>
<tr><td>64</td><td>0</td><td>0</td><td>0</td><td>100</td><td>0.5</td></tr>
</table></td></tr>
<tr><td>Injection Volume</td><td>150 µL</td></tr>
</table>

11.12. Analytical Conditions – Primary Analysis, "Long Run"

11.12.1. The conditions listed below are the recommended analytical conditions when the metabolites are requested in addition to the normal analyte list. If changes to the HPLC conditions are necessary, they will be noted in the maintenance log.

<table>
<tr><td>Column</td><td>Synergi Hydro-RP 250 x 4.6 mm, 5 um, or equivalent</td></tr>
<tr><td>Column Temperature</td><td>approximately 30°C (The column may be kept to a constant temperature with a recirculating cooler).</td></tr>
<tr><td>Mobile Phase</td><td><table>
<tr><th>RESERVOIR</th><th>SOLVENT</th></tr>
<tr><td>A</td><td>Water</td></tr>
<tr><td>B</td><td>Acetonitrile</td></tr>
<tr><td>C</td><td>50:50 Water:Methanol + 0.4% phosphoric acid</td></tr>
</table></td></tr>
<tr><td>Gradient program (updated conditions are posted on the instrument or in the maintenance log, as they are subject to change based on the column lot.)</td><td><table>
<tr><th>Time, minutes</th><th>%A</th><th>%B</th><th>%C</th><th>%D</th></tr>
<tr><td>0.0</td><td>65</td><td>5</td><td>30</td><td>0</td></tr>
<tr><td>30</td><td>60</td><td>5</td><td>32</td><td>3</td></tr>
</table></td></tr>
<tr><td>Flow Rate</td><td>1.0 mL/min</td></tr>
<tr><td>Injection Volume</td><td>500 µL</td></tr>
</table>

11.12.2. Elution order, approximate retention times, and detector wavelength used for quantitation under the conditions listed above:

Analytes	minutes	Wavelength (Channel)
TNX	16.44	250 nm (1)
DNX	19.36	250 nm (1)
HMX	20.10	250 nm (1)
MNX	21.15	250 nm (1)
RDX	22.41	250 nm (1)
Picric Acid	24.77	250 nm (1) / 358 nm (2) 358 nm used for quantitation
TNB	24.17	250 nm (1)
DNB	27.58	250 nm (1)
3,5-DNA	29.15	250 nm (1)
NB	29.29	250 nm (1)
TETRYL	29.72	250 nm (1)
Nitroglycerin	30.18	205 nm (2)
TNT	31.11	250 nm (1)
4-AM-2,6-DNT	32.87	250 nm (1)
3,4-DNT (surrogate)	33.17	250 nm (1) / 205 nm (2)
2-AM-4,6-DNT	33.80	250 nm (1)
2,6-DNT	34.92	250 nm (1)
2,4-DNT	35.49	250 nm (1)
2-NT	38.86	265 nm (1)
4-NT	40.48	265 nm (1)
3-NT	42.30	265 nm (1)
PETN	43.00	205 nm (2)

Notes:

- Nitroglycerin and PETN do not respond at 250nm detection.
- Picric Acid responds on both 250 nm and 358 nm. Picric Acid is reported from 358 nm, confirmed on the second wavelength at 250 nm.
-

11.13. Analytical Conditions – Primary Analysis, “Short Column”

11.13.1. The conditions listed below are the recommended analytical conditions for 8330 by HPLC/UV. If changes to the HPLC conditions are necessary, they will be noted on the chromatogram. This column is used for specialty analysis when reduced numbers of analytes are requested, and may not always achieve complete resolution of every analyte.

<table>
<tr><td>Column</td><td>Extend C-18 100 x 3.6 mm, 3.55 um, or equivalent</td></tr>
<tr><td>Column Temperature</td><td>approximately 30°C (The column may be kept to a constant temperature with a recirculating cooler).</td></tr>
<tr><td>Mobile Phase</td><td><table><tr><th>RESERVOIR</th><th>SOLVENT</th></tr><tr><td>A</td><td>Water</td></tr><tr><td>B</td><td>Acetonitrile</td></tr><tr><td>C</td><td>5 mM Ammonium Formate in Water</td></tr></table></td></tr>
<tr><td>Gradient program (updated conditions are posted on the instrument or in the maintenance log, as they are subject to change based on the column lot.)</td><td><table><tr><th>Time, minutes</th><th>%A</th><th>%B</th><th>%C</th><th>%D</th></tr><tr><td>0.0</td><td>0</td><td>0</td><td>25</td><td>75</td></tr><tr><td>6</td><td>0</td><td>0</td><td>30</td><td>70</td></tr><tr><td>11</td><td>0</td><td>0</td><td>35</td><td>65</td></tr></table></td></tr>
<tr><td>Flow Rate</td><td>2.5 mL/min</td></tr>
<tr><td>Injection Volume</td><td>500 µL</td></tr>
</table>

12. CALCULATIONS/DATA REDUCTION

12.1. Qualitative Identification Tentative identification occurs when a peak for an analyte is found within the established retention time window at a concentration above the reporting limit, or above the MDL if J flags are required. Confirmation is required on the cyano column or at a second UV wavelength, depending on the analyte. Client specific requirements may also define the need for second column confirmation and/or HPLC/MS confirmation. Identification is confirmed if a peak is also present in the retention time window for that analyte on the confirmatory column, at a concentration greater than the reporting limit (for DOD, to the MDL).

12.1.1. In the event that a compound identified on the primary column is not confirmed by second column analysis, the compound is reported as ND.

12.1.2. If a compound identified on the primary column is confirmed by second column analysis, the result from the primary (C18) column is reported.

12.1.3. If the RPD between the values on the confirmation column and the primary column is ≥ 40%, the data is flagged in the LIMS system and documented on a Non-Conformance Memo (NCM).

12.2. If concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. It may be necessary to dilute samples due to matrix.

12.3. Results are reported in mg/kg (ppm) for soil and µg/L (ppb) for aqueous, unless otherwise instructed.

12.4. Results are reported as dry weight since soil samples are air dried, ground with mortar and pestle, and sieved, unless otherwise specified by the client.

12.5. Unless otherwise specified by client/program, report only one dilution for each sample. Report the lowest dilution without overrange peaks. When specified by client/program (e.g., DOD QSM), report all valid dilutions as separate analyses in the LIMS.

12.6. The concentration of each analyte and surrogate in a sample is calculated as follows:

Equation 5

$$AmountFound(ppb) = \frac{V_x}{W_s} \times \frac{A_x}{\overline{Rf}}$$

Where:

Rf = Average Response Factor in ng/mL
A_x = Sample Total Area Counts
V_x = Sample Extract Final volume in mL
W_s = Initial Sample weight in g

13. METHOD PERFORMANCE

13.1. The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for reach analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006.

13.2. The laboratory must make a one time initial demonstration of capability for each individual method. Demonstration of capability for both solid and aqueous matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests is may be necessary to use more than one QC check mix to cover all analytes of interest.

13.2.1. Four aliquots of the QC check samples are analyzed using the same procedure used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid level calibration.

13.2.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these results with the acceptance criteria given in each appendix.

13.2.3. If any analyte does not meet the acceptance criteria, then test must be repeated. Only those analytes that did not meet criteria in the first test need

to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

14. 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 Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.1. Do not allow waste solvent to vent into the hoods. All solvent waste is stored in capped containers unless waste is being transferred.

14.2. Transfer waste solvent from collection cups (tri-pour and similar containers) to jugs and/or carboys as quickly as possible to minimize evaporation.

15. 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 SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Miscellaneous disposable glassware, chemical resistant gloves, bench paper and similar materials that may or may not be contaminated/hazardous. Place contaminated materials into a yellow contaminated lab trash bucket. When the bucket is full or after no more than one year, tie the plastic bag liner shut and put the lab trash into the appropriate steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.2. Extracted soil samples, used sodium sulfate, paper funnel filters, glass wool, thimbles, and extracted sediment tissue solids contaminated with solvents. Dump these materials into an orange contaminated lab trash bucket. When the bucket is full or after no more than one year, tie the plastic bag liner shut and put the lab trash into the appropriate steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.3. Waste Acetone/Acetonitrile/Methanol. Collect the waste solvents in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel waste solvent drum in the H3 closet. When full to between two and six inches

of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment.

15.4. Mixed water/methanol/acetonitrile waste from soil extraction and from instrument analysis. Collect the waste in the HPLC waste carboy. When full, or after no more than one year, dump into the blue plastic HPLC collection drum in the H3 closet. When full to no less than six inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment.

16. REFERENCES/CROSS REFERENCES

16.1. SW-846 Method 8330, "Nitroaromatics and Nitramines by High Pressure Liquid Chromatography (HPLC)", Revision 0, September 1994, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C.

16.2. SW-846 Method 8330A, "Nitroaromatics and Nitramines by High Pressure Liquid Chromatography (HPLC)", Revision 1, January 1998, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C.

16.3. SW-846 Method 8330B, “Nitroaromatics, Nitramines, and Nitrate Esters by High Performance Liquid Chromatography (HPLC), Revision 2, October 2006, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C.

16.4. SW-846 Method 3535, "Solid Phase Extraction (SPE)", Revision 0, December, 1996, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C.

16.5. SW-846 Method 3535A, "Solid Phase Extraction (SPE)", Revision 1, January, 1998, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C.

16.6. Thomas F. Jenkins, et. al., "Comparison of Cartridge and Membrane Solid-Phase Extraction with Salting-Out Solvent Extraction for Preconcentration of Nitroaromatic and Nitramine Explosives from Water", Special Report 92-95, December 1992, US Army Corps of Engineers, Cold Regions Research & Engineering Laboratory, Hanover, New Hampshire.

16.7. "Quality Control Program", WS-PQA-003

16.8. “Calibration Curves and Selection of Calibration Points”, CA-Q-P-003,

16.9. “Data Recording Requirements”, WS-PQA-008

16.10. EPA Guidance for Assessing Chemical Contaminant Data for us in Fish Advisories (Vol. 1, Table 7.1, "Recommendation for Container Materials, Preservation, and Holding Times for Fish, Shellfish, and Turtle Tissue from Receipt of Sample Processing Laboratory to Analysis")

16.11. "Federal Program Requirements", WS-PQA-021.

17. METHOD MODIFICATIONS

17.1. Deviations from reference method: This method deviates from EPA method 8330, September, 1994 revision on the following:

17.2. Acetonitrile is our preferred solvent instead of methanol in the preparation of aqueous standards.

17.3. The final solvent for extracts for HPLC/UV analysis should be no more than 25% acetonitrile in water. Higher percentages of acetonitrile cause poor chromatography.

17.4. A gradient program is run using Water/Methanol with THF and Acetonitrile as modifiers instead of isocratic at 50/50 Methanol/Water.

17.5. The amount of calcium chloride solution (5g/L aqueous) added to the soil extracts is 2:8 and is less than the original method of 1:1. Calcium chloride solution is a flocculating agent that aids in the filtration of soil extracts. Higher concentrations may cause clogging of the frit and over-pressurizing the HPLC.

17.6. The UV detector is set at 250 nm instead of 254 nm.

17.7. The injection volume is 500 uL instead of 100 uL.

17.8. Deviation from EPA Method 8330B: In some instances, solids from firing ranges are analyzed without the grinding procedure, at client request. This deviation should be documented in the case narrative by the program manager.

18. ATTACHMENTS

18.1. Table 1-Analytes and Reporting Limits

18.2. Table 2-Spiking Solutions

18.3. Table 3-Calibration Standards

18.4. Table 4-Additional Calibration Standards

18.5. Table 5- Fortifications of 8330 by HPLC/UV in aqueous, soil, and wipe matrices

19. REVISION HISTORY

19.1. WS-LC-0009, Revision 5.4, Effective 07/15/2016

19.1.1. Section 1.4 - Updated to include DOE projects.

19.1.2. Section 9.3.1 and Section 9.10, – Changed 'may' to 'must'.

19.1.3. Section 10.6.3 – Updated - "Recommended concentrations are given in Table 3, with Level 5 suggested for the first calibration verification and Level 4 suggested for subsequent calibration verification."

19.1.4. Section 11.9.1 - Updated Table to reflect current laboratory procedure.

19.1.5. Section 12.1.3 – added 'and documented on a Non-Conformance Memo (NCM)" to end of paragraph.

19.1.6. Section 11.10 – Updated Table to reflect current solvent partitions.

19.1.7. Remove the revision history entries dating 11/5/2010 and earlier. These may be found in previous versions of this SOP.

19.1.8. Editorial changes.

19.2. WS-LC -0009, Revision 5.3, Effective 06/05/2015

19.2.1. Table 3 – Deleted column 8 (1000 ng/mL calibration standard.

19.2.2. Table 4 – Deleted column 1 (10 ng/mL) and column 7 (1000 ng/mL) calibration standards for Metabolites.

19.2.3. Editorial changes

19.3. WS-LC-0009, Revision 5.2, Effective 06/17/2014

19.3.1. Changed Section 10.4.11 from, "…a five point calibration is performed every 6 months." to "…a calibration curve is valid for 12 months."

19.3.2. Changed Table 11.9.2, 11.10.1, and 11/12/2 wavelength settings that were 200nm to 205 nm. The affected analytes are PETN and Nitroglycerin.

19.3.3. Editorial changes.

19.4. WS-LC-0009, Revision 5.1, Effective 05/17/2013

19.4.1. Added line in Section 11.3.6 Table (Low Level) for DNX, TNX, and MNX.

19.4.2. Removed Section 11.4 – Solid Phase Extraction(SPE) on Aqueous Samples, using the Horizon and vacuum manifold.

19.4.3. Editorial changes.

19.5. WS-LC-0009, Revision 5, Effective 1/18/2012

19.5.1. Removed references to photo-diode array detection, and salting-out preparations.

19.5.2. Updated instrument information to include current instrumentation.

19.5.3. Moved column descriptions and specifications to the equipment section.

19.5.4. Added reference to additional compounds not previously discussed in the SOP.

19.5.5. Added remarks regarding standards from ISO 17025 accredited firms.

19.5.6. Updated analytical conditions to include new columns.

19.5.7. Clarified QC requirements for Method 8330B ground samples.

19.5.8. Added reference to new grinding SOP WS-OP-0025.

19.5.9. Removed Revision History prior to 2009.

Table 1
Analytes and Reporting Limits

Test Components	CAS Number	Abbr.	Solid Phase ug/L	DAI ug/L	8330 8330A Solid mg/kg	8330B Solid mg/kg	Wipes ug/sa
2-Amino-4,6-dinitrotoluene	35572-78-2	2-AM	0.20	10	0.25	0.25	0.40
4-Amino-2,6-dinitrotoluene	1946-51-0	4-AM	0.10	10	0.25	0.25	0.40
3,5-Dinitroaniline	618-87-1	3,5-DNA	0.5	25	0.50	0.50	NA
1,3-Dinitrobenzene	99-65-0	DNB	0.10	4.0	0.25	0.25	0.40
2,4-Dinitrotoluene	121-14-2	2,4-DNT	0.10	5.7	0.25	0.25	0.40
2,6-Dinitrotoluene	606-20-2	2,6-DNT	0.10	9.7	0.25	0.25	0.40
Ethylene glycol dinitrate	628-96-6	EGDN	0.65	50	0.50	0.50	2.0
Glycerol Trinitrate (Nitroglycerin)	55-63-0	NG	0.65	25	0.50	0.50	2.0
Hexahydro-1,3,5-trinitro-1,3,5triazine (Hexogen)	121-82-4	RDX	0.10	14	0.25	0.25	0.40
Methyl-2,4,6trinitrophenylnitramine	479-45-8	TETRYL	0.10	4.0	0.25	0.25	0.40
Nitrobenzene	98-95-3	NB	0.10	6.4	0.25	0.25	0.40
2-Nitrotoluene (o-Nitrotoluene)	88-72-2	2-NT	0.50	12	0.25	0.25	0.40
3-Nitrotoluene (m-Nitrotoluene)	99-08-1	3-NT	0.50	7.9	0.25	0.25	0.40
4-Nitrotoluene (p-Nitrotoluene)	99-99-0	4-NT	0.50	8.5	0.25	0.25	0.40
Octahydro-1,3,5,7tetranitro1,3,5,7-tetracine (Octogen)	2691-41-0	HMX	0.10	13	0.25	0.25	0.40
Picric Acid	88-89-1	PA	1.0	50	0.50	0.50	2.0
Pentaerythritol Tetranitrate	78-11-5	PETN	0.65	50	0.50	0.50	2.0
1,3,5-Trinitrobenzene	99-35-4	TNB	0.10	7.3	0.25	0.25	0.40
2,4,6-Trinitrotoluene	118-96-7	TNT	0.10	6.9	0.25	0.25	0.40
DNX (1-Nitro-3,5-dinitroso-1,3,5-triazacyclohexane)	80251-29-2	DNX	2.0	NA	NA	NA	NA
TNX (1,3,5-Trinitroso-1,3,5-triazacyclohexane)	13980-04-6	TNX	2.0	NA	NA	NA	NA
MNX (1-Nitroso-3,5-dinitro-1,3,5-triazacyclohexane)	5755-27-1	MNX	2.0	NA	NA	NA	NA

Table 2 Spiking Solutions			
Test Components - Method 8330	**CAS Number**	**Abbreviation**	**Conc'n ug/mL**
8330 List			
2-Amino-4,6-dinitrotoluene	35572-78-2	2-AM	50
4-Amino-2,6-dinitrotoluene	1946-51-0	4-AM	50
1,3-Dinitrobenzene	99-65-0	DNB	50
2,4-Dinitrotoluene	121-14-2	2,4-DNT	50
2,6-Dinitrotoluene	606-20-2	2,6-DNT	50
Hexahydro-1,3,5-trinitro-1,3,5-triazine (Hexogen)	121-82-4	RDX	50
Methyl-2,4,6-trinitrophenylnitramine	479-45-8	TETRYL	50
Nitrobenzene	98-95-3	NB	50
2-Nitrotoluene (o-Nitrotoluene)	88-72-2	2-NT	50
3-Nitrotoluene (m-Nitrotoluene)	99-08-1	3-NT	50
4-Nitrotoluene (p-Nitrotoluene)	99-99-0	4-NT	50
Octahydro-1,3,5,7-tetranitro1,3,5,7tetracine (Octogen)	2691-41-0	HMX	50
1,3,5-Trinitrobenzene	99-35-4	TNB	50
2,4,6-Trinitrotoluene	118-96-7	TNT	50
3,5-Dinitroaniline	618-87-1	3,5-DNA	50
NG/PETN			
Glycerol Trinitrate (Nitroglycerin)	55-63-0	NG	50
Pentaerythritol Tetranitrate	78-11-5	PETN	50
Surrogate			
3, 4-Dinitrotoluene	610-39-9	3,4-DNT	50
Additional Compounds (May be evaluated as separate mixes, or included in the 8330 mix, based on coelutions and request frequency)			
Ethylene glycol dinitrate (EGDN[1])	628-96-6	EGDN	50
2,4,6-Trinitrophenol (Picric Acid)	88-89-1	PA	50
DNX (1-Nitro-3,5-dinitroso-1,3,5-triazacyclohexane)	80251-29-2	DNX	50
TNX (1,3,5-Trinitroso-1,3,5-triazacyclohexane)	13980-04-6	TNX	50
MNX (1-Nitroso-3,5-dinitro-1,3,5-triazacyclohexane)	5755-27-1	MNX	50

[1] EGDN may be included with the NG/PETN spiking mix, at the same concentration.

Table 3 Calibration Standards							
Component	**Levels, ng/mL**						
	1	**2**	**3**	**4**	**5**	**6**	**7**
8330 compounds							
2-Amino-4,6-dinitrotoluene	5.0	10	20	50	100	200	500
4-Amino-2,6-dinitrotoluene	5.0	10	20	50	100	200	500
1,3-Dinitrobenzene	5.0	10	20	50	100	200	500
2,4-Dinitrotoluene	5.0	10	20	50	100	200	500
2,6-Dinitrotoluene	5.0	10	20	50	100	200	500
Hexahydro-1,3,5-trinitro-1,3,5-triazine (Hexogen)	5.0	10	20	50	100	200	500
Methyl-2,4,6-trinitrophenylnitramine	5.0	10	20	50	100	200	500
Nitrobenzene	5.0	10	20	50	100	200	500
2-Nitrotoluene (o-Nitrotoluene)	5.0	10	20	50	100	200	500
3-Nitrotoluene (m-Nitrotoluene)	5.0	10	20	50	100	200	500
4-Nitrotoluene (p-Nitrotoluene)	5.0	10	20	50	100	200	500
Octahydro-1,3,5,7-tetranitro1,3,5,7tetracine (Octogen)	5.0	10	20	50	100	200	500
1,3,5-Trinitrobenzene	5.0	10	20	50	100	200	500
2,4,6-Trinitrotoluene	5.0	10	20	50	100	200	500
3,5-Dinitroaniline	5.0	10	20	50	100	200	500
Specialty Explosives							
Nitroglycerin	-	10	20	50	100	200	500
PETN	-	10	20	50	100	200	500
EGDN		10	20	50	100	200	500
Picric Acid							
Picric Acid	-	20	40	100	200	400	1000
Surrogate							
3,4-Dinitrotoluene	5.0	10	20	50	100	200	300

Note: 3,5-DNA may be calibrated separately if coelutions occur.

Note: Nitroglycerin and PETN may be analyzed using UV detection at about 200 nm. The responses for both decrease at higher wavelengths, and they are not detected at 250nm which is the wavelength used for the other 8330 compounds.

Note: Similarly, it is recommended to use the Level 4 or 5 standard or subsequent CCVs.

Table 4 Metabolite Calibration Standards					
Component	**Levels, ng/mL**				
	1	**2**	**3**	**4**	**5**
DNX	20	50	100	200	500
MNX	20	50	100	200	500
TNX	20	50	100	200	500

Table 5 Fortifications of 8330 by HPLC/UV in aqueous, soil, and wipe matrices					
	Aqueous, ppb			**Soil**	**Wipe**
Analytes	**DAI**	**High Level SPE**	**Low Level SPE**	**ppm**	**ug/sa**
3,4-DNT (surr)	100	100	2.5	5.0	20
HMX	50	50	1.0	1.0	4.0
TNB	50	50	1.0	1.0	4.0
RDX	50	50	1.0	1.0	4.0
DNB	50	50	1.0	1.0	4.0
NB	50	50	1.0	1.0	4.0
TNT	50	50	1.0	1.0	4.0
2,4-DNT	50	50	1.0	1.0	4.0
2,6-DNT	50	50	1.0	1.0	4.0
2-AM	50	50	1.0	1.0	4.0
4-AM	50	50	1.0	1.0	4.0
2-NT	50	50	1.0	1.0	4.0
4-NT	50	50	1.0	1.0	4.0
3-NT	50	50	1.0	1.0	4.0
Nitroglycerin	100	100	5.0	5.0	20
PETN	100	100	5.0	5.0	20
EGDN	100	100	5.0	5.0	20
Picric Acid	100	100	5.0	5.0	20
3,5-DNA	100	100	2.5	5.0	20
DNX	--	--	1.0	--	--
MNX	--	--	1.0	--	--
TNX	--	--	1.0	--	--

Title: Determination of Polycyclic Aromatic Hydrocarbons (PAH) by GC/MS-SIM Internal Standard Technique [Method 8270C & 8270D]

Approvals (Signature/Date):

Koroush Vaziri Operations Manager	6-9-16 Date
Joe Schairer Health & Safety Manager / Coordinator	6/27/16 Date
Lisa Stafford Quality Assurance Manager	6/27/16 Date
Crystal Pollock Laboratory Director	6/27/16 Date

1. SCOPE AND APPLICATION

1.1. This method is suitable for the analysis of PAHs in solid, aqueous, and oily matrices by Method 8270C SIM and 8270D SIM analysis. Refer to Table 1 for a list of individual PAHs determined by this method.

1.2. The standard reporting limit (SRL) of this method for determining an individual compound is approximately 5ug/kg (wet weight) for soil/sediment samples, 50ng/L for groundwater samples, and 500ug/kg or higher for oily matrices samples. Refer to TALS for specific SRLs. Reporting limits will be proportionately higher for samples that require dilution.

1.3. This method is restricted to use by, or under the supervision of, analysts experienced in the use of capillary column gas chromatography/mass spectrometry. Because of the toxicity of the materials known or believed to contain PAH, certain precautions must be taken to prevent exposure to the analyst or to others.

1.4. When undertaking projects for Department of Defense (DoD) and or Department of Energy (DOE) the relevant criteria in QA Policy WS-PQA-021 must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. The analytical method is gas chromatography combined with internal standard mass spectrometry. This entails the addition of extraction surrogates to all samples in known quantities, matrix-specific extraction of the sample with appropriate organic solvents, and analysis of the processed extract for PAHs using high-resolution capillary column gas chromatography coupled with low mass spectrometry (GC/MS) in selective ion monitoring mode (SIM). Analyte concentrations are calculated using the internal standard technique.

3. DEFINITIONS

3.1. Large Volume Injection (LVI) – An injection into a gas chromatograph that is larger than the typical 1 or 2µL injection used for hot vaporizing injectors.

3.2. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.3. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. Interferences may be caused by contaminants in solvents, reagents, sorbents, glassware and other sample processing hardware that may lead to discrete artifacts and/or elevated backgrounds at the ions monitored. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

4.2. The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.1. Specific Safety Concerns or Requirements

5.1.1. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex and vinyl gloves provide no protection against the organic solvents used in this method. Nitrile gloves must be used.

5.1.2. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.3. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.1.4. Ensure that all instrument exhaust vents and lines are properly connected to either a laboratory vent or an appropriate filter. Instruments may not be vented to the working environment.

5.2. Primary Material Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS**

for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. See SOP WS-OP-0001 or WS-OP-0006 for details of sample preparation equipment and supplies.

6.2. GCMS Analytical System

6.2.1. Gas Chromatograph/Mass Spectrometer (GC/MS) – This method uses GC instrumentation manufactured by Agilent, Model 6890, or equivalent, and a Mass Spectrometer manufactured by Agilent, Model 5973 MS, or equivalent. The GC injection port must be designed for capillary columns. Splitless injection is recommended. See Table 8 for the recommended column for this analysis.

6.2.2. Mass Spectrometer - The mass spectrometer (MS) must be capable of operation in the Selective Ion Monitoring mode at a resolving power of 1 amu. Electron impact ionization must be used. The mass spectrometer must be capable of monitoring all of the ions listed in each of the four SIM descriptors (Table 9) with a total cycle time of 1 second or less.

6.2.3. GC/MS Interface

Any gas chromatograph to mass spectrometer interface may be used as long as it gives acceptable calibration response for each analyte of interest at the desired concentration and achieves the required tuning performance criteria. To achieve maximum sensitivity, the exit end of the capillary column should be placed in the mass spectrometer ion source without being exposed to the ionizing electron beam.

6.3. Data Acquisition System

A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all data obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and plot a Selected Ion Current Profile (SICP), a plot of the abundances of the selected ions versus time or scan number. Software must also be available to integrate, in any SICP, the abundance between specified time or scan-number limits. The data system must provide hard copies of individual ion chromatogram's for selected gas chromatographic time intervals..

6.3.1. Acquisition software: ChemStation Software version D.

6.3.1.1. The system provides the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program.

6.3.2. Data Processing Software: Chrom version. 2.1

6.3.2.1. The software can search any GC/MS data file for ions of a specific mass and can plot such ion abundances versus time or scan number. This type of plot is defined as the Extracted Ion Current Profile (EICP). .

6.3.2.2. The software allows integrating the abundances in any EICP between specified time or scan-number limits.

6.3.2.3. The most recent version of the NIST Mass Spectral Library is recommended.

7. REAGENTS AND STANDARDS

7.1. See WS-OP-0001 or WS-OP-0006 for details of reagents related to sample preparation.

7.2. Stock Standard Solution

Standard solutions can be prepared from pure standard materials or purchased as certified solutions (see analyte lists in Tables 2-4). Stock standard solutions must be replaced after one year.

7.2.1. Expiration times for all standards are measured from the time the standard is prepared or from the time that the standard ampule is opened, if the standard is supplied in a sealed ampule. If a vendor-supplied date has an earlier expiration than the date from preparation, the earlier date is used.

7.2.2. Neat Standard Materials - Neat materials expire following a period of 3 years

from the date of receipt, or sooner if problems such as degradation occur.

7.2.3. Working Standard Solutions - Working standards expire following a period of 1 year from the preparation date or the expiration date of the stock solution, whichever is earlier. The solution may be replaced sooner if evidence of degradation is observed.

7.3. Preparation of Stock Solutions

7.3.1. Internal Standards - Prepare or purchase a stock solution in $MeCl_2$ of the five internal standards listed in Table 3 at concentrations of 2000 ug/mL.

7.3.2. Store stock standard solutions in Teflon-sealed screw-cap bottles at 0-6°C and protect from light. Stock standard solutions must be checked frequently for signs of degradation and evaporation, especially just before using them to prepare calibration standard solutions or spiking solutions.

7.3.2.1. Replace stock standard solutions every 12 months or more frequently if comparison with quality control check samples indicates a problem.

7.3.3. Calibration Standards

Prepare calibration working standard solutions by combining appropriate volumes of individual or mixed calibration standards with extraction surrogate stocks, internal standard stocks and diluting to volume with methylene chloride to obtain the solution concentrations given in Table 4. The suggested range is 50 ng/mL to 5000 ng/mL and 25 ng/mL to 1000 ng/mL for LVI . Calibration solutions must be replaced after one year from the date of preparation, or sooner if empirical data indicates the standard has undergone concentration or degradation. If one of the parent solutions has an earlier expiration date, that date must be used.

7.3.3.1. Prepare an Initial Calibration Verification (ICV) or Second Source Calibration Standard (SSCS), when available, to confirm the calibration curve. This standard will be analyzed after the initial calibration.

7.3.3.1.1. All standards must be stored at 0-6°C and must be freshly prepared if the calibration verification, internal standard, or native analyte standard indicates a problem.

7.3.4. Internal Standard Spiking Solution

Use an appropriate volume of stock solution to prepare an internal standard spiking solution in methylene chloride with the concentrations shown in Table 3. Store at 6°C or less. Spike 10 µL of the internal standard spiking solution to the final sample extract just prior to analysis to achieve a final extraction of 0.5 µg/mL.

7.3.5. Calibration Verification Standard

7.3.5.1. The calibration verification standard shall be used for column performance checks to verify peak separation, and for daily calibration checks. Solution #3 or #4 from Table 4 shall be the calibration verification standard.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Refer to SOP WS-OP-0001 for sample preservation and storage.

8.2. Extracts are stored at < -10°C until at least 30 days following invoicing.

8.3. Extracts must be analyzed within 40 days of extraction.

9. QUALITY CONTROL

9.1. Control Limits

The laboratory will establish historically derived recovery limits for laboratory control samples (LCS), matrix spikes (MS), and surrogates. QAPP or project specific limits may supersede, if mutually agreed upon.

9.1.1. All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into TALS (when available) or other database so that accurate historical control charts can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.1.2. Refer to the QC Program document (WS-PQA-003) for further details of control limits.

9.2. Internal Standards

Internal standards are components similar in nature to the analytes of interest. These are added to every sample (including QC aliquots) and standard analyzed. The purpose is to enable calculations based on internal standard methodology. Internal standard recoveries are monitored to verify that instrument performance is acceptable. Criteria for standards are delineated in Section 10. If the internal standard data indicate instrument failure, the samples may require reanalysis. If not, the impact on sample data is evaluated and the data is flagged appropriately.

9.2.1. The internal standard (IS) responses in all QC and field samples are compared to the mid-point standard of the initial calibration. Alternatively, the IS response in the samples may be compared to the daily CCV, on a per batch basis, to meet client or program requirements.

9.2.2. Any samples that do not meet the same IS criteria set forth for the CCV (Section 10.4) must be evaluated for validity. If the change in internal standard response is a matrix effect confined to an individual sample, reanalysis may not be necessary, but should be verified by the department manager or client. If the change in internal standard response is due to instrumental problems, all affected samples must be reanalyzed after the problem is corrected.

9.2.3. Any IS responses outside the criteria should be noted in the case narrative.

9.3. Extraction Surrogate Spike

9.3.1. Recoveries for each of the extraction surrogates must be within the historically derived, or QAPP specific recovery limit. If the extracts are diluted > 5x the surrogates will be diluted out, and not reportable. The data flag of NC-SRD will be used to indicate this.

9.3.2. If the extraction surrogate standard recoveries are outside of the acceptable limits, the cause of the failure should be investigated. Otherwise the sample is re-extracted if sufficient sample volume is available; subject to client approval, re-extraction of a smaller sample aliquot may be appropriate to mitigate low surrogate recovery caused by matrix interferences.

9.3.3. Recommended Corrective Actions for surrogate failures in MB, LCS, or LCSD (batch QC)

9.3.3.1. Check all calculations for errors.

9.3.3.2. Verify satisfactory instrument performance.

9.3.3.3. Recalculate the data and/or reanalyze the extracts if either of the above checks reveals a problem.

9.3.3.4. If the problem persists the QC and associated samples may require re-extraction.

9.3.3.5. Review the analytical procedures with the performing laboratory personnel.

9.3.3.6. Re-extract the samples and associated QC if necessary (Example-

low surrogate recovery in MB)

9.3.3.7. Document the failure in an anomaly, and any corrective action in the narrative.

9.3.4. Corrective actions for surrogate failures in field samples or MS/MSD:

9.3.4.1. Check all calculations for error.

9.3.4.2. Ensure that instrument performance is acceptable.

9.3.4.3. Recalculate the data and/or reanalyze the extract if either of the above checks reveals a problem.

9.3.4.4. Evaluate objective evidence of matrix interference (e.g. heterogeneous sample, interfering compounds seen on chromatograms, or interferences demonstrated by prior analyses)

9.3.4.5. Re-extract if necessary.

9.3.4.6. Document the failure and note it on the final report.

9.4. Method Blanks

One method blank must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. For aqueous samples, the method blank is an aliquot of laboratory reagent water, and for solid samples, the method blank is an aliquot of sodium sulfate. For air samples, the method blank may be an aliquot of XAD resin, PUF, filter, or other matrix that is representative of the sample matrix. The QC aliquots are processed in the same manner and at the same time as the associated samples. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 10% of the measured concentration of that analyte in the associated samples, whichever is higher. The laboratory will attempt to run all samples from a preparation batch within the same sequence on the same instrument. If this is not possible, samples may be run on different sequences or different instruments, and will be accompanied by an instrument blank.

9.4.1. Reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

9.4.2. If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be done in consultation with the client.

9.4.3. If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B," and appropriate comments may be made in a narrative to provide

further documentation.

9.4.4. Refer to the QC Program document (WS-PQA-003) for further details of the corrective actions.

9.5. Laboratory Control Samples (LCS)

A laboratory control sample (LCS) is prepared and analyzed with every batch of samples. All analytes must be within established control limits. The LCS is spiked with the compounds listed in Table 5 unless otherwise specified by a client or agency.

9.5.1. If any analyte in the LCS is outside the method control limits, corrective action must occur. Corrective action may include re-extraction and reanalysis of the batch.

9.5.2. If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. (An example of acceptable reasons for not reanalyzing might be that the LCS recovery is high, the samples are ND, and sample surrogate recoveries are good).

9.5.3. If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.5.4. Ongoing monitoring of the LCS provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike/matrix spike duplicate (MS/MSD) is prepared and analyzed with every batch of samples for aqueous and solid matrices, where sufficient sample volume is available. The MS/MSD is spiked with the same set of analytes as the LCS (See Table 5). Compare the percent recovery and relative percent difference (RPD) to that in the laboratory specific historically generated limits.

9.6.1. If any individual recovery or RPD falls outside the acceptable range, corrective action must occur (WS-PQA-003, Section 7.6.5). The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.

9.6.2. If the recovery for any component is outside QC limits for both the MS/MSD

and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include re-preparation and reanalysis of the batch.

9.6.3. If an MS/MSD is not possible due to limited sample, then a LCS duplicate may be analyzed if required by the program or client. RPD of the LCS and LCSD are compared to the matrix spike limits.

9.6.4. The MS/MSD must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.7. Nonconformance and Corrective Action

Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

9.8. Quality Assurance Summaries

Certain clients may require specific project or program QC which may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

9.9. QC Program

Further details of QC and corrective action guidelines are presented in the QC Program document (WS-PQA-003). Refer to this document if in doubt regarding corrective actions.

10. CALIBRATION

10.1. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-S-005 “Calibration Curves (General)”.

10.2. Initial Calibration

The initial calibration is required before any samples are analyzed, and then intermittently throughout sample analyses as dictated by results of the continuing calibration procedures described in this section, and after any major maintenance. The GC/MS system must be properly calibrated and the performance documented during the initial calibration.

10.2.1. GC/MS Tuning Criteria

Use a compound such as perfluorotributylamine (PFTBA) to verify that the intensity of the peaks is acceptable. If PFTBA is used, mass spectral peak profiles for m/z 69, 219, and 264 must be recorded, plotted and reported. The scan should display a minimum of ± two amu (i.e. m/z 67-71 for the m/z 69

profile) and the masses be within 0.50 amu of the target masses. The tune is verified at the beginning of every continuous sequence.

NOTE: The requirement to analyze the instrument performance check solution is optional when analysis of Polyaromatic Hydrocarbons (PAHs)/pentachlorophenol is to be performed by the Selected Ion Monitoring (SIM) technique. (USEPA CLP SOW1.2)

NOTE:The EPA CLP Functional Guidelines for semivolatile organic analysis, Section II (Gas Chromatograph/Mass Spectrometer (GC/MS) Instrument Performance Check), Item B (Objective), states "GC/MS instrument performance checks are performed to ensure adequate mass resolution, identification, and to some degree, sensitivity. These criteria are not sample-specific. Conformance is determined using standard materials, therefore, these criteria should be met in all circumstances. **NOTE: This requirement does not apply when samples are analyzed by the Selected Ion Monitoring (SIM) technique. "**

10.2.2. GC Operating Conditions

The GC column performance must be documented during every analytical sequence. Table 8 summarizes GC operating conditions known to produce acceptable results with the column listed. The GC conditions must be established for the particular instrument by injecting aliquots of the calibration check standard. The valley height between benzo(b)fluoranthene and benzo(k)fluoranthene must be no more than ½ the height of the taller peak. It may be necessary to adjust the operating conditions slightly based on observations from analysis of these solutions. Thereafter, the calibration check standard must be analyzed daily to verify the performance of the system.

10.3. Calibration Procedure for Internal Standard Method

10.3.1. The internal standards used in this method are listed in Table 3. Use the base peak m/z as the primary m/z for quantitation of the standards, and the secondary m/z for confirmation purposes only. If interferences are noted one of the next two most intense masses may be used for quantitation.

10.3.2. Compounds are assigned to the internal standards as indicated in Table 6.

10.3.3. Using stock standards, prepare at least five calibration standard solutions, using the same solvent that is used in the final sample extract. Keep the internal standards at fixed concentrations. Table 4 lists the recommended calibration standard concentrations.

10.3.4. Calibrate the mass spectrometer response using a 2-5 µL aliquot of each calibration solution. Each solution must be analyzed once. Calculate the response factors (RFs) for each analyte:

10.3.4.1. Analyze each calibration standard and tabulate the area of the primary characteristic m/z against concentration for each compound and internal standard. Calculate response factors (RF), average response factors, and the percent RSD of the response factors for each compound using the equations 1, 2, and 3 and verify that the criteria in Table 7 are met. **No sample analysis may be performed unless these criteria are met.**

10.3.4.2. Mean Response Factor - Use Equation 2 to calculate the mean RF for each compound (calibration standards and surrogate standards). This is the average of the five RFs calculated for each compound (one RF calculated for each calibration solution).

10.3.5. If the %RSD for any analyte exceeds 30% then a linear calibration should be tried for all analytes with %RSD > 30%. Linear or quadratic curve fits may be used. Use of 1/Concentration2 weighting is recommended to improve the accuracy of quantitation at the low end of the curve. The analyst should consider instrument maintenance to improve the linearity of response. Otherwise the correlation coefficient (coefficient of determination for non-linear curves) must be ≥ 0.990. The "Y" intercept (as printed on the Chrom ICAL plot) must be less than ± ½ RL. If the intercept exceeds this criterion, false positives or negatives could result. If the RL is elevated for client or project requirements, use the default RL or MDL.

10.3.6. Weighting of data points

In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. $1/\text{Concentration}^2$ weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.3.7. The %RSD should be $\leq$15% for DoD and AFCEE projects, and $\leq$25% for Method 8270D criteria.

10.3.8. Initial calibration verification standard (ICV) - When available, a second source standard should be used for ICV and analyzed with the initial calibration. Each compound must be within +/- 30% of its expected value.

Under the DoD QSM, the criteria are +/- 20%D for all analytes.

Corrective actions for a failed ICV include:

- Rerun the ICV.
- Remake or acquire a new ICV.
- Evaluate the instrument conditions.
- Evaluate the initial calibration standards
- Narrate all affected data.

10.3.9. If time remains in the 12-hour period initiated by initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration.

10.3.10. Quantitation is performed using the calibration curve or average response factor from the initial curve, not the continuing calibration.

10.4. Continuing Calibration (Calibration Check)

The calibration check standard must be analyzed at the beginning of each analysis period, or at the beginning of every 12-hour shift if the laboratory operates during consecutive 12-hour shifts.

10.4.1. Open the perfluorotributylamine (PFTBA) to verify that the intensity of the peaks is acceptable. The mass spectral peak profiles for m/z 69, 219, and 264 must be recorded, plotted and reported. The scan should display a minimum of ± two amu (i.e. m/z 67-71 for the m/z 69 profile). The analysis must produce masses within 0.50 amu of the target masses to show acceptable spectral profiles. Use the same data acquisition parameters as those used during the initial calibration. This verification is performed at the beginning of every consecutive sequence and must be performed at least once a day

10.4.2. Inject a 2-5ul aliquot of the calibration check solution into the GCMS. Use the same data acquisition parameters as those used during the initial calibration.

10.4.3. Update the retention time windows for each of the compounds based on the daily standard. The retention time for the calibration standard must not change by more than 30 seconds from the most recent calibration check if no maintenance has been performed since the last check. If the retention time shifts by more than 30 seconds and no maintenance has been performed, inspect the chromatographic system for malfunctions and make the necessary corrections. Document acceptable performance with a new initial calibration curve.

10.4.4. Calculate the response factors. Refer to Section 12 for the appropriate equations.

10.4.5. Calculate the delta RF (ΔRF) which is the Relative Percent Difference (RPD) between the daily RF and the initial calibration mean RF. The measured RFs of all analytes (native and surrogate) must be within the criteria noted in Table 7, of the mean values established during the initial calibration. If this criterion is not satisfied, assess the potential impact on the data, if any possible impact is suspected appropriate corrective action must be performed. The corrective action may include re-injection of the continuing calibration standard, instrument maintenance, or injection of a new initial calibration curve, before sample extracts can be analyzed.

10.4.6. Check the EICP areas of the internal standards for the Continuing Calibration Verification Standard (CCV) versus the mid-point standard level of the most recent initial calibration sequence. If the area changes by a factor of greater than two (-50% to +100%), the analytical system must be inspected to determine the cause of the change and corrections must be made, as appropriate. If corrections are made, the CCV must be analyzed again to verify the system is operating properly. If a system malfunction is found, the samples analyzed while the system was malfunctioning must be evaluated for potential impacts, and reanalyzed as necessary.

10.4.7. Recommended corrective actions for CCV failure:

- Re-analysis CCV.
- Instrument maintenance.
- A new initial calibration curve.

10.4.8. Once the above criteria have been met, sample analysis may begin.

11. PROCEDURE

11.1. Sample Extraction

Procedures for separatory funnel liquid/liquid extraction, sonication extraction, K-D concentration and N-Evap Concentration are described in SOP WS-OP-0001. Procedures for the extraction of air samples are described in SOP WS-OP-0006.

11.2. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A

Nonconformance memo shall be used for this documentation.

11.3. GC/MS Analysis

The laboratory may proceed with the analysis of QC and field samples only after demonstrating acceptable performance as specified in Section 10.

11.3.1. Calibrate the instrument as described in Section 10.

11.3.2. All samples must be analyzed using the same instrument conditions as the preceding continuing calibration standard.

11.3.3. Approximately 1 hour before analysis, remove the sample extracts from the freezer and the internal standard solution from the refrigerator and allow them to warm to room temperature.

11.3.4. Add 10 µL of the internal standard solution to all QC and filed samples to achieve a final concentration of 500 ng/mL. If the sample volume must be changed to achieve a desired detection limit, the internal standard solution concentration must be adjusted accordingly to achieve the target concentrations of Table 4.

11.3.5. Inject the sample extract into the instrument using the same injection technique as used for the standards.

11.3.6. The data system will determine the concentration of each analyte in the extract using calculations equivalent to those in Section 12. Quantitation is based on the initial calibration, not the continuing calibration.

11.3.7. Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst or automatically by the data system.

11.3.8. Target compounds identified by the data system are evaluated using the criteria listed in Section 12.1.

11.3.9. The presence of a given PAH is qualitatively confirmed if the criteria of Section 12.1 are satisfied. The response for any quantitation ion in the sample extract should not exceed the response of the highest concentration calibration standard. If the response exceeds the highest concentration calibration standard, the sample extract should be diluted to get the analyte response within the linear range of the calibration curve.

11.4. GC/MS Analysis - Large Volume Injection (LVI)

11.4.1. For samples being analyzed by LVI, add 5 µL of the internal standard solution

to all samples and QC to achieve a final concentration of 500 ng/mL. (Final extract volume for LVI samples is 0.5 mL). If the sample volume must be changed to achieve a desired detection limit, the recovery spike solution concentration must be adjusted accordingly to achieve the target concentrations of Table 5

11.4.2. Inject a 5 µL aliquot of the sample extract on the instrument. Recommended GC/MS operating conditions are described in Table 8.

11.5. Dilutions

If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the reporting limit and the matrix allows for analysis at a lesser dilution, the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.5.1. Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than the height of the internal standards, or if individual non-target peaks are less than two times the height of the internal standards, the sample should be reanalyzed at a less dilution. This requirement is approximate and subject to analyst judgment. For example, samples containing organic acids may need to be analyzed at a higher dilution to avoid destroying the column.

11.5.2. Reporting Dilutions

11.5.2.1. In general, the most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

11.5.2.2. Under DoD QSM, all valid dilutions for a sample must be reported.

11.5.3. Each time the sample extract is diluted, internal standard solution must be added to maintain a final concentration of 500 ng/mL for each internal standard component.

11.6. Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, refrigerate them at < -10°C, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

11.7. Retention time criteria for samples

If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.7.1. If the retention time of any internal standard in any sample varies by more than 0.1 minute from the preceding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.

11.8. Troubleshooting Guide

11.8.1. Daily Instrument Maintenance

In addition to the checks listed in the instrument maintenance schedule in the Sacrament QAM, the following daily maintenance may be performed.

- Clip column front or back as necessary.
- Replace injection port liner as necessary.
- Replace gold seal as necessary.
- Replace septum as necessary.
- Perform mass calibration as necessary

11.8.2. Major Maintenance

A new initial calibration is necessary following major maintenance. Major maintenance includes changing the column, cleaning the ion volume or repeller, cleaning the source, and replacing the multiplier. Refer to the manufacturer's manual for specific guidance.

12. CALCULATIONS/DATA REDUCTION

12.1. Qualitative Analysis

Identification Criteria: The presence of a given PAH is qualitatively confirmed if the criteria below are satisfied.

12.1.1. The elution of the sample component must compare to within +/- 0.2 minutes of the GC retention time of the standard component in the daily calibration check.

12.1.2. The characteristic ions of the component in the sample must match standard component characteristic ions.

12.1.3. A primary and secondary ion will be monitored for all labeled and unlabeled analytes, which have a secondary ion >10% of the primary. The secondary ion will be used for qualitative identification. The presence of the secondary

ion will be dependent upon the analyte concentration in the sample. The secondary ion may not be present for analytes detected at < 5x the reporting limit. Samples with concentrations of the target analyte 5-10x the reporting should have the secondary ions present at a detectable level.

12.1.4. The relative intensities of the ions should agree within +/- 30% between the standard and sample spectra.

12.1.5. If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst the identification is correct, the analyst shall report that identification with the appropriate footnote and proceed with quantitation.

12.1.6. If in the judgment of the analyst the separation of benzo(b)fluoranthene and benzo(k)fluoranthene is insufficient for quantitation, the pair may be reported as a total peak, based on the average response factor of the two.

12.1.7. The internal standard areas in the samples will be monitored against the mid-point initial calibration standard to evaluate possible instrument drift, but specific criteria or corrective actions would typically be defined in project specific QAPPs.

12.2. Calculations

12.2.1. Refer to policy QA-004-SAC for rules regarding significant digits in calculations. Carry out calculations retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after the final calculation.

12.2.2. Response Factors (RF) for native PAH and Surrogate Standards –Use the data obtained during initial calibration (10.2.) or continuing calibration (10.3).

Equation 1:

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

Ax = Area of the characteristic ion for the compound being measured

Ais = Area of the characteristic ion for the specific internal standard

Cx = Concentration of the compound being measured (µg/L)

Cis = Concentration of the specific internal standard (µg/L)

12.2.3. Mean Response Factors (RFavg) — Calculate the mean RF for each target PAH and surrogate standard, internal standard and alternate standard using Equation 2 and the RFs calculated according to Equation 1.

Equation 2:

$$RRF_{avg} = \frac{1}{n}\sum_{i=1}^{n}(RRF_i)$$

Where:

RRF = RRF calculated for calibration solution "i"

n = The number of data points derived from the calibration. The minimum requirement is a five-point calibration

12.2.4. Percent Relative Standard Deviation for Initial Calibration

Equation 3:

$$\%RSD = \frac{SD}{RF_{avg}} \times 100$$

Where:

RFavg = Mean of RFs from initial Calibration for a compound.

SD = Standard deviation of RFs from initial calibration for a compound.

$$= \sqrt{\sum_{i-1}^{n} \frac{(RFi - RFavg)^2}{n = 1}}$$

RFi = RF for calibration level i

n= number of RF values used.

12.2.5. Continuing Calibration ΔRF - ΔRF is the relative percent difference (RPD) between the daily RF and the mean RF calculated during initial calibration.

Equation 4:

$$\Delta RF = \frac{RF_c - RF_{avg}}{RF_{avg}} \times 100\%$$

Where:

RFavg = Mean response factor of a given analyte

RFc = The RF of a given analyte obtained from the continuing calibration

12.2.6. Total Mass of Target PAH or Surrogate Standard in Sample – (Ms)

Equation 5:

$$C_{ex} = \frac{R_x C_{is}}{R_{is}\overline{RF}}$$

Where:

C_{ex}= Concentration in extract, µg/mL

R_x = Response for analyte

R_{is}= Response for internal standard

C_{is} = Concentration of internal standard

12.2.7. Analyte Concentration in Sample, Aqueous Samples

Equation 6a:

$$\text{Concentration, } \mu\text{g / L} = \frac{C_{ex}V_t}{V_o}$$

Where:

V_t = Volume of total extract, µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1 mL extract will mean V_t = 10,000 µL)

V_o = Volume of water extracted (mL)

12.2.8. Analyte Concentration in Sample, Sediment/Soil, Sludge (on a dry-weight basis) and Waste (normally on a wet-weight basis):

Equation 6b:

$$\text{Concentration, } \mu\text{g / kg} = \frac{C_{ex}V_t}{W_sD}$$

Where:

W_s = Weight of sample extracted or diluted in grams

D = (100 - % moisture in sample)/100, for a dry weight basis or 1 for a wet weight basis

12.2.9. MS/MSD percent recovery calculation.

Equation 7:

$$\text{Matrix Spike Recovery} = \frac{S_{SR} - S_R}{S_A} \times 100\%$$

Where:

S_{SR} = Spike sample result

S_R = Sample result

S_A = Spike added

12.2.10. Relative % Difference calculation for the MS/MSD

Equation 8:

$$RPD = \frac{MS_R - MSD_R}{1/2(MS_R + MSD_R)} \times 100$$

Where:

RPD = Relative percent difference

MS_R = Matrix spike result

MSD_R = Matrix spike duplicate result

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit (MDL)

The laboratory must generate a MDL for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION PREVENTION

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.

15. WASTE MANAGEMENT

The following waste streams are produced when this method is carried out.

15.1. 2 milliliter autovials contaminated with methylene chloride, after extracts are analyzed. After analysis, the vials are returned to long-term storage. When released for disposal, they are moved into vial waste collection carboys. When the carboy is full, or after no more than one year, it is transferred to the waste disposal area, where the vials are run through the vial eater for disposal.

15.2. Waste methylene chloride from instrument needle cleaning process. Waste methylene chloride is collected in vials as part of the instrument operation. When full, the contents of the vial are poured into collection carboys. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel solvent drum in the H3 closet. When the drum is full to between two and six inches of the top,

or after no more than 75 days, move the steel drum to the waste collection area for shipment

16. REFERENCES/CROSS REFERENCES

16.1. “Handbook of Analytical Quality Control in Water and Wastewater Laboratories”, USEPA EMSL, EPA-600/4-79-019 (3/78)

16.2. CARB Method 429. Determination of Polycyclic Aromatic Hydrocarbons (PAH) Emissions from Stationary Sources. July, 1997.

16.3. Carcinogens - "Working with Carcinogens", Department of Health, Education and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77-206, Aug., 1977.

16.4. "OSHA Safety and Health Standards, General Industry," (29CFR1910), Occupational Safety and Health Administration, OSHA 2206, (Revised, January 1976).

16.5. "Safety in Academic Chemistry Laboratories", American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

16.6. USEPA CLP SOW for Organics Analysis SOM1.2 (6/2007) Section 9.2.1

16.7. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, December 1996, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique, Method 8270C.

16.8. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update IV, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique, Method 8270D, Revision 4, February 2007

17. METHOD MODIFICATIONS

17.1. There are no deviations from the method.

18. ATTACHMENTS

18.1. Table 1- PAH Target Analytes and Standard Reporting Limits

18.2. Table 2- Extraction Surrogate Spiking Solution Concentration

18.3. Table 3- Internal Standard Spiking Solution Concentration

18.4. Table 4- Concentrations of the PAH’s in Working GC/MS Calibration Standards

18.5. Table 4A - Concentrations of the PAH's in Working GC/MS Calibration Standards using Large Volume Analysis.

18.6. Table 5-Concentrations of Compounds in Laboratory Control Spike Samples (LCS)

18.7. Table 6- Assignment of Internal Standards for Calculating RFs and Quantiting Target PAH and Surrogate Standards

18.8. Table 7- Minimum Requirements for Response Factors for Initial and Continuing Calibrations

18.9. Table 8- Recommended GC Operating Conditions for PAH analysis

18.10. Table 9- Recommended MS Operating Conditions

19. REVISION HISTORY

19.1. WS-MS-0008, Revision 2.7, Effective 06/28/2016

19.1.1. Section 1.4 – Changed to "When undertaking projects for Department of Defense (DoD) and or Department of Energy (DOE) the relevant criteria in QA Policy WS-PQA-021 must be checked and incorporated.

19.1.2. Table 4 – Changed column 1 from 50ng/mL to 25 ng/mL; removed 300 ng/mL level (column 7).

19.1.3. Editorial changes.

19.2. WS-MS-0008, Revision 2.6, Effective 03/20/2015

19.2.1. Inserted Section 6.3.1 – Acquisition Software: Chemstation Software Version D.

19.2.2. Inserted Section 6.3.2 – Data Processing Software: Chrom version 2.1

19.2.3. Changed Section 10.3.5 - The "Y" intercept (listed as "b" on the ICAL summary pages) must be less than ± ½ RL

19.2.4. Editorial changes

19.3. WS-MS-0008, Revision 2.5, Effective 06/06/2014

19.3.1. Inserted Section 1.2 – "The standard reporting limit (SRL) of this method for determining an individual compound is approximately 5ug/kg (wet weight) for soil/sediment samples, 50ng/L for groundwater samples, and 500ug/kg or higher for oily matrices samples. Refer to TALS for specific SRLs. Reporting

limits will be proportionately higher for samples that require dilution."

19.3.2. Inserted Section 6.2 – "Preventive and routine maintenance is described in the "Schedule of Routine maintenance" in the QAM, Preventative Maintenance (Section 20.2) and Schedule of Routine Maintenance (Table 20.2)."

19.3.3. Inserted Section 11.8 – "Troubleshooting Guide

19.3.4. Editorial changes.

19.4. WS-MS-0008, Revision 2.4, Effective 06/07/2013

19.4.1. Changed values in Tables 1, 3, 3A, 4, 4A, 5, 5A, 6, 7, & 8 to conform with current laboratory practices.

19.4.2. Removed Benzo(e) pyrene from analyte list in all tables.

19.4.3. Editorial changes.

19.5. WS-MS-0008, Revision 2.3, Effective 04/06/2012

19.5.1. Modifications to the SOP for adding large volume analysis procedures. Tables 3A, 4A, 5A and 8A were added for large volume analysis

19.5.2. Section 6.2.1 was modified to include instrumentation makes and models.

19.5.3. Editorial changes.

19.6. WS-MS-0008, Revision 2.2, Effective 6/25/2010

19.6.1. Editorial revisions.

19.6.2. Inserted Section 9.2

19.6.3. PAH SIM criteria changed from 25-150% to 50-200%.

19.6.4. Inserted 1000 ng/mL and 3000 ng/mL levels in Table 4.

19.7. WS-MS-0008, Revision 2.1, Effective 9/04/2009

19.7.1. Added Section 1.5, "When undertaking projects for Department of Defense (DoD) the relevant criteria in QA Policy WS-PQA-021 "DoD QSM and AFCEE QAPP Implementation" must be checked and incorporated."

Table 1	
PAH Target Analytes	
Naphthalene	Pyrene
1-Methylnapthalene	Benzo(b)fluoranthene
2-Methylnapthalene	Benzo(k)fluoranthene
Acenaphthylene	Benzo(a)pyrene
Acenaphthene	Benzo(ghi)perylene
Fluorene	Indeno(123-cd)pyrene
Phenanthrene	Dibenz(ah)anthracene
Anthracene	Benzo(a)anthracene
Fluoranthene	Chrysene

Table 2	
Extraction Surrogate Spiking Solution Concentration	
Analytes	**ug/mL**
Nitrobenzene-d5	0.5
2-Fluorobiphenyl	0.5
Terphenyl-d14	0.5

Table 3	
Internal Standards Spiking Solution Concentration	
Analytes	**ug/mL**
Naphthalene-d8	50
Acenaphthene-d10	50
Phenanthrene-d10	50
Chrysene-d12	50
Perylene-d12	50

Table 4

Concentrations of the PAH's in Working GC/MS Calibration Standaards

Components	Solution Concentrations (ng/mL)						
	1	2	3	4	5	6	7
Calibration Standard							
Naphthalene	25	100	250	500	1000	2000	5000
1-Methylnaphthalene	25	100	250	500	1000	2000	5000
2-Methylnaphthalene	25	100	250	500	1000	2000	5000
Acenaphthene	25	100	250	500	1000	2000	5000
Acenanapthalene	25	100	250	500	1000	2000	5000
Fluorene	25	100	250	500	1000	2000	5000
Phenanthrene	25	100	250	500	1000	2000	5000
Anthracene	25	100	250	500	1000	2000	5000
Fluoranthene	25	100	250	500	1000	2000	5000
Pyrene	25	100	250	500	1000	2000	5000
Benzo(a)anthracene	25	100	250	500	1000	2000	5000
Chrysene	25	100	250	500	1000	2000	5000
Benzo(b)fluoranthene	25	100	250	500	1000	2000	5000
Benzo(k)fluoranthene	25	100	250	500	1000	2000	5000
Benzo(a)pyrene	25	100	250	500	1000	2000	5000
Benzo(ghi)perylene	25	100	250	500	1000	2000	5000
Indeno(123-cd)pyrene	25	100	250	500	1000	2000	5000
Dibenz(ah)anthracene	25	100	250	500	1000	2000	5000
Extraction Surrogate Standards							
Nitrobenzene-d5	25	100	250	500	1000	2000	5000
Fluorobiphenyl	25	100	250	500	1000	2000	5000
Terphenyl-d14	25	100	250	500	1000	2000	5000
Internal Standard							
Naphthalene-d8	500	500	500	500	500	500	500
Acenaphthene-d10	500	500	500	500	500	500	500
Phenanthrene-d10	500	500	500	500	500	500	500
Chrysene-d12	500	500	500	500	500	500	500
Perylene-d12	500	500	500	500	500	500	500

Table 4A **Large Volume Analysis (5uL)** **Concentrations of the PAH's in Working GC/MS Calibration Standaards**								
Components	Solution Concentrations (ng/mL)							
	1	2	3	4	5	6	7	8
Calibration Standard								
Naphthalene	25	50	100	200	400	600	800	1000
1-Methylnaphthalene	25	50	100	200	400	600	800	1000
2-Methylnaphthalene	25	50	100	200	400	600	800	1000
Acenaphthene	25	50	100	200	400	600	800	1000
Acenanapthalene	25	50	100	200	400	600	800	1000
Fluorene	25	50	100	200	400	600	800	1000
Phenanthrene	25	50	100	200	400	600	800	1000
Anthracene	25	50	100	200	400	600	800	1000
Fluoranthene	25	50	100	200	400	600	800	1000
Pyrene	25	50	100	200	400	600	800	1000
Benzo(a)anthracene	25	50	100	200	400	600	800	1000
Chrysene	25	50	100	200	400	600	800	1000
Benzo(b)fluoranthene	25	50	100	200	400	600	800	1000
Benzo(k)fluoranthene	25	50	100	200	400	600	800	1000
Benzo(a)pyrene	25	50	100	200	400	600	800	1000
Benzo(ghi)perylene	25	50	100	200	400	600	800	1000
Indeno(123-cd)pyrene	25	50	100	200	400	600	800	1000
Dibenz(ah)anthracene	25	50	100	200	400	600	800	1000
Extraction Surrogate Standards								
Nitrobenzene-d5	25	50	100	200	400	600	800	1000
Fluorobiphenyl	25	50	100	200	400	600	800	1000
Terphenyl-d14	25	50	100	200	400	600	800	1000
Internal Standard								
Naphthalene-d8	500	500	500	500	500	500	500	500
Acenaphthene-d10	500	500	500	500	500	500	500	500
Phenanthrene-d10	500	500	500	500	500	500	500	500
Chrysene-d12	500	500	500	500	500	500	500	500
Perylene-d12	500	500	500	500	500	500	500	500

TABLE 5

Concentrations of Compounds in Laboratory Control Spike Samples (LCS)

Compound	Aqueous (ng/L)	Solid (ng/g)
Naphthalene	500	25
1-Methylnapthalene	500	25
2-Methylnapthalene	500	25
Acenaphthylene	500	25
Acenaphthene	500	25
Fluorene	500	25
Phenanthrene	500	25
Anthracene	500	25
Fluoranthene	500	25
Pyrene	500	25
Benzo(a)anthracene	500	25
Chrysene	500	25
Benzo(b)fluoranthene	500	25
Benzo(k)fluoranthene	500	25
Benzo(ghi)perylene	500	25
Indeno(123-cd)pyrene	500	25
Dibenz(ah)anthracene	500	25

Table 6 Assignment of Internal Standards for Calculating RFs and Quantitating Target PAHs and Surrogate Standards	
Analyte	**Internal Standards**
Compound	
Naphthalene	Naphthalene-d8
1-Methylnaphthalene	Naphthalene-d8
2-Methylnaphthalene	Naphthalene-d8
Nitrobenzene-d5 (surr)	Naphthalene-d8
Acenaphthylene	Acenaphthene-d10
Acenaphthene	Acenaphthene-d10
Fluorene	Acenaphthene-d10
2-Fluorobiphenyl (surr)	Acenaphthene-d10
Phenanthrene	Phenanthrene-d10
Anthracene	Phenanthrene-d10
Fluoranthene	Phenanthrene-d10
Pyrene	Phenanthrene-d10
Terphenyl-d14 (surr)	Chrysene-d12
Benzo(a)anthracene	Chrysene-d12
Chrysene	Chrysene-d12
Benzo(b)fluoranthene	Perylene-d12
Benzo(k)fluoranthene	Perylene-d12
Benzo(a)pyrene	Perylene-d12
Indeno(1,2,3-c,d)pyrene	Perylene-d12
Dibenz(a,h)anthracene	Perylene-d12
Benzo(ghi)perylene	Perylene-d12

Table 7

Minimum Requirements for Response Factors for Initial and Continuing Calibrations

	8270C			8270D		
Analyte	Initial Calibration +/-% RSD	Second Source Criteria ΔRF (%)	Continuing Calibration Criteria ΔRF (%)	Initial Calibration ≤% RSD	Second Source Criteria ≤% RSD	Continuing Calibration Criteria ≤% RSD
Naphthalene	30	30	30	20	30	20
1-Methylnaphthalene	30	30	30	20	30	20
2-Methylnaphthalene	30	30	30	20	30	20
Acenaphthene	30	30	30	20	30	20
Acenaphthylene	30	30	30	20	30	20
Fluorene	30	30	30	20	30	20
Phenanthrene	30	30	30	20	30	20
Anthracene	30	30	30	20	30	20
Fluoranthene	30	30	30	20	30	20
Pyrene	30	30	30	20	30	20
Benzo(a)anthracene	30	30	30	20	30	20
Chrysene	30	30	30	20	30	20
Benzo(b)fluoranthene	30	30	30	20	30	20
Benzo(k)fluoranthene	30	30	30	20	30	20
Benzo(a)pyrene	30	30	30	20	30	20
Benzo(g,h,i)perylene	30	30	30	20	30	20
Indeno(1,2,3-c,d)pyrene	30	30	30	20	30	20
Dibenz(a,h)anthracene	30	30	30	20	30	20
Nitrobenzene-d_5	30	30	30	20	30	20
2-Fluorobiphenyl	30	30	30	20	30	20
d_{14}-Terphenyl	30	30	30	20	30	20

+Table 8 Recommended GC Operating Conditions for PAH analysis	
Parameter	Setting
Column	HP-5MS (or equivalent) 30m x 0.25mm id x 0.25µm film
Carrier Gas/Linear Velocity	He at 40 cm/sec
Injection Mode	Splitless
Temperature Program	60°C, hold 1.0 min, 18°C /min to 150 °C, 9°C /min. to 300°C, hold until after Benzo(g,h,i)perylene elutes (approximately 1.5 minutes)
Injector Temperature	250°C

Table 9

Recommended MS Operating Conditions

Parameter	Setting	Ch	Mass	Dwell (ms)	Component
Program 1					
Type	SIM	1	54	20	d_5-Nitrobenzene
High Mass	172	2	64	20	
Low Mass	54.0	3	68	20	d_8-Naphthalene
Resolution	1 amu	4	71	20	
Ionization Mode	EI+	5	82	20	Nitrobenzene-d5
Number of Channels	21	6	99	20	
Cycles/Second	1.00	7	102	20	Naphthalene
		8	112	20	
		9	127	20	Naphthalene
		10	128	20	Naphthalene Nitrobenzene-d5
		11	132	20	
		12	134	20	d_8-Naphthalene
		13	136	20	d_8-Naphthalene
		14	141	20	2-Methylnaphthalene, 1-Methylnaphthalene
		15	142	20	2-Methylnaphthalene, 1-Methylnaphthalene
		16	151	20	Acenaphthylene
		17	152	20	Acenaphthylene
		18	153	20	Acenaphthylene Acenaphthene
		19	154	20	Acenaphthene
		20	158	20	
		21	160	20	Acenaphthene-d10
		22	162	20	Acenaphthene-d10
		23	164	20	Acenaphthene-d10
		24	170	20	2-Fluorobiphenyl
		25	171	20	2-Fluorobiphenyl
		26	172	20	2-Fluorobiphenyl
Program 2					
Type	SIM	1	57	20	
High Mass	188	2	71	20	
Low Mass	80	3	80	20	d_{10}-Phenanthrene

Table 9

Recommended MS Operating Conditions

Parameter	Setting	Ch	Mass	Dwell (ms)	Component
Resolution	1 amu	4	85	20	
Ionization Mode	EI+	5	94	20	d_{10}-Phenanthrene
Number of Channels	10	6	141	20	
		7	165	20	Fluorene
		8	166	20	
		9	174	20	
		10	176	20	Phenanthrene, Anthracene
		11	178	20	Phenanthrene, Anthracene
		12	184	20	d_{10}-Phenanthrene
		13	188	20	d_{10}-Phenanthrene
		14	264	20	
		15	266	20	
		16	268	20	
		17	330	20	
		18	332	20	
Program 3					
Type	SIM	1	57	20	
High Mass	244	2	71	20	
Low Mass	100	3	85	20	
Resolution	1 amu	4	101	20	Fluoranthene, Pyrene
Ionization Mode	EI+	5	106	20	
Number of Channels	16	6	114	20	Benzo(a)anthracene, Chrysene
Cycles/Second		7	120	20	d_{12} –Chrysene
		8	122	20	d_{14}-Terphenyl
		9	200	20	Fluoranthene, Pyrene
		10	202	20	Fluoranthene, Pyrene
		11	208	20	
		12	212	20	d_{14}-Terphenyl
		13	226	20	Benzo(a)anthracene Chrysene
		14	228	20	Benzo(a)anthracene Chrysene
		15	236	20	d_{12} –Chrysene
		16	240	20	d_{12} –Chrysene

Table 9

Recommended MS Operating Conditions

Parameter	Setting	Ch	Mass	Dwell (ms)	Component
		17	244	20	d_{14} –Terphenyl
Program 4					
Type	SIM	1	57	20	
High Mass	279	2	71	20	
Low Mass	125	3	8/5	20	
Resolution	1 amu	4	126	20	Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene
Ionization Mode	EI+	5	132	20	d_{12}-Perylene
Number of Channels	16	6	138	20	Indeno(1,2,3-cd)pyrene Benzo(g,h,i)perylene
Cycles/Second		7	139	20	Dibenz(a,h)anthracene
		8	146	20	
		9	252	20	Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene
		10	253	20	Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene
		11	260	20	d_{12}-Perylene
		12	264	20	d_{12}-Perylene
		13	276	20	Indeno(1,2,3-cd)pyrene Benzo(g,h,i)perylene
		14	277	20	Indeno(1,2,3-cd)pyrene Benzo(g,h,i)perylene
		15	278	20	Dibenz(a,h)anthracene
		16	279	20	Dibenz(a,h)anthracene
		17	284	20	
		18	288	20	
		19	292	20	

Title: Extraction of Semivolatile Organic Compounds for Analysis by Method 8270C and 8270D, Based on SW-846 3500 Series and 3600 Series, and PAH-SIM by Internal Standard and Isotope Dilution Procedures

[Methods 3510C, 3550B/C, 3546, and 3580]

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1. SCOPE AND APPLICATION

This SOP describes procedures for preparation (extraction and concentration) of semivolatile organic analytes in aqueous, TCLP leachate, soil and waste matrices for analysis by Gas Chromatography/Mass Spectrometry (GC/MS). The procedures are based on SW-846 and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA).

1.1. Extraction procedures for the following determinative methods are covered: 8270C and 8270C (modified). 8270C (modified) encompasses analysis for 1,4-Dioxane, PAHs by Selected Ion Monitoring (PAH-SIM), and PAHs by Selected Ion Monitoring-Isotope Dilution (PAH-SIM-ID). The extraction procedures here may be appropriate for other determinative methods when appropriate spiking mixtures are used.

1.2. Other extraction procedures (PFE, SPE, accelerated Soxhlet, etc.) may be used but are not currently covered in this SOP.

1.3. Refer to WS-PQA-021 for criteria specific for DoD/DOE projects.

2. SUMMARY OF METHOD

2.1. Separatory Funnel Extraction: A measured volume of sample is adjusted, if necessary, to a specified pH and serially extracted with methylene chloride using a separatory funnel.

2.1.1. This method is subdivided into two extraction methods: a 1000mL nominal sample volume extracted in 2L separatory funnels (henceforth referred to as a 1L separatory funnel extraction in this SOP), and a 250mL sample volume extracted in a 500mL separatory funnels (henceforth referred to as a Large Volume Injection/Reduced Volume Initiative or LVI/RVE in this SOP).

2.2. Microwave Extraction: A measured weight of sample, typically 10g, is extracted with methylene chloride in a microwave extraction apparatus.

2.3. Sonication Extraction: A measured weight of sample, typically 30 g, is mixed with anhydrous sodium sulfate to form a free flowing powder. This is solvent-extracted three times using an ultrasonic horn.

2.4. Soxhlet Extraction: A measured weight of sample, typically 15g, is mixed with anhydrous sodium sulfate to form a free flowing powder. This is extracted with refluxing solvent for 16-24 hours.

2.5. Concentration: Procedures are presented for drying and concentrating the extracts to final volume for analysis.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

4.3. To prevent analyte loss, extracts which are not immediately concentrated by KD must be protected from light during storage. Extracts may be either refrigerated or stored in an opaque container.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) 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-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex and vinyl gloves provide no protection against most of the organic solvents used in this method. Nitrile gloves must be used. Latex gloves may be used for methanol.

5.1.2. The use of solvent dispensing pumps and solvent squeeze bottles are high risk activities and a face shield must be worn over safety glasses or goggles while using a dispensing pump.

5.1.3. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore, all samples must be opened, transferred, and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.4. When Soxhlet extractions are performed overnight or unattended, special precautions must be taken. Open the chiller valves to the system about 15 minutes before the heating elements are turned on and check every condenser to ensure that it is cold and functioning properly before turning the heating elements on. Check every condenser again about 15 minutes after turning the heating elements on to ensure that they are still cold and functioning properly. If the system is left operating overnight or unattended for an extended period, the first chemist to come back into the lab must again check every condenser to ensure that it is still cold and functioning properly.

5.1.5. Laboratory procedures such as microextraction of water samples in VOA vials, repetitive use of pipettes, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.1.6. The use of separatory funnels to extract aqueous samples with methylene chloride creates excessive pressure very rapidly. Venting should be done at a minimum of four rotations followed by eight rotations on the first extraction cycle and eight rotations on the second extraction cycle. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, and a face shield must be worn over safety glasses or goggles when it is performed. Alternately, the process may be performed behind a closed fume hood sash. After each of the three extraction cycles, ensure that the stoppers are removed from the top of the separatory funnels.

5.1.7. The use of ultrasonic sonicator systems to extract soil samples creates a hazard to hearing. Sonication must be performed inside of a fume hood, with all sashes on the hood closed. The operator must wear hearing protection while the sonicators are functioning.

5.1.8. Assembly and disassembly of glassware, including stopcocks and stoppers, creates a risk of breakage and cuts. All staff members shall wear Kevlar® or similar cut-resistant gloves over chemically resistant gloves when assembling and disassembling glassware.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	**Hazards**	**Exposure Limit (2)**	**Signs and symptoms of exposure**
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Sodium Hydroxide	Corrosive	2 mg/m^3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric Acid (1)	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Glassware for 8270 should be cleaned by acid wash. Glassware for 8270 Modified should be cleaned with soap and water, rinsed with water and dried in an oven at 400°C for at least 2 hours. Refer to SOP WS-OP-0011 for details of glassware cleaning. Kilned glassware requires rinsing with acetone, hexane, and DCM before use.

WARNING: Heat resistant gloves must be used when handling kilned glassware.

6.2. Equipment and supplies for extraction procedures

6.2.1. Separatory funnel, 2L (Teflon or Glass)

6.2.2. Separatory funnel, 500mL (Teflon or Glass)

6.2.3. Separatory funnel rotator with timer

6.2.4. Balance: >1400 g capacity, accurate ± 0.1 g

6.2.5. pH indicator paper, wide-range: covers extraction pH

6.2.6. Graduated cylinder: 1 liter. (Other sizes may be used)

6.2.7. Erlenmeyer flask, beaker, or jar: 125 & 300 mL (other sizes optional)

6.2.8. Solvent dispenser pump or 100 mL graduated cylinder

6.2.9. Round or flat bottom flask: 250, 500 mL or 1 L

6.2.10. Boiling chips: Contaminant free, approximately 10/40 mesh (Teflon® PTFE, carbide or equivalent).

6.2.11. Cooling condensers

6.2.12. Heating mantle: Rheostat controlled, with timer

6.2.13. Beakers: 250 & 400 mL, graduated

6.2.14. Balance: >100 g capacity, accurate to ± 0.01g

6.2.15. Soxhlet extractor

6.2.16. Sonicator (at least 300 watts), with sonicator horn, 3/4 inch. Preventive and routine maintenance is described in the "Schedule of Routine Maintenance" in the QAM, Preventative Maintenance (Section 20.2) and Schedule of Routine Maintenance (Table 20.2).

6.2.17. Kuderna-Danish (KD) apparatus: 500 mL, with 10mL concentrator tube, and 3-ball macro Snyder Column

6.2.18. Locally assembled solvent vapor condensing/recovery system for use with 6.2.17 above.

6.2.19. Water bath: Heated, with concentric ring cover, capable of temperature control (± 5°C) up to 95°C. The bath must be used in a hood or with a solvent recovery system.

6.2.20. Nitrogen blowdown apparatus

6.2.21. Culture tubes: 10 mL, 16 mm x 100 mm

6.2.22. Autosampler vials and caps

6.2.23. Gastight syringe 1 mL or 0.5 mL that is manufactured to a certified volume delivery tolerance of ± 0.01 mL

6.2.24. Glass wool

To ensure glass wool is free of contaminants, it is necessary to soxhlet rinse glass wool in methylene chloride for 18 hours.

6.2.25. Glass funnel: 75 X 75 mm

6.2.26. Disposable pipets

6.2.27. Aluminum foil

6.2.28. Paper towels

6.2.29. 60 mL VOA vial w/ teflon lined caps

6.2.30. Pressure rotor and pressure reactor

6.2.31. Extraction vessel and extraction vessel cover

6.2.32. Glass inserts

6.2.33. Microwave – Milestone Ethos EX

7. REAGENTS AND STANDARDS

7.1. Reagents for Extraction Procedures

All reagents must be ACS reagent grade or better unless otherwise specified.

7.1.1. Sodium hydroxide (NaOH) solution, 10 N, reagent grade

7.1.2. Granular sodium chloride, multi-compendial, manufacturer grade

7.1.3. Sulfuric acid (H_2SO_4), concentrated; reagent grade

7.1.4. Sulfuric acid (1:1): Carefully add 500 mL of H_2SO_4 to 500 mL of reagent water. Mix well.

WARNING: Always add concentrated acids or bases to water, never add water to the concentrated acid or base solution. Due to the reaction between acid and water, add the acid slowly to water to prevent breaking of the mixing vessel

7.1.5. Millipore grade water for 8270 and organic-free deionized reagent water for 8270 SIM and 8270 SIM 1,4-dioxane. Both Millipore and deionized water must be free of the analytes of interest as demonstrated through the analysis of method blanks, in accordance with SOP WS-QA-0014, “Monitoring of Reagent-Grade Laboratory Water.”

7.1.6. Sodium sulfate (Na_2SO_4), granular, anhydrous, reagent grade: Purify by heating at 400°C for a minimum of two hours.

7.1.7. Extraction/exchange solvents: Methylene chloride, pesticide quality or equivalent, and 1:1 methylene chloride:acetone. Ultra resi-analyzed grade.

7.1.8. Acetone – Ultra resi-analyzed grade: Used for cleaning

7.1.9. Nitrogen, reagent grade

7.1.10. N-hexane: used for rinsing.

7.2. Reagents for Cleanup Procedures

7.2.1. Oasis HLB Extraction Cartridge: 500 mg pre-packed in 6mL cartridges with polyethylene frits (Waters part # 186000115, or equivalent).

7.2.2. Activated Silica Gel: Activate for at least 1 hour at 190°C (± 5°C). Store at 130-200°C in a covered glass container.

7.3. Standards

7.3.1. Source Standards

Source standards are purchased as certified solutions or prepared from neat materials. Semivolatile source standards are stored in a refrigerator at ≤ 6ºC, unless otherwise specified by the manufacturer. All source standards must be protected from light.

7.3.1.1. Source standard solutions must be replaced after one year (from the time of preparation, if prepared in house, or from the time the ampule is opened if purchased), or the manufacturer's expiration date, whichever is sooner. Standards must be allowed to come to room temperature before use.

7.3.1.2. Expired standards must be rotated out of the lab to the Hazardous Waste storage area.

7.3.2. Surrogate Spiking Standards/Isotope Dilution Internal Standards

Prepare or purchase surrogate spiking standards as described in Tables 4 and 5. Surrogate spiking standards are prepared as dilutions of the source standards. Surrogate spiking solutions must be refrigerated and protected from light. The standards are given a one year expiration date from the time of preparation, or the expiration date of the stock solution, whichever is sooner. Standards may be replaced sooner if there is reason to believe that the standard has degraded or concentrated.

7.3.3. Matrix Spiking and Laboratory Control Spiking Standards.

The same spiking solution is used for the Matrix Spike (MS), Matrix Spike Duplicate (MSD), and the Laboratory Control Sample (LCS). Prepare MS/MSD/LCS spiking standards as described in Table 4. Spiking standards are purchased or prepared as dilutions of the source standards. Spiking solutions must be refrigerated and protected from light. The standards are given a one year expiration date from the time of preparation, or the expiration date of the stock solution, whichever is sooner. Standards may be replaced sooner if there is reason to believe that the standard has degraded or concentrated.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored at 0 - 6°C in glass containers with Teflon®-lined caps.

8.3. Holding Times

8.3.1. Extraction is initiated within 7 days of the sampling date for aqueous samples, 14 days for solid and waste samples.

8.3.2. For TCLP leachates, extraction is initiated within seven days from when the leaching procedure is initiated.

8.3.3. Analysis of the extracts is completed within forty days of extraction.

9. QUALITY CONTROL

9.1. Quality Control Batch

The batch is a set of up to 20 field samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a MB, an LCS, and a MS/MSD. (In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD). If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSDs. See policy WS-PQA-003 for further definition of the batch.

9.2. Insufficient Sample

If insufficient sample is available to process a MS/MSD, then a second LCS (LCSD) may be processed, if precision data is required by the client. The LCS pair is then evaluated according to the MS/MSD RPD criteria. Use of a LCS pair in place of a MS/MSD must be documented as a Non Conformance Memo (NCM) in TALS.

9.3. Method Blank (MB)

A MB consisting of all reagents added to the samples must be prepared and analyzed with each batch of samples. Surrogates are spiked into the method blank at the same level as the samples. The method blank is used to identify any background interference or contamination of the analytical system, which may lead to the reporting of elevated concentration levels or false positive data.

9.3.1. Aqueous MB use 1000mL (1L separatory funnel extraction) or 250mL (LVI/RVE) of organic free reagent water (deionized water) or Millipore water spiked with the surrogate. The MB goes through the entire analytical procedure, including any cleanup steps.

9.3.2. Solid MB use 30g (for 8270) or 10g (for PAH SIM) of kilned sodium sulfate for 3550B/C and 10g sodium sulfate (PAH SIM) for 3546 spiked with the surrogate. The MB goes through the entire analytical procedure, including any cleanup steps.

9.3.3. TCLP MB (LB) use 200 mL of leachate fluid spiked with the surrogate. The

MB goes through the entire analytical procedure, including any cleanup steps.

9.3.4. Waste MB use methylene chloride spiked with the surrogates. The MB goes through the entire analytical procedure, including any cleanup steps.

9.4. Laboratory Control Sample (LCS)

LCS is a well-characterized, laboratory generated samples used to monitor the laboratory's day to day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision. The LCS goes through the entire analytical procedure, including any cleanup steps.

9.4.1. The LCS is made up in the same way as the method blank (see Sections 9.3.1 - 9.3.4) but is spiked with the LCS standard and the surrogate.

9.5. Surrogates

9.5.1. Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples.

9.5.2. Each applicable sample, MB, LCS, and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A MS is an environmental sample to which known concentrations of target analytes have been added. A MSD is a second spiked aliquot of the same sample, which is prepared and analyzed along with the sample and MS. The MS and MSD are spiked with the LCS standard and the surrogate.

9.7. Initial Demonstration of Capability

The initial demonstration and method detection limit studies described in Section 13 must be acceptable before analysis of samples may begin.

9.8. Quality Assurance Summaries

Certain clients may require specific project or program QC that may supersede these method requirements. Quality Assurance Summaries (QAS) should be developed to address these requirements.

9.9. TestAmerica Sacramento QC Program

Further details of QC and corrective action guidelines are presented in the TestAmerica Sacramento QC Program document (WS-PQA-003). Refer to this document if in doubt regarding corrective actions.

10. CALIBRATION

10.1. On the day of use, measure either 0.5 mL or 1.0 mL of solvent into an autovial using a 1mL or 0.5mL gastight syringe that is calibrated in accordance with SOP WS-QA-0004. Seal the autovial – this is the 'check' or 'reference' vial. On the day of use, measure 10.00mL of solvent into a culture tube using a 5.00mL gastight syringe or sereological pipet that is calibrated in accordance with SOP-WS-QA-0004. Record the autovial or culture tube lot number, solvent, gastight syringe or sereological pipet ID, date, and initial in a controlled logbook. The autovials containing the sample extracts are then compared against the "check" vial to ensure that the final volume is consistently 1.0 ± 0.03 mL or 0.5 ± 0.015 mL depending on the analysis.

10.2. On a daily basis, calibrate any auto-pipettors to be used in accordance with SOP WS-QA-0004.

10.3. On the day of use, calibrate any balances to be used in accordance with SOP WS-QA-0041.

10.4. Prior to use, ensure any containers used for quantitative steps (e.g final volume) are calibrated in accordance with WS-QA-0004.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Separatory Funnel Liquid/Liquid Extraction of Water Samples

11.2.1. Remove surrogate and LCS spiking standards from refrigerator and allow standards to warm to room temperature.

11.2.2. If the sample bottle contains a layer of sediment greater than approximately ½"

in depth, contact the department manager and the project manager to determine how to proceed with the sample extraction. Options may include the following:

a) Decanting and extracting only the liquid fraction in a separatory funnel before adding the surrogate and/or spike solutions.
b) Adding the surrogate and/or spike solutions to the sample with the sediment still in the original sample container.
c) Processing the aqueous portion as described in a) along with creating a new sample id for the soil matrix. The sample bottle would then be rinsed with 60 mL of DCM, spiked with an appropriate volume of surrogate solution and shaken for 3 minutes. The addition of 60 mL of DCM will be repeated 2 more times and all solvents combined to create a second extract for the sample that is to be added to the extraction batch of water samples

NOTE: In the case of option "b", after adding the appropriate surrogates/spikes, the sample would be gently swirled for mixing purposes and then gently poured into the separatory funnel, thus minimizing the amount of sediment transferred to the extraction vessel during the process. The original sample container would then be rinsed with 60 mL of DCM. This DCM rinseate would then be poured into the extraction vessel.

11.2.3. Measure the initial sample pH with wide-range pH paper by dipping a disposable pipette into the sample and wetting the pH paper. Record on the extraction benchsheet. If sample is a leachate (e.g. TCLP), compare the current pH against the leachate log and note on the benchsheet if there is any discrepancy.

11.2.4. The normal sample volume is 1 liter for 1L separatory funnel extraction and 250mL for LVI/RVE. Other sample volumes may be used to obtain specific reporting limits, or accommodate dirty samples and reduced sample volumes, diluted to 1 liter or 250mL with organic free reagent water (deionized water).

11.2.5. Obtain sample weight(s) to ± 0.1 g. Assume a density of 1 g/mL and record the difference as the sample volume on the benchsheet to the nearest milliliter. This may be done in two ways depending if a dilution is necessary.

11.2.6. For samples not requiring a dilution, weigh the gross container on a tared balance and after extraction has been initiated, weigh the empty container on a tared balance and use the difference as the sample weight.

11.2.7. For samples requiring a dilution, subsample the desired aliquot amount in accordance with SOP-QA-0018 into a tared container. Dilute to 1L nominal for 1L separatory funnel extraction and 250mL nominal for LVI/RVE with organic free reagent water (deionized water).

11.2.7.1. For TCLP samples, use 200mL of leachate and dilute to 1L in organic free reagant water.

Note: *Alternative methods of measurement of sample volume include a) transferring the sample to a measuring cylinder and b) marking a meniscus on the sample bottle and then measuring the volume of water required to fill the bottle to the meniscus after the sample is transferred. The former method is not recommended because of the risk of cross contamination while the latter is not recommended because of poor accuracy. However, either method may be necessary for specific client programs.*

11.2.8. Prepare a MB, LCS and MS/MSD for each batch as specified in Section 9 of this SOP.

11.2.9. For a TCLP batch prepare a MB as specified in Section 9 of this SOP. In addition, prepare a TCLP method blank (LB) by measuring 200 mL of buffer solution used in the leaching procedure and dilute to 1L nominal using the method described in 11.2.4. No leachate LCS is required by this method.

11.2.10. After samples have warmed to room temperature add 50g granular sodium chloride, multi-compendial, to all of the 8270 samples that are to be extracted within a batch, including the method blank (MB), laboratory control sample (LCS), laboratory control sample duplicate (LCSD) if present, matrix spike (MS), and matrix spike duplicate (MSD). Shake the bottles containing the samples well in order to ensure that sodium chloride has been completely dissolved.

11.2.11. Add the surrogate and spike standard to the necessary samples as described in Section 9 and Table 11 of this SOP. Return spiking solutions to the refrigerator as soon as possible after spiking.

Note: If the sample bottle is completely full, it may be difficult to add the spike solutions to the bottle. In this case, transfer part of the sample to the separatory funnel and then add the spike to the bottle.

1L Separatory Funnel Extraction Spiking Volumes

Test	Surrogate Solution		Matrix Spike Solution	
	Volume	Name	Volume	Name
8270C	0.5mL	8270Surrogate	1.0mL	8270 Spike 1,2, and 3 (1mL each)
1,4-Dioxane	500µL	1,4 Dioxane Surrogate	500µL	1,4-Dioxane
PAH-SIM	0.5mL	PAH Surrogate	0.5mL	PAH Spike Mix
PAH-SIM ID	0.10mL	PAH-IS	0.05mL	PAH-ID N.S.

8270D SIM AK	1mL	PAH Surrogate	200uL (aqueous) 400uL (solids)	100uL LL 8270 SP1 & 100uL LL 8270 SP2 200uL 8270 SP1 & 200uL 8270 SP2

LVI/RVE Spiking Volumes				
Test	**Surrogate Solution**		**Matrix Spike Solution**	
	Volume	**Name**	**Volume**	**Name**
PAH-SIM	250uL	PAH Surrogate	250uL	PAH Spike Mix

Note: Contents of each solution are described in the Tables in Section 18.7

11.2.12. For regular 8270C samples, adjust sample pH to between 1 and 2. Use the minimum amount of 1:1 H_2SO_4 necessary. Recheck the sample with pH paper by dipping a disposable pipette into the sample and wetting the pH paper.

Note: *Samples for PAH-SIM and 1,4-Dioxane analysis are extracted at neutral pH (pH 5 – pH 9). These samples should not require pH adjustment. If samples do require pH adjustment, use the minimum amount of 1:1 H_2SO_4 or 10N NaOH as necessary.*

11.2.13. Record adjusted pH, spiking volumes, standard numbers, and reagent lots on the benchsheet.

11.2.14. Mix well, or if the sample contains sediment, decant before transfer. Transfer the sample to the separatory funnel. Rinse the sample bottle with 60mL (100 mL for PAH SIM) methylene chloride for 1L separatory funnel extraction and 30mL (50 mL for PAH SIM) for LVI/RVE and transfer to the 2L glass or Teflon separatory funnel for 1L separatory funnel extraction or 500mL glass or Teflon separatory funnel for 250 mL LVI/RVE.

11.2.15. Seal and shake or rotate the separatory funnel vigorously for 2 minutes with periodic venting to release excess pressure. As described in 5.1.6, venting should be done at a minimum of four rotations followed by eight rotations on the first extraction cycle and eight rotations on the second extraction cycle.

WARNING: Methylene chloride creates excessive pressure very rapidly! Therefore, initial venting should be done immediately after the separatory funnel has been sealed and inverted. Vent into hood away from analysts and other samples.

11.2.16. Allow the organic layer to separate from the water phase until complete visible separation has been achieved (at least 10 minutes). If the emulsion interface

between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. One ideal method is to swirl after resting for the required amount of time, wait approximately one minute, and then drain the extract without collecting the emulsion. On the third and final time, add a squirt of DCM to identify where the DCM and water are separated and collect only the DCM. Alternatively, on the final pour collect the emulsion and use a second filtration step at KD with glass wool and pipet off excess water.

***Note:** 15 – 20 mL of methylene chloride is expected to dissolve in 1 L of water. Thus, solvent recovery could be as low as 40 mL from the first shake and still be acceptable for 1L separatory funnel extraction. Subsequent shakes should recover at least 50 mL of solvent for 1L separatory funnel extractions. Apply the same ratios for LVI/RVE*

11.2.17. Fill a funnel with anhydrous kilned sodium sulfate (10-15g for LVI/RVE, nearly a full funnel for 1L separatory funnel extraction). The funnel can be plugged with glass wool to hold the sodium sulfate. Rinse the funnel with a small amount of methylene chloride, and discard the rinsate. Drain the solvent extract from the separatory funnel through the prepared filtration funnel into a clean glass container. The extract may be drained directly into the KD flask. Close the stopcock just before the water level begins draining out of the separatory funnel. If the sodium sulfate becomes saturated with water, add more to the funnel or replace the existing sodium sulfate with fresh drying agent.

11.2.18. Repeat the extraction process two more times using fresh portions of solvent – 60 mL (100 mL for PAH SIM) for 1L separatory funnel extraction and 30 mL (50 mL for PAH SIM) for LVI/RVE, combining the three solvent extracts in the collection container. Ensure that the amount of water collected is minimal. If extraction at a secondary pH is not required, proceed to Section 11.2.20.

11.2.19. If extraction at a secondary pH is required, adjust the pH of the sample in the separatory funnel to between 11 and 12 with a minimum amount of 10 N NaOH. Measure with pH paper and record the adjusted pH on the benchsheet. Serially extract with three 60 mL portions of methylene chloride for 1L separatory funnel extraction and three 30 mL portions of methylene chloride for LVI/RVE, as outlined in Sections 11.2.15 to 11.2.18. Collect these three extracts in a separate bottle. Once again, minimize the amount of water collected.

11.2.20. Dispose the solvent-contaminated water remaining in the extractor into the LLE waste drum. Waste methylene chloride goes to the methylene chloride waste drum for disposal.

11.2.21. If the extract is not concentrated immediately, refrigerate it to protect from light. Alternatively, the extract bottle may be wrapped with foil to protect from light for short periods of time (i.e., overnight). Refer to Section 11.6 for concentration.

11.3. Method 3546, Solid Extraction by Microwave (PAH SIM only)

11.3.1. Remove surrogate and matrix spiking solutions from refrigerator and allow standards to warm to room temperature.

11.3.2. Homogenize the sample by mixing thoroughly. (See SOP WS-QA-0018). Discard any foreign objects such as sticks, leaves and rocks, unless the client requires extraction of this material. If the sample consists primarily of foreign materials consult with the client (via the Project Manager or Administrator).

11.3.3. Weigh 10g of sample (10g for biologics) ± 1.0 g into an extraction vessel. Record the weight to the nearest 0.01 g in the appropriate column on the benchsheet.

11.3.3.1. For visually dirty samples, it may be necessary to weigh the sample into a glass insert to be placed into an extraction vessel. Contact your supervisor to determine if this is the case.

11.3.4. Mix weighed sample with a spatula. If clay clumps or other sample aggregation is evident, add sodium sulfate (not to exceed 5 g) as needed to facilitation disaggregation and mix as necessary.

11.3.5. Prepare a MB, LCS and MS/MSD for each batch as specified in Section 9 of this SOP.

11.3.6. Add the surrogate and spike standard to the necessary samples as described in Section 9 and Table 7 of this SOP. Return spiking solutions to the refrigerator as soon as possible after spiking.

11.3.7. Immediately add 25mL of DCM to extraction vessels. Note: Steps 11.3.6 - 11.3.7 should be performed rapidly to avoid loss of the more volatile extractables.

11.3.8. Add flared extraction vessel covers to each extraction vessel containing sample and QC aliquots.

11.3.8.1. It is important that each cover fits snugly to ensure a proper seal. The cover should not slide easily or loosely inside the extraction vessel, but should require some finger pressure to insert firmly. A cover flaring tool should be used.

11.3.8.2. For the visually wettest sample, add the thermowell liner into the extraction vessel cover to create the representative sample that the ATC temperature sensor can be inserted into.

11.3.9. Place each extraction vessel into a pressure reactor. Screw on the pressure cap/safety lid. The pressure cap should be hand tightened until the sealing valve is flush with the top of the cap.

11.3.9.1. For the representative sample created in 11.3.8.2, add the protection foil and appropriate safety lid.

11.3.10. Place all the extraction vessels into the rotor so that the pressure-release valves are facing outside of the rotor on the outside ring and inside toward the center on the inside ring.

11.3.10.1. Place the rotor in the microwave oven and insert the ATC temperature sensor into the representative sample (11.3.8.2 and 11.3.9.1).

11.3.11. Close the microwave oven and start the appropriate extraction profile.

11.3.11.1. A 10 minute ramp from 25C to 100 C, hold for 25 minutes at 100C, followed by a 15 minute cooldown to 25C

11.3.12. After the extraction period and cooldown, pressure reactors should be at ambient temperature prior to removal from the rotor and opening.

11.3.13. Proceed to Section 11.6, Concentration

11.3.13.1. If the samples are going to be stored in the extraction vessels (at 0 - 6°C), the cap needs to be retightened after cooldown.

11.3.13.2. Discard any glass inserts. To clean extraction vessels and pressure cap/safety lid, use soap and water, allow to air dry, then follow with a DCM solvent rinse. Use only solvent to rinse the probe. Allow to air dry prior to use.

11.4. Method 3550B and 3550C, Sonication

11.4.1. Remove surrogate and matrix spiking solutions from refrigerator and allow them to warm to room, temperature.

11.4.2. Homogenize the sample by mixing thoroughly. (See SOP WS-QA-0018). Discard any foreign objects such as sticks, leaves and rocks, unless the client requires extraction of this material. If the sample consists primarily of foreign materials consult with the client (via the Project Manager or Administrator).

11.4.3. Weigh 30g (10g for PAH-SIM) of sample ± 1.0 g into a 250 mL beaker, 400 mL beaker or glass jar of sufficient size that the sodium sulfate and extraction solvents can be added. Record the weight to the nearest 0.1 g in the appropriate column on the benchsheet or in TALS. Use 30 g of sodium sulfate for the method blank and LCS.

11.4.4. Mix weighed sample with a spatula adding enough anhydrous sodium sulfate (approximately 30 g) to be free flowing. (If the sample is not free flowing extraction efficiency may be reduced)

11.4.5. Prepare a method blank, LCS and MS/MSD for each batch as specified in Section 9 of this SOP.

11.4.6. Add the surrogate and spike standard to the necessary samples as described in Section 9 and Table 7 of this SOP. Return spiking solutions to the refrigerator as soon as possible after spiking.

11.4.7. Immediately add 60 mL (or up to100 mL if required due to the nature of the sample matrix) of 1:1 methylene chloride:acetone for 8270C and methylene chloride only for PAH-SIM to the beaker.

Note: *Steps 11.4.5 - 11.4.7 should be performed rapidly to avoid loss of the more volatile extractables.*

11.4.8. Make sure that all sonicator horns have been cleaned prior to sonicating. To achieve a clean sonicator horn, immerse each sonicator horn in a tri-pour beaker of de-ionized (DI) water for 2-3 pulses. Immediately rinse the horns with acetone to remove any excess water. Finish by rinsing each horn with dichloromethane (DCM). Sonicators are ready for use.

11.4.9. Place the bottom surface of the appropriate disrupter horn tip approximately ½ inch below the surface of the solvent, but above the sediment layer.

11.4.10. Sonicate for 3 minutes, making sure the entire sample is agitated. If the W-380 or W-385 sonicator is used the output should be set at 6 for the 3/4 inch high gain (Q) horn or 10 for the 3/4 inch standard horn with mode switch on pulse, and percent-duty cycle knob set at 50%.

Note: *Do not use Microtip probe.*

WARNING: Hearing protection is required when sonicating samples.

11.4.11. Decant into a clean beaker or clear tall round jar.

11.4.12. Repeat the extraction two more times with additional 60-100 mL minimum portions of solvent each time. Decant and retain the extraction solvent after each sonication. On the final sonication pour the entire sample (sediment and

solvent) into the jar and rinse with an additional 10 mL-20 mL of the methylene chloride/acetone.

11.4.13. If the extract is not concentrated immediately, refrigerate it to protect from light. Alternatively, the extract bottle may be wrapped with foil to protect from light for short periods of time (i.e., overnight). Refer to Section 11.6 for concentration.

11.4.14. Sonicator Tuning (see SOP WS-OP-0016, Sonicator Tuning).

11.4.14.1. Tune the sonicator according to manufacturer's instructions. The sonicator must be tuned at least every time a new horn is installed.

11.5. Waste Dilution

11.5.1. This method is used for materials that are soluble in an organic solvent.

11.5.2. Remove surrogate and matrix spiking solutions (see Table 7) from refrigerator and allow to warm to room temperature

11.5.3. Tare the vial, then transfer approximately 1g of sample to the vial. Record the weight to the nearest 0.01 g.

11.5.4. Prepare a method blank, LCS and MS/MSD for each batch as specified in Section 9 of this SOP.

11.5.5. Add the surrogate and spike standard to the necessary samples as described in section 9 and Table(s) 6 and 7 of this SOP. Return spiking solutions to the refrigerator as soon as possible after spiking. Note, it may not be necessary to spike all analytes and comparing the analyst list in TALS to the analytes in each standard may be beneficial.

11.5.6. Compare the volume to that of the calibrated reference container, and carefully add methylene chloride to make the final volume of 10 mL. Ensure to record the lot of the container that extracts are brought to final volume in.

11.5.7. If the sample is suspected to contain water, add approximately 2g of sodium sulfate to the samples. Cap and shake for 2 minutes.

11.5.8. Aliquot 1.0 mL into a calibrated autosampler vials using a reference container. Ensure to record the lot of the autosampler vial. Store the vial box in the refrigerator at 0-6°C in the SVOA instrument lab until ready for GC/MS.

11.5.8.1. The excess in the culture tube is stored in the refrigerator at 0 - 6°C for a minimum of 30 days (can be used for additional re-analysis as needed) and disposed in accordance with Section 15.

11.6. Concentration

According to the type of analysis and any cleanup procedures needed, different final volumes may be required. Below are the "normal" final volumes for tests covered by this SOP:

Test	Aqueous	Solid
8270C	1.0 mL	1.0 mL
1,4-Dioxane	1.0 mL	NA
PAH-SIM (all except LVI/RVE)	1.0 mL	1.0 mL
PAH-SIM LVI/RVE	0.5mL	NA
8270D SIM AK	1.0 mL	1.0 mL

11.6.1. Kuderna-Danish (KD) Method:

11.6.1.1. For aqueous samples, loosely plug the stem of a 75mm x 75mm glass funnel with glass wool and rinse well with DCM. For solid samples place a folded piece of Whatman #1 filter paper into the glass funnel.

11.6.1.2. For LVI/RVE samples, add approximately 10-15 g of anhydrous sodium sulfate to the funnel cup. For 1L separatory funnel extraction, add anhydrous sodium sulfate to approximately half way up the funnel. For solid samples, add approximately 5-10 g of anhydrous sodium sulfate to the funnel cup.

11.6.1.3. Pour the sample through the prepared glass funnel into the KD flask. For 8270 Aqueous, add the base portion through one funnel, then filter the acid portion through a new funnel. Add one or two clean boiling chips to the KD flask.

11.6.1.4. Rinse the top of the funnel as well as the sample container with DCM.

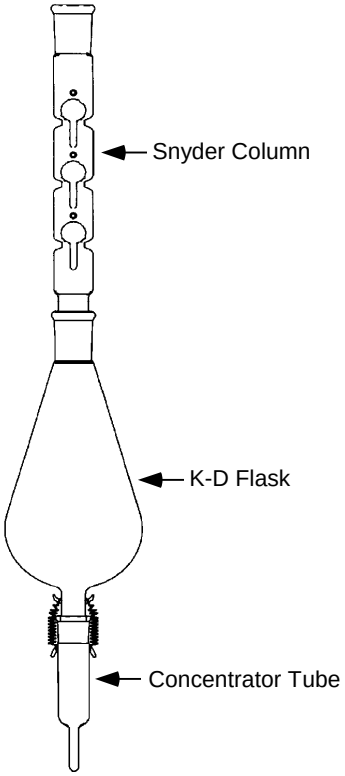
11.6.1.5. Prepare the three ball Snyder columns by rinsing them with DCM to ensure that the balls are not stuck and that the column will work properly.

WARNING: Use of cut-resistant gloves (Kevlar® or similar material) is required when assembling or disassembling glassware.

11.6.1.6. Assemble a Kuderna-Danish concentrator as shown in Figure 1 by attaching a 10 mL concentrator tube to the 500 mL KD flask. Transfer the sample to the KD flask. Add one or two clean boiling chips and the extract to be concentrated to the KD flask and attach a three-ball Snyder column. Add approximately 1 mL of clean methylene chloride to the top of the Snyder column (this is

important to ensure that the balls are not stuck and that the column will work properly).

Figure 1 KD Setup



11.6.1.7. Place the KD apparatus on a water bath (70-75ºC for all except 8270 solid at 80-85ºC) so that the tip of the concentrator tube is submerged. The water level should not reach the joint between the concentrator and the KD flask. At the proper rate of distillation, the balls will actively chatter but the chambers should not flood. Attach the solvent vapor recovery device to the top of the Snyder column.

11.6.1.8. Concentrate to 5-15 mL. The Snyder column may be insulated if necessary to maintain the proper rate of distillation. For 8270 Aq, add a squirt of DCM to the top of the Snyder column at about 30 mL.

Note: *It is very important not to concentrate to dryness as analytes will be lost.*

WARNING: Do not concentrate to dryness as an unsafe condition may be created.

11.6.1.9. Remove the KD apparatus from the water bath and allow it to cool and drain. For 8270 aqueous, rinse the inside of the joint with a

minimum amount of DCM rotating the flask as necessary to ensure the rinsate covers the sides of the KD flask. For 8270 aqueous, repeat a second time. If the level of the extract is above the level of the concentrator tube joint, add new boiling chips and continue to distill the solvent as necessary. Once the KD concentration apparatus has been removed from the bath, place the assembly on the rack and let it cool to room temperature. Rinse the joint at which the KD flask is attached to the concentrator tube with acetone in order to remove any water stuck to the joint.

11.6.2. Nitrogen Evaporation to Final Concentration

Note: If the extracts are proceeding to clean-up, skip to Section 11.6.3 (Nitrogen Evaporation to Volume for Clean-up).

11.6.2.1. Evaporate the solvent using a gentle stream of nitrogen. The nitrogen flow will form a slight depression on the surface of the solvent, but should not create splattering of the extract.

11.6.2.2. During the course of the evaporation rinse the sides of the evaporation tube twice with approximately 1 mL of clean solvent. The first rinse should be about half way through the process, with the second rinse when the solvent volume gets close to 1 mL. Concentrate the solvent to below 1mL (below 0.5 mL for PAH-SIM LVI analysis) and quantitatively transfer the extract to the storage vial.

11.6.2.2.1. For 8270 Aq., dunk the concentrator tube in water, heated to a maximum of 3.5 on the N-evap. Remove when the sovlent level is at about 700uL on the concentrator tube (~500uL of solvent).

11.6.2.3. Quantitatively transfer the extract to calibrated autosampler vials. Compare the volume to that of a calibrated reference, and carefully add solvent to raise the level to the proper amount. Ensure to record the lot of the container that extracts are brought to final volume in. The extract is now ready for instrumental analysis.

Note: *It is very important not to concentrate to dryness as analytes will be lost.*

Note: *The final concentration and volume measurement steps are critical. Use care when concentrating and make certain that the final volume measurement is accurate.*

11.6.3. Nitrogen Evaporation to Volume for Clean-up

Note: If the extracts are not proceeding to clean-up, go to the previous section (Nitrogen Evaporation to Final Concentration).

11.6.3.1. Transfer the entire extract to an evaporation tube. Rinse the concentrator tube with 1-2 mL of the appropriate solvent and transfer the solvent rinsate to the evaporation tube.

11.6.3.2. Evaporate the solvent using a gentle stream of nitrogen. The nitrogen flow will form a slight depression on the surface of the solvent, but should not create splattering of the extract.

11.6.3.3. During the course of the evaporation rinse the sides of the evaporation tube twice with approximately 1mL of clean solvent. The first rinse should be about half way through the process, with the second rinse when the solvent volume gets close to 1 mL. Concentrate the solvent to 1 – 1.2 mL.

Note: *It is very important not to concentrate to dryness as analytes will be lost.*

Note: *As the entire sample will go through the clean-up column and be adjusted to an exact volume afterward, the extract volume at this step is not critical.*

11.6.3.4. Proceed to the appropriate clean-up option in the following sections.

11.6.4. Nitrogen evaporation for final volume (Microconcentration)

11.6.4.1. Aliquot 5 mL and transfer to 8 mL screw cap test tube.

11.6.4.2. Evaporate the solvent using a gentle stream of nitrogen. The nitrogen flow will form a slight depression on the surface of the solvent, but should not create splattering of the extract.

11.6.4.3. During the course of the evaporation rinse the sides of the evaporation tube twice with approximately 1 mL of clean solvent. The first rinse should be about half way through the process, with the second rinse when the solvent volume gets close to 1 mL. Concentrate the solvent to 1.0 mL.

Note: *It is very important not to concentrate to dryness as analytes will be lost.*

Note*: As the entire sample will go through the clean-up column and be adjusted to an exact volume afterward, the extract volume at this step is not critical.*

11.7. Clean-up of 8270 extracts with HLB column cartridges

This pre-packed column may be used to trap co-extracted matrix contamination and yield cleaner 8270 extracts. This clean-up is recommended for 8270 extracts from soil and heavily contaminated water.

Refer to Section 11.9 for the HLB lot verification procedure.

Note: *Systems for eluting multiple cleanup cartridges include the Supelco, Inc. Solid Phase Extraction (SPE) assembly, Zymark Benchmate, or equivalent.*

11.7.1. Attach a vacuum manifold to a vacuum pump or water aspirator with a trap installed between the manifold and the vacuum. Adjust the vacuum in the manifold to 5-10 psi.

11.7.2. Place a valve liner (Supelco, P/N# 57059, disposable flow control valve liner, or equivalent) into each port of the vacuum manifold.

WARNING: Use of vacuum systems creates a significant risk of implosion. Thoroughly inspect all glassware and do not use any that has been chipped, rubbed, cracked, or marred in any fashion.

WARNING: Ensure that the exhaust line from the vacuum pump is secured well inside of a fume hood so that it cannot fall out of the hood.

11.7.3. Place one HLB cartridge into the vacuum manifold for each sample extracts.

11.7.4. Prior to cleanup of samples, pre-elute each cartridge with 5 mL of DCM, then 5 mL of MeOH.

11.7.5. During this step, adjust the vacuum applied to each cartridge so that the flow through each cartridge is approximately 2 mL/min.

11.7.6. Add 2 mL of MeOH, drain through the column but stop the flow when the MeOH reaches the top of the cartridge. Do not let the cartridge go dry.

11.7.7. Place a rack of clean, labeled 16 mL test tubes into the manifold and replace the manifold top. Make sure that the solvent line from each cartridge is placed inside the appropriate tube.

11.7.8. After the clean tubes are in place, vacuum to the manifold is restored, add the extract to the appropriate cartridge. This is the entire sample.

11.7.9. The extract concentrates are then eluted through the column with 10 mL of acetone and are collected into the 16 mL test tube held in the rack inside the vacuum manifold.

11.7.10. Use nitrogen evaporation (Section 11.6.2) to concentrate the extract to approximately 1 mL. Add 5 mL DCM and re-concentrate to 1.0 mL (for a thorough removal of acetone).

11.7.11. Once returned to 1.0 mL, the sample is ready for instrumental analysis.

Note: *It is very important not to concentrate to dryness as analytes will be lost.*

Note: *The final concentration and volume measurement steps are critical. Use care when concentrating and make certain that the final volume measurement is accurate.*

11.8. Clean-up of 8270SIM-PAH Extracts

11.8.1. Use clean 20mm glass columns packed with a plug of DCM rinsed glass wool.

11.8.2. Rinse the glass wool and column with Hexane

11.8.3. Add 4cm of activated silica gel (~10g) DCM rinsed, then a 2cm layer of kilned sodium sulfate. Tap the column while adding these reagents to prevent channeling. Elute and discard 40 mL Hexane.

11.8.4. Add the sample extract to the top of the column by quantitative transfer with 2 rinses. (The sample volume can be between 1 and 5 mL).

11.8.5. Immediately after adding the sample, elute the column with 25 mL of hexane.

11.8.6. Just before the last of the hexane hits the sodium sulfate layer, add 25 mL of hexane:methylene chloride (2:3)

11.8.7. Collect the entire elute mix in a 250mL flask.

11.8.8. Following the Kuderna Danish Concentration section in this SOP, concentrate the extract to low volume (approximately 5 mL) and complete the solvent re-exchange by adding 50 mL of DCM and concentration to approx 5 mL.

11.8.9. Proceed to the Nitrogen Evaporation to Final Concentration (Section 11.6.2).

11.9. HLB Column Verification

Each lot of HLB cartridges must be evaluated before use, following this performance check procedure:

11.9.1. Obtain 1.0 mL of a 50 ug/mL mid-level solution of 8270 compounds in DCM (prepared from the GCMS calibration stock). This can be obtained from the GCMS group.

11.9.2. In a clean test tube add 1.0 mL of this HLB check solution (8270 standard).

11.9.3. Add this to a pre-washed HLB cartridge.

11.9.4. Process this per the instructions in the Clean-up of 8270 Extracts section (Section 11.7). For a blank sample, repeat this step on another cartridge loaded with 2 mL of clean DCM.

11.9.5. Deliver 1.0 mL of the final cleaned HLB check and blank to the GCMS

instrument group for analysis.

11.9.6. Analyze the HLB check solution and blank, per method 8270. Expected recoveries are 70-120% of all analytes and surrogates with the exception of the ones listed below, which cannot typically meet this criterion. At a minimum, the recoveries will meet the current LCS limits for soils. Additionally, there should be no indication of peaks interfering with target compounds.

11.9.7. Compounds with Low Recoveries Expected in HLB Checks

- Pyridine
- Aniline
- Benzoic Acid
- 4-Chloraniline
- 2,4-Dinitrophenol
- 4,6-Dinitro-2-methylphenol
- 3, 3'-Dichlorobenzidine

12. CALCULATIONS/DATA REDUCTION

12.1. Not applicable.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of

the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. 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 Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. 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 SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Extracted aqueous/TCLP leachate samples contaminated with methylene chloride are collected at the fume hood in a 5-gallon or smaller carboy. If the samples are not at a neutral pH, add small quantities of sodium bicarbonate to bring the waste to neutral. Stir well. Once neutralized, immediately pour the carboy contents into a blue plastic LLE drum in the H3 closet. When full to between one and four inches of the top, or after no more than 75 days, move the LLE drum to the waste collection area for shipment.

15.2. Extracted soil samples and thimbles, sodium sulfate, glass wool/filter paper contaminated with methylene chloride and acetone from sonication and soxhlet extraction. Pour any excess liquid from the extracted soil samples as outlined below (Section 15.5). Dump the extracted soil and thimbles into an orange contaminated lab trash bucket. When the bucket is full or at the end of the day, tie the plastic bag liner shut and put the lab trash into the appropriate collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.3. Used assorted disposable glassware and materials contaminated with methylene chloride. Dump the solid waste into a yellow contaminated lab trash bucket. When the bucket is full or at the end of the day, tie the plastic bag liner shut and put the lab trash

into the appropriate steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.4. Waste Streams Produced by the Method - Waste methylene chloride generated during glassware and sodium sulfate cleaning and various rinses. Keep waste methylene chloride separated from waste flammable solvents. Collect the waste methylene chloride in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel methylene chloride waste drum in the H3 closet. When the drum is full to six inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment.

15.5. Mixed flammable solvent waste generated during soil extraction, glassware and sodium sulfate cleaning. Collect the waste solvents in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the mixed flammable solvent steel drum in the H3 closet. When full to between two and six inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment.

16. REFERENCES/CROSS REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III

Method 3500B, Organic Extraction and Sample Preparation, Revision 2, December 1996

Method 3510C, Separatory Funnel Liquid Liquid Extraction, Revision 3, December 1996

Method 3520C, Continuous Liquid-Liquid Extraction, Revision 3, December 1996

Method 3540C, Soxhlet Extraction, Revision 3, December 1996

Method 3550B, Ultrasonic Extraction, Revision 2, December 1996

Method 3580A, Waste Dilution, Revision 1, July 1992

Method 3600C, Cleanup, Revision 3, December 1996

Method 3511, Organic Compounds in Water by Microextraction, Revision 0, November 2002

16.2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update IV

Method 3500C, Organic Extraction and Sample Preparation, Revision 3, February 2007

Method 3546, Microwave Extraction, Revision 0, February 2007

Method 3550C, Ultrasonic Extraction, Revision 3, February 2007

16.3. EPA Memorandum (Dated August 5, 2010) Regarding "Spiking (Prior To vs. After Sample Drying) Issue in SW-846 Organic Extraction Methods"

17. METHOD MODIFICATIONS

17.1. Some surrogate spiking concentrations are modified from those recommended in SW-846, in order to make the concentrations more consistent with the calibration levels in the determinative methods.

17.2. Aqueous sample volumes may be determined by weight.

17.3. Spiking for Method 3550C is performed after mixing with drying agents, as per the EPA surrogate spiking memo (Section 16.3, above).

17.4. Addition of sodium chloride during 8270 aqueous extractions was added to this method to improve benzoic acid recoveries. Method 3510 in SW-846 does not include the use of sodium chloride.

18. ATTACHMENTS

18.1. Table 1 – 8270 List 1 Standard 1

18.2. Table 2 – Additional Stock Solutions

18.3. Table 3 – PAH-SIM Stock Standard

18.4. Table 4 – Standard Reagent Mixtures

18.5. Table 5 – PAH-SIM Isotope Dilution Analyte Solution

18.6. Table 6 – TestAmerica Sacramento 8270C Extraction Summary

18.7. Table 7 – Spiking table

18.8. Table 8 – LLE/Sonication Volume Table

19. REVISION HISTORY

19.1. WS-OP-0001, Revision 5.1, Effective 07/27/2017

19.1.1. Revised Section 6.1 to read, "Glassware for 8270 should be cleaned by acid wash. Glassware for 8270 Modified should be cleaned with soap and water, rinsed with water and dried in an oven at 400°C for at least 2 hours. Refer to SOP WS-OP-0011 for details of glassware cleaning. Kilned glassware requires rinsing with acetone, hexane, and DCM before use."

19.1.2. Section 11.2.6, changed “tared container” to “empty container”.

19.1.3. Section 11.2.11, revised names and spiking volumes in table.

19.1.4. Removed Section 11.4.11, “Place the prepared funnel on a collection apparatus (beaker or KD).”

19.1.5. Section 11.4.11 (previously 11.4.12), added “or clear tall round jar.”

19.1.6. Section 11.6.1.12, revised “For 1L separatory funnel extraction, add anhydrous sodium to nearly fill up the funnel cup” to “For 1L separatory funnel extraction, add anhydrous sodium sulfate to approximately half way up the funnel.”

19.1.7. Added Section 17.4, “Addition of sodium chloride during 8270 aqueous extractions was added to this method to improve benzoic acid recoveries. Method 3510 in SW-846 does not include the use of sodium chloride.”

19.1.8. Table 6, adjusted surrogate and spike volumes for PAH LVI and 8270 SIM AK.

19.1.9. Table 6, added “50g NaCl” to 8270C (AQ, 1LS) and removed 8270C (AQ, LVI).

19.1.10. Table 7, adjusted surrogate and spike volumes for 1,4-Dioxane, PAH SIM LVI, Waste, and 8270 SIM AK.

19.1.11. Editorial changes.

19.2. WS-OP-0001, Revision 5.0, Effective 05/17/2017

19.2.1. Changed the Methods reference in the title to “Methods 3510C, 3550B/C, 3546, and 3580.”

19.2.2. Section 2.2, changed 15g to 10g and removed “(10g for biologics)”.

19.2.3. Removed Section 5.1.9, “The use of vacuum systems during rotovap concentration presents the risk of imploding glassware. All glassware used during vacuum operations must be thoroughly inspected prior to each use. Glass that is chipped, scratched, cracked, rubbed or marred in any manner must not be used under vacuum. It must be removed from service and replaced.” Rotovap not used in this procedure.

19.2.4. Section 5.2, added hexane to the table.

19.2.5. Section 6.2.7, removed “French Square” before “jar”. Switching to non-square jars.

19.2.6. Removed Section 6.2.9, “Continuous liquid/liquid extractor.”

19.2.7. Added Section 7.1.2, “Granular sodium chloride, multi-compendial, manufacturer grade.”

19.2.8. Section 7.1.5, added “for 8270 and organic-free deionized reagent water for 8270 SIM and 8270 SIM 1,4-dioxane.”

19.2.9. Sections 7.1.7 and 7.1.8, added “Ultra resi-analyzed grade.”

19.2.10. Section 7.1.10, added “N-hexane: Used for rinsing.”

19.2.11. Section 8.2, changed “4 ± 2°C” to “0 - 6°C.”

19.2.12. Section 9.2, added “(LCSD)” and added “in TALS.”

19.2.13. Section 9.3.2, added “(for 8270) or 10g (for PAH SIM)”.

19.2.14. Section 9.3.2, replaced “15g ottowa sand” with “10g sodium sulfate (PAH SIM).”

19.2.15. Section 11.2.11, added the correct spike and spiking volumes for 8270_SIM_1,4 dioxane.

19.2.16. Added Section 11.2.10, “After samples have warmed to room temperature add 50g granular sodium chloride, multi-compendial, to all of the 8270 samples that are to be extracted within a batch, including the method blank (MB), laboratory control sample (LCS), laboratory control sample duplicate (LCSD) if present, matrix spike (MS), and matrix spike duplicate (MSD). Shake the bottles containing the samples well in order to ensure that sodium chloride has been completely dissolved.”

19.2.17. Section 11.2.11, removed 8270C test from LVI/RVE Spiking Volumes table because 8270C is not currently done by LVI.

19.2.18. Section 11.2.11, added “8270D SIM AK” to table.

19.2.19. Section 11.2.14, edited to read “Mix well, or if the sample contains sediment, decant before transfer. Transfer the sample to the separatory funnel. Rinse the sample bottle with 60mL (100 mL for PAH SIM) methylene chloride for 1L separatory funnel extraction and 30mL (50 mL for PAH SIM) for LVI/RVE and transfer to the 2L glass or Teflon separatory funnel for 1L separatory funnel extraction or 500mL glass or Teflon separatory funnel for 250 mL LVI/RVE.”

19.2.20. Added Section 11.3.13.1, “If the samples are going to be stored in the extraction vessels (at 0 - 6°C), the cap needs to be retightened after cooldown.”

19.2.21. Removed Section 11.4.11, “Loosely plug the stem of a 75 mm x 75 mm glass funnel with glass wool and/or line the funnel with filter paper. Add 10-20 g of anhydrous sodium sulfate to the funnel cup.” This is done at KD.

19.2.22. Removed Section 11.4.12, “Place the prepared funnel on a collection apparatus (beaker or KD).” This is done at KD.

19.2.23. Revised Section 11.4.13 to read, “Decant into a clean beaker.”

19.2.24. Section 11.5.7, added “If the sample is suspected to contain water.”

19.2.25. Section 11.6, added “8270D SIM AK” to table.

19.2.26. Section 11.6.1.3, added “add the base portion through one funnel, then filter the acid portion through a new funnel.”

19.2.27. Section 11.6.1.9, added “Once the KD concentration apparatus has been removed from the bath, place the assembly on the rack and let it cool to room temperature. Rinse the joint at which the KD flask is attached to the concentrator tube with acetone in order to remove any water stuck to the joint.”

19.2.28. Added Section 15.4, “Waste Streams Produced by the Method - Waste methylene chloride generated during glassware and sodium sulfate cleaning and various rinses. Keep waste methylene chloride separated from waste flammable solvents. Collect the waste methylene chloride in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel methylene chloride waste drum in the H3 closet. When the drum is full to six inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment.”

19.2.29. Updated Table 2 in Attachments.

19.2.30. Updated Table 6 in Attachments. Added 8270D SIM for Aqueous 1LS and Solid Sonication.

19.2.31. Updated Table 7 in Attachments. Added 8270D SIM for 1LS and 3550B/C.

19.3. WS-OP-0001, Revision 4.3, Effective 11/04/2016

19.3.1. Revised Section 11.4.7 to “Immediately add 60 mL (or up to 100 mL if required due to the nature of the sample matrix) of 1:1 methylene chloride:acetone.”

19.3.2. Revised Table 6 as part of the reduced solvent initiative:

(Solid, SONC) 3 x 60 mL	8270C
(Solid, SONC) 3 x 60 mL	PAH
(Solid, SONC) 3 x 60 mL	PAHHD

19.3.3. Editorial changes

19.4. WS-OP-0001, Revision 4.2, Effective 02/12/2016

19.4.1. Updated copyright statement on cover page

19.4.2. Section 11.8.3 – added DCM rinsed to 4 cm of activated silica gel and kilned sodium sulfate

19.4.3. Editorial changes.

19.5. WS-OP-0001, Revision 4.1, Effective 08/22/2014

19.5.1. Rearranged the Attachments to reflect the “Standard Standards” spiking mixtures and how they are implemented in the facility. Tables 1-6 (formerly the composition of the spike mixes) have been replaced by Tables 1-5. References in the text have been adjusted.

19.5.2. Removed Section 11.4 (Microextraction for Waters) as this process is no longer performed. Renumbered accordingly

19.5.3. Removed Section 11.6 (Method 3550C) and incorporated with Section 11.5. Renumbered accordingly.

19.5.4. Removed Section 11.7 (Soxhlet Extraction) as this process hasn’t been performed for some time. Renumbered accordingly.

19.5.5. Editorial changes.

19.6. WS-OP-0011, Revision 4.0, Effective 08/15/2013

19.6.1. Inserted Section 2.1.1, describing the two types of separatory funnel extraction.

19.6.2. Inserted Section 2.3 describing microwave extraction.

19.6.3. Added references for methods 3500C and 3550C (Update IV) as well as method 3546.

19.6.4. Removed sections regarding continuous liquid-liquid extraction.

19.6.5. Section 7.2, changed storage conditions of source standards to reflect manufacturer’s recommended conditions.

19.7. WS-OP-0001, Revision 3.7, Effective 11/07/2012

19.7.1. Section 11.8: Separated PAH SIM and PAH SIM-ID in table.

19.7.2. Editorial changes.

19.8. WS-OP-0001, Revision 3.6, Effective 04/20/2012

19.8.1. Inserted Section 11.4 – Microextraction for water

19.8.2. Editorial changes

Table 1			
Stock Standard - 8270 List 1 Standard 1			
Analyte Name	**Concentration µg/mL**	**Analyte Name**	**Concentration µg/mL**
1,1'-Biphenyl	1000	1,2,4,5-Tetrachlorobenzene	1000
1,2,4-Trichlorobenzene	1000	1,2-Dichlorobenzene	1000
1,3-Dichlorobenzene	1000	1,3-Dinitrobenzene	1000
1,4-Dichlorobenzene	1000	1,4-Dioxane	1000
1-Methylnaphthalene	1000	2,2'-oxybis[1-chloropropane]	1000
2,3,4,6-Tetrachlorophenol	1000	2,4,5-Trichlorophenol	1000
2,4,6-Trichlorophenol	1000	2,4-Dichlorophenol	1000
2,4-Dimethylphenol	1000	2,4-Dinitrophenol	2000
2,4-Dinitrotoluene	1000	2,6-Dinitrotoluene	1000
2,6-Dichlorophenol	1000	2-Chlorophenol	1000
2-Chloronapthalene	1000	2-Methylphenol	1000
2-Methylnapthalene	1000	2-Nitrophenol	1000
2-Nitroanaline	1000	3-Methylphenol	500
3 & 4 Methylphenol	1000	3-Nitroaniline	1000
4,6-Dinitro-2-methylphenol	2000	4-Bromophenyl phenyl ether	1000
4-Chloro-3-methylphenol	1000	4-Chloroanaline	1000
4-Chlorophenyl phenyl ether	1000	4-Methylphenol	500
4-Nitroanliline	1000	4-Nitrophenol	2000
Acenapthylene	1000	Acenaphthene	1000
Aniline	1000	Acetophenone	1000
Azobenzene	1000	Anthracene	1000
Benzo[a]pyrene	1000	Benzo[a]anthracene	1000
Benzo[g,h,i]perylene	1000	Benzo[b]fluoranthene	1000
Benzyl alcohol	1000	Benzo[k]fluoranthene	1000
Bis(2-chloroethyl)ether	1000	Bis(2-chloroethoxy)methane	1000
Butyl benzyl phthalate	1000	Biz(2-ethylhexyl) phthalate	1000
Chrysene	1000	Carbazole	1000
Dibenzofuran	1000	Dibenz(a,h)anthracene	1000
Dimethyl phthalate	1000	Diethyl phthalate	1000
Di-n-octyl phthalate	1000	Di-n-butyl phthalate	1000
Diphenylamine	850	Flouranthene	1000
Fluorene	1000	Hexachlorobenzene	1000
Hexachlorobutadiene	1000	Hexachlorocyclopentadiene	1000
Hexachloroethane	1000	Hexadecane	1000
Indeno[1,2,3-cd]pyrene	1000	Isophorone	1000
Naphthalene	1000	n-Decane	1000
Nitrobenzene	1000	N-nitrosodimethylamine	1000
N-nitrosodi-n-propylamine	1000	Pentachlorophenol	2000
N-nitrosodiphenylamine	1000	n-Octadecane	1000
Phenanthrene	1000	Phenol	1000
Pyrene	1000	Pyridine	2000

Table 2		
Additional Stock Standard Mixes		
Mix Name	**Analyte Name**	**Concentration ug/mL**
8270 List 1 Standard 9	3,3'Dichlorobenzidine	2000
	Benzidine	2000
8270 List 1 Standard 10	Benzoic Acid	2000
	Indene	2000
8270 List 1 Standard 11	Atrazine	2000
	Benzaldehyde	2000
	Caprolactam	2000
8270 List 2 Standard 7	Kepone	2000
1,4-Dioxane Standard	1,4-Dioxane	2000
8270 Surrogate Stock	Nitrobenzene-d5	5000
	p-Terphenyl-d14	5000
	Phenol-d5	5000
	2-Fluorobiphenyl	5000
	2-Fluorophenol	5000
	2,4,6-Tribromophenol	5000
	Nitrobenzene-d5	5000
	p-Terphenyl-d14	5000
	Phenol-d5	5000

Table 3			
PAH-SIM Stock Standard			
Analyte Name	**Concentration ug/mL**	**Analyte Name**	**Concentration mg/mL**
Naphthalene	2000	2-Methylnaphthalene	2000
1-Methylnaphthalene	2000	Acenaphthylene	2000
Acenaphthene	2000	Anthracene	2000
Fluorene	2000	Phenanthrene	2000
Fluoranthene	2000	Pyrene	2000
Benzo(a)anthracene	2000	Chrysene	2000
Benzo(b)fluoranthene	2000	Benzo(k)fluoranthene	2000
Benzo(a)pyrene	2000	Indeno(1,2,3-cd)pyrene	2000
Dibenzo(a,h)anthracene	2000	Benzo(ghi)perylene	2000

Table 4					
Standard Reagent Mixtures					
Name	**Component**	**Volume of Solution Used**	**Final Volume**	**Solvent**	**Final Concentration (range, ug/mL)**
8270 Spike 1	8270 List 1 Standard 1	20.00mL	200mL	99% MeOH (from VOA)	100 - 200
8270 Spike 2	8270 List 1 Standard 9	10.00mL	200mL	99% MeOH (from VOA)	100
8270 Spike 3	8270 List 1 Standard 10	20.00mL	200mL	Acetone	100 - 200
	8270 List 1 Standard 11	10.00mL			
8270 Spike 4	8270 List 2 Standard 7	1.25mL	50mL	Acetone	50
8270 Spike 5	Custom DMSO Standard	1.00 mL	100mL	MeOH	
1,4 Dioxane Spike	1,4 Dioxane Standard	1.00mL	100mL	MeOH	20
PAH Spike	PAH Spike Standard	0.125mL	500mL	MeOH	0.5
8270 Surrogate	8270 Surrogate Standard	10.00mL	500mL	90:10 MeOH:DCM	100
1,4 Dioxane Surrogate	8270 Surrogate Standard	20.00mL	200mL	MeOH	10
PAH Surrogate	Custom BNA Surrogate Mix	2.50mL	500mL	MeOH	0.5
LL 8270 Spike 1	8270 Spike 1	10.00mL	100.00mL	99% MeOH (from VOA)	10 - 20
LL 8270 Spike 2	8270 Spike 2	10.00mL	100.00mL	99%MeOH (from VOA)	10

Table 5
Components of PAH-SIM Internal Standard (Surrogate) Solution
(Used only for PAH-SIM-ID)

Analyte Name	Concentration (µg /mL)	Analyte Name	Concentration (µg /mL)
Naphthalene-d8	20.0	Acenaphthylene-d8	20.0
Acenaphthene-d10	20.0	Fluorene-d10	20.0
Phenanthrene-d10	20.0	Fluoranthene-d10	20.0
Pyrene-d10	20.0	Benzo(a)anthracene-d12	20.0
Chrysene-d12	20.0	Benzo(b)fluroanthene-d12	20.0
Benzo(k)fluoranthene-d12	20.0	Benzo(a)pyrene-d12	20.0
Perylene-d12	20.0	Indeno(1,2,3-cd)pyrene	20.0
Dibenzo(a,h)anthracene-d14	20.0	Benzo(g,h,i)perylene-d12	20.0

Table 6
TestAmerica Sacramento 8270C Extraction Summary

Method (Method Code)	Sample Amount	Surrogate	LCS	pH 1 / pH 2	Extract Solvent/Vol.	Extraction Time	Comments
8270C (Aq, 1LS)	1L	0.5mL 8270 Surrogate	1.0mL 8270 Spikes	1-2 / 11-12	3 x 60mL DCM 3 x 60mL DCM	2min shake, 10min rest	Leachates use 200mL diluted to 1L with rgt water. FV=1.0mL 50g NaCl
8270C (Solid, SONC)	30g			NA	3 x 60mL DCM:ACE 1:1	3 min	FV=1.0mL
8270C (Waste Dil'n)	1 g	100uL 8270 Inst Surrogate (5000ug/mL)	See table 7	NA	10mL	NA	Don't concentrate
8270C (Solid, MAE)	15g	0.12mL 8270 source	1.0mL 8270 Spikes	NA	25mL DCM:ACE 1:1	25min	FV=1.0mL
1,4-Dioxane (Aq, 1LS)	1L	500 uL 1,4 Dioxane Surrogate	500uL 1,4 Dioxane Spike	NA	3 x 60mL DCM	2min shake, 10min rest	FV=1.0mL
PAH (Waste Dil'n)	1 g	0.25 1,4 dioxane surrogate	0.5 mL 8270 L1 Std 1 20X	NA	10mL	NA	Don't concentrate
PAH (Aq, 1LS)	1L	0.5mL PAH Surrogate	0.5mL PAH Spike Mix	NA	3 x 60mL DCM	2min shake, 10min rest	FV=1.0mL
PAH (Solid, SONC,)	10 g			NA	3 x 60mL DCM	3 min	
PAH (Aq, LVI)	250mL	0.25mL PAH Surrogate	0.25mL PAH Spike	NA	3 X 30mL	2min shake, 10min rest	FV=0.5mL
PAH (Solid, MAE)	10g	0.5mL PAH Surrogate	0.5mL PAH Spike	NA	25mL DCM	NA	FV=1.0mL
PAH-ID (Aq, 1LS)	1L	100 µL of PAH –ID I.S.. (20 µg/µl)	50 µL of PAH –ID N.S. (20 µg/µl)	NA	3 x 60mL DCM	2min shake, 10min rest	FV=0.5mL
PAH-ID (Solid, SONC)	10 g			NA	3 x 60mL DCM	3 min	
8270D SIM AK (Aq., 1LS)	1L	1.0mL PAH Surrogate	100uL LL 8270 SP1 & 100uL LL8270 SP2 (10ug/mL)	1-2 / 11-12	3 x 60mL DCM 3 x 60mL DCM	2min shake, 10min rest	FV=1.0mL
8270D SIM AK (Solid, SONC)	10 g		200uL LL 8270 SP1 & 200uL LL8270 SP2 (10ug/mL)	NA	3 x 60mL DCM:ACE 1:1	3 min	

1LS = 1L Separatory Funnel; LVI = LVI/RVE Separatory Funnel; MAE = Microwave; SONC = Sonication; SOX = Soxhlet;

Table 7					
Spiking Table					
Method	**Test**	**Surrogate**		**Spike**	
		Volume	**Name**	**Volume**	**Name**
1LS	8270C	0.5mL	8270 Surrogate	1.00mL	8270 Spike 1
				1.00mL	8270 Spike 2
				1.00mL	8270 Spike 3
				1.00mL	8270 Spike 4 (spike only when needed into separate LCS)
				1.00mL	8270 Spike 5 (spike only when needed into separate LCS)
1LS	1,4-Dioxane	0.5mL	1,4-Dioxane Surrogate	0.5mL	1,4-Dioxane Spike
1LS	PAH-SIM	0.5mL	PAH Surrogate	0.5mL	PAH Spike
1LS	8270D	1.00mL	8270D Surrogate	1.00mL	8270D Spike 1
					8270D Spike 2
					8270D Spike 3
LVI	8270C	1.2mL	1,4-Dioxane Surrogate	0.25mL	8270 Spike 1
				0.25mL	8270 Spike 2
				0.25mL	8270 Spike 3
LVI	PAH-SIM	0.25mL	PAH Surrogate	0.25mL	PAH Spike
MAE	8270C	0.5mL	8270 Surrogate	1.00mL	8270 Spike 1
				1.00mL	8270 Spike 2
				1.00mL	8270 Spike 3
MAE	PAH-SIM	0.5mL	PAH Surrogate	0.5mL	PAH Spike
3550B/C	8270C	0.5mL	8270 Surrogate	1.00mL	8270 Spike 1
				1.00mL	8270 Spike 2
				1.00mL	8270 Spike 3
3550B/C	PAH-SIM	0.5mL	PAH Surrogate	0.5mL	PAH Spike
3550B/C	8270 D SIM AK (aq)	1.00mL	PAH Surrogate	100uL	LL 8270 SP1 (10ug/mL)
				100uL	LL 8270 SP2 (10ug/mL)
	8270D SIM AK (s)			200uL	LL 8270 SP1 (10ug/mL)
				200uL	LL 8270 SP2 (10ug/mL)
Waste	8270C	100uL	8270 Inst Surrogate (5000 ug/mL	1.00mL	8270 List 1 Standard 1 (Inst Std)
				1.00mL	8270 List 1 Standard 9 (Inst Std)
Waste	PAH-SIM	0.25mL	1,4-Dioxane Surrogate	0.5mL	8270 List 1 Standard 1 20X

APPENDIX E

CREDERE STANDARD OPERATING PROCEDURES

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Standard Operating Procedure CA-1
Field Activity Documentation

Effective Date: August 2, 2016
Revision: 1

Allison Drouin, Author — 8/2/2016 Date

Theresa Patten, Technical Review — 8/2/2016 Date

Revision	Date	Reason for Revision
1	8-2-2016	EPA and Maine DEP Comments for SOP use with Generic Brownfields Quality Assurance Project Plan

TABLE OF CONTENTS

1. OBJECTIVE AND APPLICABILITY

1.1 OBJECTIVE

The purpose of this Standard Operating Procedure (SOP) is to provide a standard for the documentation of field activities by Credere Associates, LLC (Credere) employees. If followed correctly this SOP will allow for the following:

- Consistency in field reporting to better assimilate documentation between various field staff
- Better understanding by third party users of the documentation
- Defensibility of data collected for a project in the court of law

1.2 APPLICABILITY

This SOP should be used for the documentation of all field activities including site reconnaissance, onsite meetings, sampling events, and oversight.

2. PROCEDURE

2.1 REQUIRED EQUIPMENT

The following is a list of required equipment for documenting field activities:

- Dedicated project bound field logbook with water resistant consecutively numbered pages
- Ink pen (all weather pens are best)
- Field sampling data forms
- Camera
- Backup battery(s)
- Task specific tools for measurement (see task specific SOP for appropriate tools)

2.2 INITIAL PROJECT DEDICATED LOGBOOK SETUP

At the start of a project a dedicated logbook containing bound, consecutively numbered, water resistant pages will be started to be used for the duration of the project. Use of a project dedicated logbook facilitates locating field information and permits reference to prior field events while in the field. In the event that a project logbook is full, a second (or additional thereafter) will be started and each logbook will be consecutively number in order of chronology.

Each logbook will be labeled on the front cover with the following:

- Project name
- Project location and main address
- Credere project number and project manager
- Project (or respective logbook) start and end date

Additionally, the inside cover of the logbook and back of the logbook shall be labeled with the following in case of misplacement:

> If lost, please return to:
> Credere Associates, LLC
> Attn: (project manager on cover)
> 776 Main Street
> Westbrook, Maine 04092
> (207) 828-1272

Field books should be protected from the elements as best possible; pages should not be removed from a logbook; only factual and objective language shall be used.

Exception to Project Specific Logbook

- A client specific logbook may be substituted for instances where numerous small projects are frequently conducted (i.e., brownfields program, environmental services contract, mini-bid program).
- Credere employees should keep a general logbook for short-term projects that are unlikely to result in long term work. For example, for a single site visit or single drilling job that are unlikely to results in additional project work. Individual entries in a general logbook will contain the project name, location, address, project number, and project manager.

2.3 LOGBOOK ENTRIES

A logbook entry should contain enough detail to allow a third party to recreate the occurrences of a task. In addition to task specific information, the logbook entry should also note all modifications to a plan, health and safety precautions and upgrades, public concern, and visitors to the project site.

Daily Entry

A daily entry shall begin at the top of a new page and printed legibly. Entries will be recorded using a 24-hour time clock and entered in the order that they occur. A new entry for each day will begin with the following:

- Date and project at the top of the page
- First and last name of Credere employee followed by initials and time of arrival onsite
- Full names and initials of additional team members and the time of their arrival onsite
- Scope of work for the day
- Weather (e.g., temperature, precipitation, wind directions)
- Subcontractors and duties (e.g., for drilling: drilling company, foreman, make and model of drill rig, type of drilling to be performed, etc.)
- Calibration details for field equipment (e.g., photoionization detector, water quality meter)

If field activities extend beyond one page, each successive page shall have the date, project and initial of person doing logbook entries written at the top. If the logbook changes hands during a daily entry, the initial writer shall sign after their last entry and the full name and initials of the new writer shall be entered consecutively.

The final page of a daily entry shall have open lines marked diagonally with a strike and shall be signed and dated.

Error Corrections

All error corrections will be crossed out with a single line to maintain legibility of the original entry, and the correction will be entered, dated and initialed. At no time should an entry be blacked out or made illegible.

Partial List of Data to be Recorded

The following is a partial list of information typically recorded during field tasks:

- Level of personal protective equipment (PPE)
- Changes to PPE
- Changes in personnel
- Sampling equipment serial numbers
- Equipment calibration details
- Decontamination procedures
- Field screening results
- Observations
- Unusual circumstances
- Problems encountered
- Problem resolutions
- Name and time onsite/offsite of anyone who enters the Site
- Correspondence with project managers
- Sample IDs
- Sample depths
- Method of collection
- Sample times
- Sample analysis requested
- Sample preservation
- Sample volume collected
- QA/QC samples
- Sample duplicate locations
- Site sketch
- Changes in weather

2.4 PHOTOGRAPHS

Photographs are a useful tool in documenting field conditions and communicating observations to other staff. If photographs are of a subject that is indistinguishable (i.e., a soil macrocore, test pit sidewall, surface water sheen), the photograph number and a description of the photograph subject shall be included in the daily logbook entry. If the camera has a time and date stamp, make sure it is on and correct. When taking photographs consider including a frame of reference to indicate scale (e.g., a ruler, a person). Additionally, a wipe board appropriately labeled can aid in identifying photographs.

2.5 FIELD DATA FORMS

Some field tasks have dedicated field forms to facilitate field data collection. If field forms are used these forms are also part of the legal record for the site and should be treated as such.

Field forms should be referenced in the daily logbook entry. Procedures for data entry and error correction should be followed when using field forms. Field forms should be thoroughly completed at the time of the field task to avoid missing information.

3. QUALITY ASSURANCE/QUALITY CONTROL

Logbooks, field data forms, and chains of custody (COCs) shall be checked for consistency. (Note: see SOP CA-16 for specifics on preparation of a COC). In general, the following shall be checked at the close of a field task, sampling event, or project:

- Level of detail in notes is sufficient to recreate a sample, task, or method
- Completeness and accuracy of logbook entries, forms and COCs
- Consistency of data between logbook, field forms and COC
- Equipment calibration records
- Activity documentation compliance with this SOP

4. FIELD DATA MANAGEMENT

Copies of logbook entries, field forms, calibration forms, COCs, photographs, and any other field activity documentation shall be uploaded to the Credere server for easy access during report preparation. Originals of field activity documentation will be kept in the permanent project central file.

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Standard Operating Procedure CA-2
Equipment Decontamination

Effective Date: March 17, 2016
Revision: 0

Allison Drouin, Author — 3/17/2016 Date

Theresa Patten, Technical Review — 3/17/2016 Date

Judd Newcomb, Technical Review — 3/17/2016 Date

Revision	Date	Reason for Revision

TABLE OF CONTENTS

1. OBJECTIVE AND APPLICABILITY

1.1 OBJECTIVE

The purpose of this Standard Operating Procedure (SOP) is to provide guidance for the decontamination of equipment used for the collection of samples or during remedial activities. In circumstances where dedicated equipment is not available or cost effective, if followed correctly this SOP will allow for the following:

- Reduced cross contamination during sampling
- Reduction in incidental transfer of contaminated media to the support zone or other clean areas

A site-specific decontamination objective should be included in site investigation or remedial plans prior to implementation of the work. Preplanning facilitates implementation of decontamination procedures, creates safer working conditions, and assists in meeting the investigation objectives.

Credere Associates, LLC (Credere) SOPs are a guidance, and state requirements, site-specific plans, and special conditions may require alternative approaches.

1.2 APPLICABILITY

This SOP should be used during the collection of samples from media where use of dedicated or disposable sampling equipment is not feasible. Decontamination includes both the removal of physical debris such as caked on soil, sediment or dust, and the removal of possible bonded compounds to sampling equipment through deactivation with a solvent or acid. At a minimum, decontamination should occur at the beginning and end of each sampling day to prevent transfer of contaminants to the support zone or clean storage areas, as well as between individual samples during the course of the day.

2. PROCEDURE

2.1 NECESSARY EQUIPMENT

The following is a list of required equipment for decontamination:

- Appropriate personal protection equipment (PPE)
- Decontaminant selected from Table 1
- Deionized (DI) water, or distilled when DI is unavailable
- 2 High volume spray bottles
- 2 Buckets
- Scrub brush
- Paper towels
- Aluminum foil
- Polyethylene sheeting
- Disposal container appropriate to level of contamination (drum, contractor bag)
- Steamer (optional)
- Pressure washer (optional)

Table 1: Chemical Specific Decontaminant Selection

Chemical	Stage 1 Gross Contamination	Stage 2 Deactivation Rinse
Organics	Simple Green/Alconox/Liquinox	Methanol[1]
Inorganics	Alconox/Liquinox	10% Nitric Acid
Inorganic PCBs	ZEP Heavy Duty Citrus Degreaser	Thorough DI rinse

1 – methanol should not be used as a rinse if methanol is a contaminant of concern.

2.2 TRAINING AND HEALTHY AND SAFETY

Credere employees should have received 40-hour Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) training pursuant to 29 CFR 1910.120 and be current on their 8-hour refresher. Employees must have 24 hours of training specifically in decontamination procedures described herein and proven competence of the SOP in compliance with OSHA 29 CFR 1910.120(e)(4).

Employees will follow the Credere generic and/or Site-specific health and safety plan when implementing decontamination procedures.

2.3 GENERAL METHOD OF DECONTAMINATION

Bulk contamination is defined as caked on soil, sediment or dust present on sampling equipment that can be manually wiped, washed, or rinsed away. The following is the general procedure used for removal of bulk contamination:

1. Dry-brush item to remove bulk mud, chunks, and visible contamination from the tool
2. Rinse tool with water to remove additional bulk contamination

3. Scrub tool with scrub brush to remove additional bulk contamination
4. Wash tool with Stage 1 decontaminant selected from Table 1
5. Rinse tool with DI (or distilled) water
6. Rinse tool with Stage 2 rinse selected from Table 1
7. Rinse again with DI (or distilled water)
8. Dry with paper towels
9. Cover with aluminum foil or use immediately for next sample

This general method of decontamination may be modified based on site-specific requirements. The method of decontamination used, including the selected decontaminants, should be recorded in the field logbook.

More aggressive methods of bulk contamination removal such as air knifing, pressure washing and steam removal may be necessary for large equipment; however, these methods should be avoided where possible due to difficulty containing removed contamination.

Credere employees are not responsible for decontaminating subcontractor's equipment (i.e., drilling equipment, excavator buckets); however, field staff should assure the subcontractor is appropriately decontaminating their equipment. In the event that decontamination procedures are not being followed by the subcontractor, field staff should consult with the project manager and a stop work order may be implemented.

At the end of a sampling day or close of a larger project, all sampling equipment should be decontaminated in a controlled indoor environment, inspected for damage, and made ready for subsequent use.

2.4 DECONTAMINATION WASTE DISPOAL

Decontamination waste should be disposed on a site specific basis. In general, wash water can be discharged onsite unless signs of contamination (odor, sheen) are observed. If wash water is observed to be contaminated, water will be containerized and stored onsite pending characterization. Solid materials such as paper towels, scrub brushed, and aluminum foil can be presumed household waste unless obvious signs of contamination (odor, oily residue, etc.) are present.

3. QUALITY ASSURANCE/QUALITY CONTROL

Quality assurance will be obtained through collection of an equipment blank for laboratory analysis of the appropriate site-specific contaminants of concern (COC) that will indicate the efficacy of the decontamination process. The equipment blank should be collected by rinsing the respective equipment with DI water and collecting the rinsate as a sample. An equipment blank may not be required for all projects or COCs depending on the data quality objectives of a project.

4. DECONTAMINATION DOCUMENTATION

The method of decontamination, selected decontaminant solutions from Table 1, and the type of equipment used for sampling should be recorded in the field logbook. Additionally, the frequency of decontamination throughout a field day and documentation of decontamination prior to each sample and at the beginning/end of each field day should be properly documented in the field logbook in accordance with Credere SOP CA-1.

5. REFERENCES

Credere Associates, LLC, *Standard Operating Procedure CA-1 Field Activity Documentation*, Revision 0, dated August 4, 2015.

Maine Department of Environmental Protection, *Standard Operating Procedure, RWM-DR-017, Equipment Decontamination Protocol, Revision 03*: dated March 23, 2009.

New Hampshire Department of Environmental Services, *Decontamination Procedure, SOP No. HWRB-15, Revision 3*: dated January 2012.

U.S. DoD, *Environmental Field Sampling Handbook, Revision 1.0*, dated April 2013.

U.S. Environmental Protection Agency, *Compendium of Superfund Field Operations Methods*, dated December 1987.

U.S. Environmental Protection Agency Environmental Response Team, *Sampling Equipment Decontamination*, SOP#: 2006, dated August 11, 1994.

U.S. Environmental Protection Agency, Region 4, Science and Ecosystem Support Division, Operating Procedures, *Field Equipment Cleaning and Decontamination*, SESDPROC-205-R3, dated December 18, 2015.

Vermont Department of Environmental Conservation, *Investigation and Remediation of Contaminated Properties Rule*, dated July 27, 2017.

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Standard Operating Procedure CA-4
Soil Description

Effective Date: March 17, 2016
Revision: 0

Allison Drouin, Author — Date: 3/17/2016

Theresa Patten, Technical Review — Date: 3/17/2016

Judd Newcomb, Technical Review — Date: 3/17/2016

Revision	Date	Reason for Revision

TABLE OF CONTENTS

ATTACHMENTS

1. OBJECTIVE AND APPLICABILITY

1.1 OBJECTIVE

The purpose of this Standard Operating Procedure (SOP) is to provide guidance for the observation and description of soil during soil borings, geotechnical drilling, test pitting or grab soil sampling. If followed correctly this SOP will allow for the following:

- Safety of employees performing the sampling
- Collection of reproducible and consistent soil description data between Credere staff and between projects

Credere Associates, LLC (Credere) SOPs are a guidance, and state requirements, site-specific plans, and special conditions may require alternative approaches.

1.2 APPLICABILITY

This SOP should be used during soil borings, geotechnical drilling, test pitting, and soil sampling to describe observed soil, note soil properties, note changes in stratigraphy, and identify visual sources of contamination. This SOP compliments Credere SOPs CA-5: Environmental and Geotechnical Sampling, CA-6: Test Pitting, CA-7: Headspace Field Screening, and CA-8: Monitoring Well Installation.

2. PROCEDURE

2.1 NECESSARY EQUIPMENT

The following is a list of required equipment for soil description:

- Photoionization detector (PID) (if applicable)
- Measuring tape or folding yardstick
- Spoon
- Folding table
- Polyethylene sheeting
- Hand lens
- Soil boring or test pit field logs (**Attachment A**)
- Soil description field guide (**Attachment B**)
- Clip board
- Site plan
- Field logbook
- Indelible pens and markers
- Zip lock bags or soil jars
- Nitrile gloves and other PPE per Health and Safety Plan

2.2 TRAINING AND HEALTHY AND SAFETY

Credere employees should have received 40-hour Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) training pursuant to 29 CFR 1910.120 and be current on their 8-hour refresher. Employees must have 24 hours of training specifically in soil observation and description procedures described herein and proven competence of the SOP in compliance with OSHA 29 CFR 1910.120(e)(4).

Employees will follow the Credere generic health and safety plan (HASP) and the applicable site-specific HASP when implementing these procedures.

2.3 SOIL DESCRIPTION METHODS

When describing soil the primary objective is to accurately describe soils in accordance with an accepted standard to ensure consistency between individual samples, borings, field staff, and projects.

Field Preparation

1. Upon arrival at the Site, begin a field logbook entry recording the Credere staff, scope of work for the day, and weather conditions in accordance with Credere SOP CA-1. Also record the drilling/excavation contractor, foreman, drill rig/excavator make and model, hammer details (if applicable; height, weight, automatic, manual), tooling details, bucket volume, etc.

2. Calibrate PID according to product specifications. Keep record of calibration in the field logbook including PID make and model, serial number, calibration gas and concentration, and span gas check.
3. Set up folding table and cover with clean polyethylene sheeting.
4. The drilling subcontractor will then obtain an observable sample via a macrocore, in line sampling system, split spoon, or excavator bucket per the following methods:
    a. Direct Push Macrocore or In-line Sampling System: The macrocore/casing will be advanced into the subsurface using vibratory force to push through overburden. Note the difficulty of advancing the core and any comments the driller provides. The sample will be extracted in a polyethylene liner and cut open by the contractor for observation. The total **penetration** of the macrocore will be the depth the core was advanced, usually 48 or 60 inches unless refusal was encountered. Measure the soil in the macrocore, or **recovery**, using a measuring tape or folding yard stick and record these measurements as penetration/recovery on the field log.
    b. Split Spoon Sampling: The driller will set up the 2 inch diameter (1.5 or 1 3/8-inch inner diameter), 2-foot long split spoon to begin advancing by placing the split spoon at the ground surface, or at the top of the interval to be sampled using extension rods. The driller will mark 6-inch increments on the rods beginning at the ground surface. The driller will then begin hammering the rods typically using a manual or automatic 140 pound hammer with 30 inch (free fall) drop. Each time the hammer hits the rods is a **blow**. Count the number of blows required to advance the rod 6 inches. Continue this process until the rod has been advanced 2 feet. There should be 4 blow count readings for each 2 foot sample. Record these readings continuously on the field log. To obtain the standard penetration test (SPT) N-value, sum the blow counts of the second and third 6-inch intervals (i.e., the middle two blow counts). This N-value will be used to obtain the density during soil description.

        The split spoon will be removed from the borehole and the driller will open the split spoon. The total **penetration** of the split spoon will be the depth the core was advanced, usually 24 inches unless refusal was encountered. Measure the soil in the split spoon, or **recovery**, using a measuring tape or folding yard stick and record these measurements as penetration/recovery on the field log.
    c. Test Pit: Soil samples will be obtained using standard excavation techniques. This method requires attention to obvious color or grain size and composition changes to obtain samples of individual stratigraphy. Measure the depth of stratigraphic changes using a measuring tape on the sidewall of the excavation.
5. If required, field screen soil samples in accordance with Credere SOP CA-7: Headspace Field Screening or other required field screening methods.

Soil Description

Soil descriptions will include: measurement of the unit in inches, density, color, moisture content, major constituents (all capitalized), minor constituents (first letter capitalized), modifiers, (depositional environment) (classification). **If multiple soil types are observed in one split spoon, measure the thickness of the soil type in actual inches observed and describe the soil types separately.**

EXAMPLES:

Soil descriptions from a 60/50 recovery core sample:

- 0-12" Dense, brownish-yellow, moist, fine SAND, trace Gravel, (fluvial) (SP)
- 12-40" Soft, brown, dry, SILT, (glaciomarine) (ML)
- 40-50" Loose, dark-brown, wet, fine to medium SAND and SILT, some Gravel, (glacial till) (SM)

Density

Although it may be pertinent in some instances to note difficulty of advancing direct push or difficulty of excavation relative to other cores collected from a boring or across the Site, true density characteristics are measured using the Standard Penetration Test (SPT) during sampling using a 2" diameter (1.5 or 1-3/8 inch inner diameter) split spoon as described in the field preparation, **Section 4.b** above. The N-value is obtained by summing the second and third (i.e., the middle two) blow counts. Use the N-value to determine density according to the following table:

Granular Soils		Fine Grained Soils	
N-Value	**Density**	**N-Value**	**Density**
0-4	Very loose	<2	Very soft
4-10	Loose	2-4	Soft
10-30	Medium dense	4-8	Medium stiff
30-50	Dense	8-15	Stiff
>50	Very dense	15-30	Very stiff
		>30	Hard

Note: Granular soils include soil with sand and/or gravel as the predominant grain size. Fine grained soils include silt and/or clay as the predominant grain size. (Sand and silt should be considered granular.)

Color

Color should be compared to the color gauge on the **Attachment B** field soil identification card. Modifiers such as mottled, stained, oxidation, etc. should also be listed.

Moisture

The moisture content should be described based on the following table:

Moisture Descriptor	Description
Dry	No moisture, dusty, crumbles
Moist	Damp, no visible water
Wet	Visible free water, droplets, core shows obviously water

Proportions

The predominant grain size will be listed in all capitals in the soil descriptions. Predominant soil types will be those with greater than 35% of the total volume with the most dominant soil type listed first. For example if there is 50% sand and 40% silt, the description will be SAND and SILT; however, 60% silt and 38% sand will be SILT and SAND. Grain size proportions will be based on the following percentages:

- and 35-50%
- some 20-35%
- little 10-20%
- trace <10% present

Grain Size

Soil grain sizes can be determined using the following sieve/measurements and relative comparisons:

Grain Type	Grain Size	Sieve Passing	Measurement (inches)	Relative Description
Boulder	Boulder	NA	>12	Larger than basketball
Cobble	Cobble	NA	3-12	Fist to basketball size
Gravel	Coarse	NA	0.75-3	Thumb to fist size
	Fine	No. 4 – 0.75 inches	0.19-0.75	Pea to thumb size
Sand	Coarse	No. 10 – No. 4	0.079-0.19	Rock salt to pea size
	Medium	No. 40 – No. 10	0.017-0.079	Sugar to rock salt size
	Fine	No. 200 – No. 40	0.0029-0.017	Sugar size
	Very fine			<Sugar, barely visible grains
Silt	Silt	No. 200	0.0029	Flour size, stains gloves
Clay	Clay	No. 200	0.0029	Flour size, can be rolled

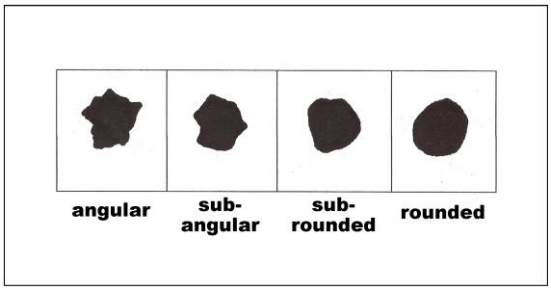
A grain size particle chart may also be helpful in determining grain size in the field. Some general rules of thumb include:

- Silt and clay particles cannot be observed as individual grains and appear as a solid mass.
- Silt typically stains nitrile gloves when rubbed between ones fingers.
- Clay can be rolled (silt may also roll but will crumble upon continued rolling).

- Gravel will take up most of the diameter of the core or split spoon and appear as pulverized pieces that are larger than the core in the case of most coarse gravel.
- Cobbles and boulders cannot be described in core or split spoon samples; however, may be inferred from auger cuttings or observed in test pits.

Gravel and some sand sizes (typically coarse) can also be described based on their shape. This detail sometimes provides addition evidence of a depositional environment. The following can be used to describe grain shape:

- Angular: Sharp edges and distinct planar sides
- Sub-angular: Rounded edges with distinct, planar sides
- Sub-rounded: Well rounded corners
- Rounded: Smooth curved sides, no edges



Modifiers

In some cases a thickness of soil is not significant enough to describe individually or a constituent is not significant enough to be included in the major and minor descriptions; therefore, modifiers are included near the end of the descriptions. Some modifiers include the following:

- Parting 0 to 1/16 inch thick
- Seam 1/16 to ½ inch thick
- Layer ½ to 12 inches thick
- Stratum Greater than 12 inches thick
- Pocket Small, erratic deposit, <1’
- Varve Alternating seams of sand, silt and/or clay
- Occasional One or less/foot of thickness
- Frequent More than one/foot of thickness
- Stratified Alternating layers of varying material or color with layers at least ¼” thick
- Laminated Alternating layers of varying material or color with the layers less than ¼” thick
- Mottled Blocky color variation
- Homogeneous Same color and appearance throughout

Depositional Environment

Possible depositional environments are highly varied and of too great a variety to describe herein. The following table summarizes some depositional environments commonly encountered in Maine as well as their typical characteristics:

Depositional Environment	Description of Deposition	Typical Characteristics
Fill	Material anthropogenically placed for purposes of regrading	Sand, silt or gravel not native to the location. Highly variable.
Urban Fill	Material historically placed during development in urban or developed environments	Sand and silt matrix mixed with brick, concrete, coal fragments, clinker, coal ash, glass, leather, metal scraps, porcelain, etc.
Lacustrine	Deposited in a lake environment	Highly variable depending on the lake setting consisting primarily of alternating sand, silt, and clay with areas of deltaic deposition at the margins.
Fluvial	Deposited in a river environment	Depending on the flow speed during deposition, soil will be well sorted, poorly graded boulders, cobbles, gravel, sand or silt. Typically shows a coarsening or fining trend with depth and rounded particles.
Glaciomarine	Deposited in a marine environment during land submergence after retreat of glacial ice	Soft, fine grained with some fine sand deposited over glacial till. Can be massive in certain locations.
Glacial Till	Deposited beneath glacial ice	Densely compacted sand, silt, clay and gravel typically over bedrock. Particles are typically angular.

Classification

Soil classification is based on the major constituents identified in a soil description. In some cases it may be pertinent to consider minor constituents in the classification depending on the percentages. The following table presents a simplified soil classification scheme for use preliminarily in the field; however, a more detailed soil classification table is included as **Attachment C** to be used as more detailed soil information becomes available through laboratory geotechnical analysis or professional judgement:

Predominant Grain Size	Classification	Description
GRAVEL	GW	Well graded gravel, >2 gravel sizes
	GP	Poorly graded gravel, <2 gravel sizes
	GM	Gravel and Silt
	GC	Gravel and Clay
SAND	SW	Well graded sand, >2 sand sizes
	SP	Poorly graded sand, <2 sand sizes
	SM	Sand and Silt
	SC	Sand and Clay
SILT	ML	Inorganic Silt
	MH	Organic Silt

Predominant Grain Size	Classification	Description
CLAY	CL	Inorganic Clay
	CH	Organic Clay
PEAT	PT	Peat

3. QUALITY ASSURANCE/QUALITY CONTROL

Quality assurance (QA) and quality control (QC) will be obtained through daily self-review of field logs and field notes for completion. Junior staff may be required to collected representative volumes of each soil described for later review by project managers, particularly for geotechnical work.

Field documentation will be reviewed to assure it is compliant with Credere SOP CA-1: Field Activity Documentation and methods used for soil description are compliant with the protocol herein.

4. DOCUMENTATION

The field form in Attachment A should be used to describe soil. This form allows easy transfer of data to the project manager and shows the relative depths and changes in stratigraphy. The form is also designed for easy computation into Credere's electronic boring log program, gINT Logs. Data collection can reasonably considered complete if all fields on the log form are populated. Additionally, the following information should be recorded in the field logbook in compliance with Credere SOP CA-1:

- Date of sampling event
- Credere personnel
- Scope of work
- Weather
- Contractor/foreman/equipment make and model
- Calibration details
- Site conditions
- Changes in scope
- General timeline of field activities

5. REFERENCES

ASTM D 1586-11 *Standard Test Method for Standard Penetration Test (SPT) and Split Barrel Sampling of Soils*, ASTM International, West Conshohocken, PA, 2011, www.astm.org

ASTM D 2487-11 *Standard Practice for Classification of Soils for Engineering Purposes (Unified Soil Classification System)*, ASTM International, West Conshohocken, PA, 2011, www.astm.org

ASTM D 2488-09a *Standard Practice for Description and Identification of Soils (Visual Manual Procedure)*, ASTM International, West Conshohocken, PA, 2009, www.astm.org

ASTM D 5434-12 *Guide for Field Logging of Subsurface Explorations of Soil and Rock*, ASTM International, West Conshohocken, PA, 2012, www.astm.org

Credere Associates, LLC, *Standard Operating Procedure CA-1 Field Activity Documentation*, Revision 0, dated August 4, 2015.

Burmister, *Procedures for Testing Soil, Suggested Methods of Test for Identification of Soils*: dated 1958

Commonwealth of Massachusetts, Department of Environmental Protection, *Standard References for Monitoring Wells, Section 3.5 Soil Classification*. January 1991

Scientific Engineering Response and Analytical Services (SERAS), *Standard Operating Procedure, Description and Identification of Soils*, Revision 0.0: dated February 23, 2004.

US DoD, *Environmental Field Sampling Handbook, Revision 1.0,* dated April 2013.

US Environmental Protection Agency, *Compendium of Superfund Field Operations Methods*, dated December 1987.

Vermont Department of Environmental Conservation, *Investigation and Remediation of Contaminated Properties Rule,* dated July 27, 2017.

Attachment A

Soil Boring and Test Pit Logs
2015

Credere Associates LLC

SITE INFORMATION	WELL SPECIFICATIONS
Project Number/Client:	**Well Depth (feet bgs):**
Site Location:	**Screen Length (feet):**
Date Start/Finish:	**Annulus materials:**
Credere Representative:	**DRILLING EQUIPMENT**
CONTRACTOR	**Equipment (make/model):**
Drilling Contractor:	**Casing/Auger/Core Diameter:**
Foreman:	**Casing Material:**

Boring/Well ID:

TOC Elevation:
TOR Elevation:
GS Elevation:

Depth	Sample Information					Soil Description and Classification
	Sample No./Type	Pen/Rec (inches)	Depth (feet)	Blows (/6")	PID (ppm) (RF=1.0)	
5						
10						
15						
20						
25						

Remarks and Well Details

Boring No:

CREDERE ASSOCIATES, LLC
TEST PIT SAMPLING LOG
Credere Associates, LLC - 776 Main Street, Westbrook, Main 04092 - (207) 828-1272

TEST PIT DATA:

PROJECT NAME: _____ DATE: _____

PROJECT NUMBER: _____ LOCATION ACTIVITY

TEST PIT LOCATION ID: _____ START: _____

CREDERE REPRESENTATIVE: _____ END: _____

CONTRACTOR/FOREMAN/EQUIPMENT: _____

NOTES: ***SAMPLE DETAILS:***

FIELD ANALYSIS DATA:

DEPTH (FT)	SAMPLE DEPTH (FeeT)	PID (ppm)	Lab Sample	SOIL DESCRIPTION / NOTES
0				
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				

Attachment B

Field Soil Identification Card

CREDERE ASSOCIATES, LLC FIELD SOIL IDENTIFICATION

Blue-gray
Light-gray
Gray
Dark-gray
Olive-gray
Greenish-gray
Reddish-brown
Yellowish-red
Brownish-yellow
Dark-brown
Brown
Light-brown
Beige
Tan

GRAIN SIZE

BOULDER	Boulder	Larger than basketball
COBBLE	Cobble	Fist to basketball size
GRAVEL	Coarse	Thumb to fist size
	Fine	Pea to thumb size
SAND	Coarse	Rock salt to pea size
	Medium	Sugar to rock salt
	Fine	Sugar size
	Very Fine	<Sugar, barely visible grains
SILT	Silt	Flour size, stains gloves
CLAY	Clay	Flour size, can be rolled

CLASSIFICATION

GRAVEL	GW	Well graded gravel, >2 gravel sizes
	GP	Poorly graded gravel, <2 gravel sizes
	GM	Gravel and Silt
	GC	Gravel and Clay
SAND	SW	Well graded sand, >2 sand sizes
	SP	Poorly graded sand, <2 sand sizes
	SM	Sand and Silt
	SC	Sand and Clay
SILT	ML	Inorganic Silt
	MH	Organic Silt
CLAY	CL	Inorganic Clay
	CH	Organic Clay
PEAT	PT	Peat

EXAMPLE SOIL DESCRIPTION

0-12" Density, color, moisture, MAJOR constituents, Minor constituents, modifiers (depositional environment) (classification)

0-12" Soft, reddish-brown, wet, SILT and CLAY, (glaciomarine) (ML-CL)

12-24" Dense, light-brown, wet, SILT and very fine SAND, little Gravel, trace Clay, homogeneous (glacial till) (SM)

MODIFIERS

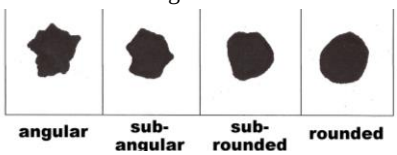
Parting	0 to 1/16 inch thick
Seam	1/16 to ½ inch thick
Layer	½ to 12 inches thick
Stratum	Greater than 12 inches thick
Pocket	Small, erratic deposit, <1'
Varve	Alternating seams of sand, silt and clay
Occasional	One or less/foot of thickness
Frequent	More than one/foot of thickness
Stratified	Alternating layers of varying material or color with layers at least ¼" thick
Laminated	Alternating layers of varying material or color with the layers less than ¼" thick
Mottled	Blocky color variation
Homogeneous	Same color and appearance throughout

MOISTURE

Dry	No moisture, dusty, crumbles
Moist	No visible water, damp
Wet	Visible free water, droplets

GRAVEL ANGULARITY

Angular	Sharp edges and distinct planar sides
Sub-angular	Rounded edges with distinct planar sides
Sub-rounded	Well rounded corners
Rounded	Smooth curved sides, no edges



ROCK QUALITY DESIGNATION (RQD)

Calculate RQD by summing the total length of rock pieces greater than 4" (do not consider mechanical drilling breaks, consider only natural fractures and weathered breaks). Divide the sum by the total core run length and multiply by 100%.

RQD = sum of length of pieces >4"/total core length x 100%

Rock Mass Quality:	0-25%	Very Poor
	25-50%	Poor
	50-75%	Fair
	75-90%	Good
	0-100%	Excellent

SOIL %

Trace	0-10%
Little	10-20%
Some	20-35%
And	35-50%

SAMPLE TYPES

P	Piston sampler
C	Core sampler
SPT	2" Split spoon
PT	Pitcher tube
ST	Shelby tube
BL	Hand cut block
BU	Bulk sampler 3"+

STANDARD PENETRATION TEST (SPT)

Using a 2 inch outer diameter split barrel sampler and 140 lb hammer dropped 30 inches, drive sampler four 6-inch intervals for a total of 24 inches and record blows for each interval. EXAMPLE: 12-14-22-19.

First 6 inches:	This interval is for seating the sampler, drive maximum of 100 blows
Second 6 inches:	Drive maximum of 100 blows.
Third 6 inches:	Drive maximum of 100 blows
Fourth 6 inches:	Drive maximum of 100 blows

If more than 100 blows are counted and the rods are not advancing, consider refusal and record inches advanced EXAMPLE: 12-45-100/3. Sum the second and third interval blow counts to obtain SPT N-value.

PENETRATION and RECOVERY (pen/rec)

Measure of the total depth a core was advanced over the inches of soil collected in the core. Penetrations typically 60 or 48 for direct push and 24 inches for split spoon sampling. Penetration adjusted when refusal is encountered.

EXAMPLE: 60/24

DENSITY/CONSISTENCY

Granular Soils		Silt/Clay	
N-Value	**Density**	**N-Value**	**Density**
0-4	Very loose	<2	Very soft
4-10	Loose	2-4	Soft
10-30	Medium dense	4-8	Medium stiff
30-50	Dense	8-15	Stiff
>50	Very dense	15-30	Very stiff
		>30	hard

Attachment C

Detailed Soil Classification Chart

UNIFIED SOIL CLASSIFICATION SYSTEM

UNIFIED SOIL CLASSIFICATION AND SYMBOL CHART

COARSE-GRAINED SOILS (more than 50% of material is larger than No. 200 sieve size.)			
GRAVELS More than 50% of coarse fraction larger than No. 4 sieve size	Clean Gravels (Less than 5% fines)		
		GW	Well-graded gravels, gravel-sand mixtures, little or no fines
		GP	Poorly-graded gravels, gravel-sand mixtures, little or no fines
	Gravels with fines (More than 12% fines)		
		GM	Silty gravels, gravel-sand-silt mixtures
		GC	Clayey gravels, gravel-sand-clay mixtures
SANDS 50% or more of coarse fraction smaller than No. 4 sieve size	Clean Sands (Less than 5% fines)		
		SW	Well-graded sands, gravelly sands, little or no fines
		SP	Poorly graded sands, gravelly sands, little or no fines
	Sands with fines (More than 12% fines)		
		SM	Silty sands, sand-silt mixtures
		SC	Clayey sands, sand-clay mixtures
FINE-GRAINED SOILS (50% or more of material is smaller than No. 200 sieve size.)			
SILTS AND CLAYS Liquid limit less than 50%		ML	Inorganic silts and very fine sands, rock flour, silty of clayey fine sands or clayey silts with slight plasticity
		CL	Inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays
		OL	Organic silts and organic silty clays of low plasticity
SILTS AND CLAYS Liquid limit 50% or greater		MH	Inorganic silts, micaceous or diatomaceous fine sandy or silty soils, elastic silts
		CH	Inorganic clays of high plasticity, fat clays
		OH	Organic clays of medium to high plasticity, organic silts
HIGHLY ORGANIC SOILS		PT	Peat and other highly organic soils

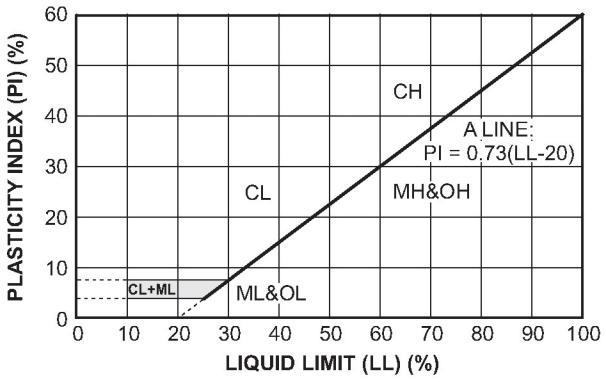
LABORATORY CLASSIFICATION CRITERIA

GW	$C_u = \frac{D_{60}}{D_{10}}$ greater than 4; $C_c = \frac{D_{30}}{D_{10} \times D_{60}}$ between 1 and 3	
GP	Not meeting all gradation requirements for GW	
GM	Atterberg limits below "A" line or P.I. less than 4	Above "A" line with P.I. between 4 and 7 are borderline cases requiring use of dual symbols
GC	Atterberg limits above "A" line with P.I. greater than 7	
SW	$C_u = \frac{D_{60}}{D_{10}}$ greater than 4; $C_c = \frac{D_{30}}{D_{10} \times D_{60}}$ between 1 and 3	
SP	Not meeting all gradation requirements for GW	
SM	Atterberg limits below "A" line or P.I. less than 4	Limits plotting in shaded zone with P.I. between 4 and 7 are borderline cases requiring use of dual symbols.
SC	Atterberg limits above "A" line with P.I. greater than 7	

Determine percentages of sand and gravel from grain-size curve. Depending on percentage of fines (fraction smaller than No. 200 sieve size), coarse-grained soils are classified as follows:

Less than 5 percent ..... GW, GP, SW, SP
More than 12 percent ..... GM, GC, SM, SC
5 to 12 percent ..... Borderline cases requiring dual symbols

PLASTICITY CHART



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Standard Operating Procedure CA-5
Environmental Soil Sampling

Effective Date: May 27, 2016
Revision: 0

Allison Drouin 5/27/2016
Allison Drouin, Author Date

Theresa Patten 5/27/2016
Theresa Patten, Technical Review Date

Judd R. Newcomb 5/27/2016
Judd Newcomb, Technical Review Date

Revision	Date	Reason for Revision

TABLE OF CONTENTS

1. OBJECTIVE AND APPLICABILITY

1.1 OBJECTIVE

The purpose of this Standard Operating Procedure (SOP) is to provide guidance for the collection of soil samples from surficial and subsurface soil. If followed correctly, this SOP will allow for the following:

- Safety of employees performing the sampling
- Collection of representative samples with reproducible results

Credere Associates, LLC (Credere) SOPs are guidance, and state requirements, site-specific plans, and special conditions may require alternative approaches.

1.2 APPLICABILITY

This SOP should be used during soil sampling activities. This SOP does not cover procedures for field screening or description of soil, which are provided in Credere SOPs CA-7: Headspace Field Screening and CA-4: Soil Description, respectively. This SOP also does not cover the use of Encore® brand samplers.

2. PROCEDURE

2.1 NECESSARY EQUIPMENT

The following is a list of required equipment:

- Subcontractor drilling/excavation equipment or sample collection device (e.g., hand auger, shovel, etc.)
- Polyethylene sheeting
- Folding table (optional, for convenience)
- Sweeping brush
- Stainless steel spoons
- Stainless steel bowls
- Wooden stakes, marking paint, or pin flags
- Laboratory provided sample containers, cooler and ice
- Plastic syringes (if sampling for volatiles)
- Decontamination fluids
- Deionized or distilled water
- One 5-gallon buckets with water and detergent, one 5-gallon bucket with water, and scrub brush (if using shovel or hand auger)
- Paper towels
- Appropriate personal protection equipment (PPE)
- Site plan
- Field logbook
- Chain of custody
- Ink pens
- Digital camera

2.2 TRAINING AND HEALTHY AND SAFETY

Credere employees must have received 40-hour Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) training pursuant to 29 CFR 1910.120 and be current on their 8-hour refresher. Employees must have 24 hours of training specifically in soil sampling procedures described herein and proven competence of the SOP in compliance with OSHA 29 CFR 1910.120(e)(4).

Employees will follow the Credere generic health and safety plan (HASP) and the site-specific HASP when collecting soil samples.

2.3 SOIL SAMPLE LOCATING AND TARGET DEPTHS

Soil sample locating is highly site-specific. Additionally, sample locations laid out in a work plan may require field adjustment based on site conditions (e.g., bias toward obvious evidence of contamination or just beyond it depending on sample objectives). Typically, sample locations are selected based on the location of historical source areas, around previously detected

contamination for delineation purposes, or in areas presumed to be unimpacted for background comparison.

Sample target depths can be based on soil observations and field screening methods. Target depths are also selected based on sample objectives typically targeting the greatest area of contamination or beyond that area for delineation purposes. Some common sample target depths include the following:

- Surface soil (0 to 2 feet below ground surface [bgs])
- Depth of observed fill (anthropogenic material, non-native material)
- Depth of greatest field screening response
- Depth of groundwater interface
- Depth of a specific geologic feature (e.g., sand/gravel seam)
- Interval above confining layer or bedrock
- Contaminant saturated soil
- First encountered native soil (i.e., beyond contamination)
- A specific pre-designated depth for delineation purposes

Unless targeting a specific interval, non-volatile samples should consist of a 2-foot interval. Volatile samples will be collected from a specific exact depth due to the nature of the collection procedure.

2.4 SOIL SAMPLE COLLECTION PROCEDURES

Soil samples can be collected from the surface (0 to 2 feet bgs) or subsurface via a number of methods. Hand tools such as shovels or hand augers can be used to collect samples from the surface. Drilling equipment such as a hollow stem auger with split spoon samples or direct-push drill rig with macrocore or other type of sampling device can be used to obtain samples from the subsurface. If cores of soil are collected using drilling equipment, soil should be continuously logged in accordance with Credere SOP CA-4 to observe the designated target depth features. Samples should generally be collected in order of increasing impacts where possible.

Sample Preparation

1. Upon arrival at the Site, begin a field logbook entry recording the Credere staff, scope of work for the day, and weather conditions in accordance with Credere SOP CA-1. Also record the drilling contractor, foreman, drill rig make and model, and tooling details.
2. Calibrate field instruments according to Credere SOP CA-11 and product specifications. Keep record of calibration in the field logbook including instrument make and model, serial number, calibration gas and concentration, and span gas check.
3. Set up the sampling station with the optional folding table, truck tailgate, or on the ground surface by covering with clean polyethylene sheeting.

4. Prepare decontamination station. Note whether decontamination fluids must be containerized or can be discharged to the ground surface per the work plan.
5. Decontaminate sampling tools and cover with aluminum foil to keep clean until sampling or place on clean polyethylene sheeting.
6. Locate sample locations in accordance with the prepared work plan or project objectives. Mark the locations with stakes/paint/pin flags. Note any adjustments to sample locations based on field conditions in the log book.
7. Begin collecting soil samples with hand tools or drilling to target depth using standard techniques for the respective method. Describe soil in accordance with Credere SOP CA-4 and field screen soil, where applicable, in accordance with Credere SOP CA-7.

Grab Sample Collection

A grab sample represents a single location or interval within the soil column or on a soil surface. Grab samples can represent any soil thickness; therefore, even though an interval may span 2+ feet it is still considered a grab sample. According to the work plan, samples will be collected in order of decreasing volatility.

1. Don clean nitrile gloves prior to collecting each sample.
2. Determine the representative soil to be sampled:
    a. Direct measure to two feet below the ground surface (or other specified shallow interval) for surface soil samples.
    b. Direct measure and collect representative soil from a test pit. Soil for sampling should be collected from the center of the mass not in contact with the excavator bucket.
    c. In a direct push liner, determine the proportion of soil that represents the target depth based on the penetration and recovery of the core. (e.g., To collect a 6 to 8 foot sample from a 5 to 10 foot core with a penetration/recovery of 60/55, divide 55 by 60 and multiply by 12 inches (for the top) and 36 inches (for the bottom) [6 feet is 12 inches into the penetration and 8 feet is 36 inches into the penetration]. Directly measure and collect 11 to 33 inches from the recovery to represent 6 to 8 feet.). Use caution not to collect slough of caved in soil, which is generally present as loose soil in the top of the direct push liner.
    d. Soil collected in split spoon samplers can be proportionally divided in half or quarters to represent an interval. It may be necessary to collect soil from multiple 24-inch split spoons to obtain soil for a target interval. (e.g., To collect soil for a 5 to 7 foot sample collect the bottom half of recovery from the 4 to 6 foot split spoon and top half of recovery from the 6 to 8 foot split spoon). Use caution not to collect slough of caved in soil, which is generally present as loose soil in the top of the split spoon sampler.
3. If sampling for volatiles, using a dedicated syringe obtain 10 grams of soil directly from the representative soil as determined per above and transfer to a 40 mL methanol

preserved VOA. Remove any sand grains from the cap threads and securely replace the VOA vile cap. Failure to remove soil from the cap threads can result in leaking of preservative out of or leaking of cooler ice melt into the container, which both may impact the data. **If sampling for only volatiles, also collect a syringe full of soil and cap the syringe for percent moisture analysis by the lab.** Place the filled syringe and VOA vile together in a bubble wrap sleeve. If other analyses are requested, this can be performed from other unpreserved soil volume.

4. Collect the remaining representative soil into a decontaminated stainless steel bowl using gloved hands or a decontaminated stainless steel spoon. Remove coarse gravel and organic detritus. If additional volume is needed due to poor recovery of many analyses, additional cores may be warranted. Homogenize the soil in the bowl, clayey or silty soil may require extra effort to homogenize well, and begin filling the remaining sample containers according to the following. If multiple sample containers are to be filled, alternate between containers adding a spoonful at a time to each.
    a. SVOCs, PAHs, EPH, and PCBs: Fill an amber 4 to 8 oz. glass jar. Wipe away sand from the cap threads and replace cap. Failure to remove sand from the cap threads can result in leaking of cooler ice melt into the container, which may impact the data.
    b. Metals: Fill a clear 4 to 8 oz. glass jar to the neck. Wipe away sand from the cap threads and replace cap. Failure to remove sand from the cap threads can result in leaking of cooler ice melt into the container, which may impact the data.
5. Label the sample container with the boring ID and depth as the sample ID: CA-SB-1 (0-2/1) where CA-SB-1 is the location, 0-2 is the interval for non-volatile analyses, and 1 is the grab depth for the volatile analysis.
6. Place samples immediately on ice. Record the sample time, ID, analyses, preservative, and sample volume in the field log book.

Composite Sample Collection

Composite sampling involves collecting multiple grab samples, or aliquots, from multiple locations or discontinuous depths and combining them for a more generalized and average result. Sampling procedures are generally consistent with above with the following modifications:

- Composite volatile samples can be collected in one of two ways:
    - Laboratory composite: Collect one 40 mL VOA vile from each location to be composited per the grab sample methodology. These grab samples will be submitted with one sample ID and laboratory composite will be requested on the chain of custody. The lab will extract from each vile and composite for one analysis under laboratory controlled conditions.
    - Field composite: Depending on the number of aliquots, request a volatile container from the laboratory with a proportional amount of methanol to the number of aliquots required. Collect 5 grams of soil at each aliquot location into the one VOA vile provided by the laboratory. If three aliquots or less are

required, 3 grams (or 5 grams for two aliquots) can be collected from each location for field compositing in a standard 40 mL VOA vile.

- Composite sampling for other analyses simply requires collection of soil from more than one grab location per the work plan into the same decontaminated stainless steel bowl. Soil from the multiple locations is then homogenized and placed in laboratory provided glassware the same as it would have been for a grab sample. The same volume of soil should be collected from each aliquot location for homogenization.

2.5 POST-SAMPLING PROCEDURE

The following procedure should be completed after collection of soil samples:

1. Complete the chain of custody using the notes recorded in the field logbook in accordance with Credere SOP CA-16.
2. Decontaminate non-dedicated equipment for use at the next sample location or for transport in accordance with Credere SOP CA-2. If using a shovel or hand auger to collect soil samples, using 5-gallon bucket of detergent and rinse water to remove bulk soil and perform subsequent decontamination with appropriately selected decontamination fluids, rinse, and dry method per Credere SOP CA-2.
3. Using the sweeping brush, brush away soil from the previously collected sample, and wipe polyethylene sheeting with a damp paper towel. If sampled soil was muddy, replace polyethylene sheeting or slide sheeting over the edge of the table to advance to a clean surface.
4. Store the sample containers on ice being sure to avoid pooled water in the cooler. Regularly replace ice and drain meltwater.

3. QUALITY ASSURANCE/QUALITY CONTROL

Quality assurance (QA) and quality control (QC) will be obtained through collection of additional samples. The types of samples to be collected are dependent on the data quality objectives and should be defined in the project's proposal/scope of work, Site-Specific Quality Assurance Project Plan (SSQAPP), regulatory/client guidance, or similar. Types of QA/QC samples that may be collected include the following:

- Field duplicates (optional)
- Matrix spike/matrix spike duplicates (MS/MSD) (optional)
- Trip blanks (should be included with volatile analyses)
- Equipment blanks (e.g., after decontaminating an auger or sampler)
- Temperature blanks (laboratory specific, should be included in sample coolers for certain labs)

Additionally, field documentation will be reviewed to assure it is compliant with Credere SOP CA-1: Field Activity Documentation and methods used for sampling are compliant with the protocol herein.

4. FIELD DOCUMENTATION

The following information should be recorded in the field logbook in compliance with Credere SOP CA-1:

- Date of field activity
- Credere personnel
- Scope of work
- Weather (particularly precipitation)
- Health and safety precautions
- Contractor/foreman/equipment make and model (if applicable)
- Changes in scope or deviations from the work plan
- Correspondence with Project Managers and/or Clients
- Sketches of sample locations relative to Site features and measurements (i.e., "swing ties") from permanent landmarks where location accuracy is required
- Sample details including IDs, time of collection, requested analyses, volume of sample collected, and preservatives
- Decontamination procedures
- General timeline of field activities

5. REFERENCES

Credere Associates, LLC, *Standard Operating Procedure CA-1: Field Activity Documentation*, Revision 0, dated August 4, 2015.

Credere Associates, LLC, *Standard Operating Procedure CA-2: Equipment Decontamination*, Revision 0, dated March 17, 2016.

Credere Associates, LLC, *Standard Operating Procedure CA-4: Soil Description*, Draft, dated TBD.

Credere Associates, LLC, *Standard Operating Procedure CA-7: Headspace Field Screening*, Draft, dated TBD.

Credere Associates, LLC, *Standard Operating Procedure CA-16: Chain of Custody Preparation*, Draft, dated TBD.

Maine Department of Environmental Protection, Standard Operating Procedure, *Protocol for Collecting Soil Samples,* SOP RMW-DR#006, April 3, 2009.

New Hampshire Department of Environmental Services, *Soil Sampling Procedure*, SOP No. HWRB-11, Revision 1, January 2012

U.S. DoD, *Environmental Field Sampling Handbook, Revision 1.0,* dated April 2013.

U.S. Environmental Protection Agency Environmental Response Team, *Soil Sampling*, SOP#: 2012, dated February 18, 2000.

U.S. Environmental Protection Agency, *Standard Operating Procedure for Soil, Sediment and Solid Waste Sampling*, SOP EIASOP_SOILSAMPLING2, Revision 2, dated February 13, 2004.

U.S. Environmental Protection Agency, Region 4, Science and Ecosystem Support Division, Operating Procedure, *Soil Sampling*, SESDPROC-300-R3, Revision 3, dated August 21, 2014.

Vermont Department of Environmental Conservation, *Investigation and Remediation of Contaminated Properties Rule,* dated July 27, 2017.

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Standard Operating Procedure CA-6
Test Pitting

Effective Date: March 17, 2016
Revision: 0

Allison Drouin 3/17/2016
Allison Drouin, Author Date

Theresa Patten 3/17/2016
Theresa Patten, Technical Review Date

Judd R. Newcomb 3/17/2016
Judd Newcomb, Technical Review Date

Revision	Date	Reason for Revision

TABLE OF CONTENTS

1. OBJECTIVE AND APPLICABILITY

1.1 OBJECTIVE

The purpose of this Standard Operating Procedure (SOP) is to provide guidance for the excavation of test pits. If followed correctly this SOP will allow for the following:

- Safety of employees overseeing test pitting activities
- Observation of subsurface condition
- Determination of the presence or extent of subsurface features

Credere Associates, LLC (Credere) SOPs are a guidance, and state requirements, site-specific plans, and special conditions may require alternative approaches.

1.2 APPLICABILITY

This SOP should be used during the excavation of exploratory test. Samples collected from test pits should be collected in accordance with Credere SOP CA-5: Environmental and Geotechnical Soil Sampling, and soil should be logged in accordance with Credere SOP CA-4: Soil Description.

2. PROCEDURE

2.1 NECESSARY EQUIPMENT

Most equipment for test pitting will be supplied by a subcontractor hired to excavate test pits. The following is a list of required equipment:

- Subcontractor excavating equipment
- Appropriate personal protection equipment (PPE)
- Photoionization detector (PID) (if applicable)
- Site plan
- Field logbook
- Test pit logs (**Appendix A**)
- Ink pens
- Measuring tape with weighted end
- Camera
- Polyethylene sheeting (as a backup if contractor cannot supply)

2.2 TRAINING AND HEALTHY AND SAFETY

Credere employees must have received 40-hour Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) training pursuant to 29 CFR 1910.120 and be current on their 8-hour refresher. Employees must have 24 hours of training specifically in test pit excavation procedures described herein and proven competence of the SOP in compliance with OSHA 29 CFR 1910.120(e)(4).

Employees will follow the Credere generic health and safety plan (HASP) when implementing test pit excavation procedures and the site-specific HASP for the project. **Credere staff should never enter an unshored excavation greater than 3 feet in depth, and should never enter an excavation where volatiles or other oxygen displacing atmospheres are present without appropriate training or PPE. Use extreme caution when measuring sidewalls and standing close an excavation's edge, particularly for excavations with steep sidewalls and evidence of sloughing near the groundwater table.** Excavation should cease and the project manager should be contacted immediately if any of the following unknown conditions are encountered:

- Unexpected odors such as petroleum, natural gas, or exhaust
- Non-aqueous phase liquid (NAPL) is observed on water within an excavation
- Drums or unanticipated underground storage tanks (USTs)
- Unmarked utilities

If volatiles are a contaminant of concern, ambient air should be monitored continuously during period of open excavation.

2.3 TEST PIT LOCATING

Test pit locating is highly site-specific; however, is typically focused around suspect subsurface structures, anomalies identified by geophysical surveys, areas of fill, and known areas of contamination. Additionally, test pit locations laid out in a work plan may require adjustment if field conditions present issues (e.g., bedrock encountered shallower than expected, new contamination encountered, etc.). Generally, test pit locations should be selected based on the project objectives for the test pit and to most efficiently assess overall project objectives. Objectives for a test pit may include:

- Assessment of the extent of subsurface structures or fill
- Determination of fill thicknesses
- Detailed observation of subsurface stratigraphy

Before selecting test pit locations, a comprehensive review of any prior environmental data, local geology, hydrology, topography, and chemical characteristics of the subsurface is advantageous.

2.4 TEST PIT EXCAVATION METHOD

The following steps should be implemented when excavating test pits:

1. Upon arrival at the Site, begin a field logbook entry recording the Credere staff, scope of work for the day, and weather conditions. Also record the excavation contractor, foreman, excavator make and model, and bucket size.
2. Calibrate the PID according to product specifications if applicable. Keep record of calibration in the field logbook including PID make and model, serial number, calibration gas and concentration, and span gas check.
3. Locate test pits in accordance with the prepared work plan or project objectives. Note any adjustments to test pit locations based on field conditions in the log book.
4. Begin excavation to target depth using standard techniques for excavation. If excavation is unearthing unidentified anomalies, excavation should advance carefully to avoid damage the anomaly (e.g., puncturing an underground storage tank or damaging utilities).
5. If soil is known to be contaminated, soil should be stockpiled adjacent to the test pit on 10-mil polyethylene sheeting to avoid cross contamination of surface soil adjoining the test pit. It may be beneficial to reserve some surface soil in a separate pile to backfill with relatively clean material at the surface.
6. Once the test pit has been excavated to the target depth or can no longer progress due to physical limitations (e.g., nearby structural integrity, bedrock encountered, groundwater causing collapse), the following test pit observations and/or activities should be completed:
    a. Describe any identified anomalies including size, depth to the anomaly, condition, etc. on the Test Pit Log (**Attachment A**).

b. Describe the soil stratigraphy in accordance with Credere SOP CA-4: Soil Description and record descriptions on the Test Pit Log (**Attachment A**). Measure distinct stratigraphic layers along the sidewall.

c. Record if groundwater is present and at what depth it appears to be entering the excavation.

d. If applicable, collect any field screening or soil analytical samples from the test pit. Record field screening results on the Test Pit Log with a drawing showing field screening locations within the test pit.

e. Measure the total depth, width, and length of the test pit and draw a schematic of the orientation (include north arrow) and relative location on the test pit log.

f. Photograph pertinent test pit features and sidewalls including measuring tape for scale to pertinent features including observed contaminant layers, archeological features, unidentified utilities, etc. (lighting can often make this difficult).

7. Backfill and compact the excavation in approximate 1 foot lifts placing soil back into the excavation in the general order it was removed with the goal of have clean topsoil at the surface to prevent exposure. Use reserved topsoil at the surface if this material was previously segregated.

8. The excavator bucked should at a minimum be brushed of any bulk soil and also rinsed if possible prior moving to the next test pit. Certain contaminants and investigations may require a more stringent decontamination procedure.

3. QUALITY ASSURANCE/QUALITY CONTROL

Quality assurance (QA) and quality control (QC) will be obtained through strict adherence to work plans, appropriate communication with project managers, and proper field documentation.

Field documentation will be reviewed to assure it is compliant with Credere SOP CA-1: Field Activity Documentation and methods used for test pitting are compliant with the protocol herein.

4. FIELD DOCUMENTATION

The following information should be recorded in the field logbook in compliance with Credere SOP CA-1:

- Date of field activity
- Credere personnel
- Scope of work
- Weather
- Contractor/foreman/equipment make and model
- Changes in scope
- Correspondence with Project Managers
- Test pit details (depth, width, length)
- Significant observations
- Decontamination procedures
- General timeline of field activities

5. REFERENCES

Credere Associates, LLC, *Standard Operating Procedure CA-1 Field Activity Documentation*, Revision 0, dated August 4, 2015.

Credere Associates, LLC, *Standard Operating Procedure CA-4 Soil Description*, Draft, dated TBD.

Credere Associates, LLC, *Standard Operating Procedure CA-5 Environmental and Geotechnical Soil Sampling*, Draft, dated TBD.

Commonwealth of Massachusetts Department of Environmental Protection, *Standard References for Monitoring Wells*, WSC-310-91, dated January 1991.

US DoD, *Environmental Field Sampling Handbook, Revision 1.0*, dated April 2013.

US Environmental Protection Agency, *Compendium of Superfund Field Operations Methods*, dated December 1987.

APPENDIX A

Test Pit Log

2016

CREDERE ASSOCIATES, LLC
TEST PIT SAMPLING LOG
Credere Associates, LLC - 776 Main Street, Westbrook, Main 04092 - (207) 828-1272

TEST PIT DATA:

PROJECT NAME: _____ DATE: _____

PROJECT NUMBER: _____ LOCATION ACTIVITY

TEST PIT LOCATION ID: _____ START: _____

CREDERE REPRESENTATIVE: _____ END: _____

CONTRACTOR/FOREMAN/EQUIPMENT: _____

NOTES: ***SAMPLE DETAILS:***

FIELD ANALYSIS DATA:

DEPTH (FT)	SAMPLE DEPTH (FeeT)	PID (ppm)	Lab Sample	SOIL DESCRIPTION / NOTES
0				
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				

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Standard Operating Procedure CA-7
Headspace and Field Screening

Effective Date: May 20, 2016
Revision: 0

Allison Drouin, Author — 5/20/2016 Date

Theresa Patten, Technical Review — 5/20/2016 Date

Judd Newcomb, Technical Review — 5/20/2016 Date

Revision	Date	Reason for Revision

TABLE OF CONTENTS

1. OBJECTIVE AND APPLICABILITY

1.1 OBJECTIVE

The purpose of this Standard Operating Procedure (SOP) is to provide guidance for the field screening of samples. If followed correctly this SOP will allow for the following:

- Safety of employees performing the sample screening
- Collection of data sufficient for making project decisions

Credere Associates, LLC (Credere) SOPs are a guidance, and state requirements, site-specific plans, and special conditions may require alternative approaches.

1.2 APPLICABILITY

This SOP should be used during screening of samples in cases where screening data is used to supplement analytical laboratory data or to appropriately select locations/depths for submittal to a laboratory.

2. PROCEDURE

2.1 NECESSARY EQUIPMENT

The following is a list of required equipment for field screening:

- Appropriate personal protection equipment (PPE)
- 10.2 to 10.6 eV photoionization detector (PID) with moisture filter and 100 (part per million by volume) ppm_v isobutylene calibration gas
- Soil jars and aluminum foil (if in New Hampshire or Massachusetts)
- 3 mil metalized polyethylene bags (if in Maine)
- Pre-prepared Oil-in-Soil oleophilic dye tests
- Oil-free water
- Site plan
- Field logbook
- Indelible marking pens
- Camera

2.2 TRAINING AND HEALTHY AND SAFETY

Credere employees must have received 40-hour Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) training pursuant to 29 CFR 1910.120 and be current on their 8-hour refresher. Employees must have 24 hours of training specifically in field screening procedures described herein and proven competence of the SOP in compliance with OSHA 29 CFR 1910.120(e)(4).

Employees will follow the Credere generic health and safety plan when implementing field screening procedures, and the site-specific HASP for the project.

2.3 FIELD SCREENING METHOD

When field screening, the general objective is to quickly analyze a large volume of samples in the field in order to target the best sampling locations/depths for analytical analyses in accordance with the project objectives. Field screening may also be used to collect large quantities of data to supplement laboratory analytical data.

The Massachusetts Department of Environmental Protection (MassDEP) uses a jar headspace method for screening organics in soil, the Maine (DEP) uses a bag headspace method for field screening soil, and New Hampshire Department of Environmental Services (NHDES) SOP references use of both methods as long as one method is used consistently across a Site.

Jar headspace should be used in the three states for screening groundwater. State requirements/SOPs outside of Maine, New Hampshire, and Massachusetts should be referenced prior to sampling to ensure this SOP is compliant.

Screening Preparation

1. Upon arrival at the Site, begin a field logbook entry recording the Credere staff, scope of work for the day, and weather conditions.
2. Calibrate PID according to product specifications using 100 ppm_v isobutylene gas and set unit to a response factor of 1.0. Keep record of calibration in the field logbook including PID make and model, serial number, calibration gas and concentration, lamp energy rating, and span gas check. Affix moisture filter to tip of PID probe.
3. Obtain soil from the surface, macrocore, split spoon, test pit, or other means for field screening. Groundwater purged from monitoring wells may also be screened to assess if purge water is appropriate for discharge to the Site.
4. Screen soil or groundwater in accordance with one of the following methods

Jar Headspace

Jars will be used to field screen headspace in Massachusetts and New Hampshire according to the following steps:

1. Label jar lid with sample depth.
2. Place approximately 200 grams (approximately a 5 oz. can of tuna) of soil in an 8 to 16 ounce glass jar. Remove >½ inch gravel or vegetation. Cover the top with aluminum foil and replace the lid. Shake vigorously for 30 seconds and allow to accumulate headspace for at least 15 minutes in a warm location (>32 degrees Fahrenheit). Do not leave samples more than 2 hours.
3. After headspace has accumulated for 15 minutes, shake jar again for 30 seconds. Settle soil to the bottom of the jar.
4. Remove the lid and insert PID through the aluminum foil seal using care not to contact the soil within the jar.
5. Observe PID readings for 15 seconds and record the highest reading obtained.

Bag Headspace

Metalized 3 mil polyethylene bags (e.g., Associated Bag Company Item Number 183-52) will be used to field screen headspace in Maine or New Hampshire. Note: Standard Ziploc or sandwich bags are <u>not acceptable</u> for field screening bags; if metalized bags are not available, default to the jar headspace method as these materials are readily available at most grocery stores. Bag headspace screening will be conducted according to the following steps:

1. Label bag with sample depth.
2. Place approximately 200-250 grams (approximately a 5 oz can of tuna) of soil into the metalized bag and seal bag. Knead the soil to break up clumps and shake vigorously for 30 seconds. Allow bag to accumulate headspace for at least 10 minutes in a warm location (>32 degrees Fahrenheit). Do not leave samples more than 30 minute as volatiles will dissipate through the bag.

3. After headspace has accumulated for 15 minutes, knead/shake bag again for 30 seconds and let stand for another 2 minutes. Settle soil to the bottom of the bag.
4. Open a small hole in the seal and quickly insert the PID probe using care not to contact the soil within the bag.
5. Observe PID readings for 15 seconds or until readings begin to fall, and record the highest reading obtained.

Oleophilic Dye Test

Oleophilic dye test will be used at Sites where fuel oil or heavier is the anticipated petroleum type. Dye test will be used in conjunction with PID to assess if soil is petroleum saturated, residual petroleum is present or if no petroleum is present. Dye test do not give an indication of the relative concentration of petroleum other than saturated or unsaturated. Dye test screening will be conducted according to the following steps:

1. Ensure the pre-prepared Oil-in-Soil dye test has a dye cube beneath the lid and an indicator bead present.
2. Using the markings on the pre-prepared Oil-in-Soil dye test jar, fill to first line with soil and to second line with petroleum free water.
3. Shake jar until the dye cube beneath the jar lid has completely dissolved.
4. Allow to sit for 10 minutes.
5. Determine results based on the following table:

Result	Observation
Saturated	Obvious due in soil matrix and in water (stains sides of jar)
Positive	Bead is dyed dark-pink/red, no coloration in water
Slightly positive	Bead is dyed light-pin, no coloration in water
Undetected	No coloration on bead (remains white) or in water

3. QUALITY ASSURANCE/QUALITY CONTROL

Quality assurance (QA) and quality control (QC) will be checked through duplicate screening where necessary and through frequent calibration checks according to the following:

- Jar headspace screening results will be checked through duplicate screening at a minimum of one screening sample per 20 screening samples. Two jars will be filled and screened in succession. PID results will be compared and should be within a 20% difference range.
- If bag headspace readings will be used to make project decisions beyond selection of locations for laboratory sampling (i.e., if field screening will be used for UST Site Assessment in accordance with Maine DEP Chapter 691 in lieu of laboratory analyses), bag headspace readings will be performed in triplicate.
- If bag headspace readings will be used only for selection of samples for laboratory analysis and for relative concentrations comparison, one screening sample per every 20 screening samples will be screened in duplicate. PID results will be compared and should be within a 20% difference range.

Additionally, to confirm the reliability of concentrations detected by the PID, the PID will at a minimum be calibrated at the beginning of each field day and checked at the end of the field day. The PID will be "bump checked" by taking a reading of the 100 ppm_v isobutylene gas every two hours of field screening or if VOC concentrations exceed 1,000 ppm_v to ensure the PID remains properly calibrated.

Additionally, field documentation will be reviewed to assure it is compliant with Credere SOP CA-1: Field Activity Documentation and methods used for field screening are compliant with the procedures herein.

4. SCREENING DOCUMENTATION

The following information should be recorded in the field logbook in compliance with Credere SOP CA-1:

- Date of sampling event
- Credere personnel
- Instrument calibration information
- Weather
- Scope of work/field screening target locations
- Description of screened media
- Screening method used
- Screening sample IDs, time of collection, location and depth of collection
- Modifier information such as odor, staining or oxidation/reduction
- QA/QC results
- Decontamination procedure (if applicable)

5. REFERENCES

Credere Associates, LLC, *Standard Operating Procedure CA-1 Field Activity Documentation*, Revision 0, dated August 4, 2015.

Maine Department of Environmental Protection, *Compendium of Field Testing of Soil Samples for Gasoline and Fuel Oil,* SOP No. TS004, Revision 2.1, dated October 15, 2012.

Massachusetts Department of Environmental Protection, *Commonwealth of Massachusetts Underground Storage Tank Closure Assessment Manual, Appendix A – Jar Headspace Analytical Screening Procedures*. DEP Policy #WSC-402-96, dated April 9, 1996.

New Hampshire Department of Environmental Services, *Jar Headsapce technique – Field Screening Soil Samples*, SOP No. HWRB-12, Revision 2, dated January 2012.

US DoD, *Environmental Field Sampling Handbook, Revision 1.0,* dated April 2013.

US Environmental Protection Agency, *Compendium of Superfund Field Operations Methods*, dated December 1987.

Vermont Department of Environmental Conservation, *Investigation and Remediation of Contaminated Properties Rule,* dated July 27, 2017.

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Standard Operating Procedure CA-8
Monitoring Well Installation

Effective Date: October 20, 2017
Revision: 0

Allison Drouin, Author — Date: 10/20/2017

Theresa Patten, Technical Review — Date: 10/20/2017

Judd Newcomb, Technical Review — Date: 10/20/2017

Revision	Date	Reason for Revision
1	10/20/2017	Amended well construction log.

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ATTACHMENT

1. OBJECTIVE AND APPLICABILITY

1.1 OBJECTIVE

The purpose of this Standard Operating Procedure (SOP) is to provide guidance for the installation of groundwater monitoring wells. If followed correctly this SOP will allow for the following:

- Safety of employees performing the sampling
- Locating of monitoring wells in strategic locations to meet project objectives
- Assessment of aquifer/groundwater characteristics
- Construction of wells that permit efficient collection of reproducible sampling conditions

Credere Associates, LLC (Credere) SOPs are a guidance, and state requirements, site-specific plans, and special conditions may require alternative approaches.

1.2 APPLICABILITY

This SOP should be used during the installation of standard groundwater monitoring wells in overburden or bedrock conditions. This SOP should serve as guidance for locating, selecting well materials and making field determinations of well construction details (e.g., screen depths, annulus material thicknesses, etc.).

The SOP does not cover installation of packers in wells, specific extraction well construction, FLUTe lined/Waterloo wells, or other Site specific innovative well features.

2. PROCEDURE

2.1 NECESSARY EQUIPMENT

Most equipment for well installation will be supplied by a subcontractor hired to perform drilling activities. The following is a list of required equipment:

- Subcontractor drilling equipment
- Well materials
- Well annulus materials
- Well completion materials (concrete, road box, stand pipe)
- Appropriate personal protection equipment (PPE)
- Photoionization detector (PID) (if applicable)
- Water level meter or interface probe (for NAPL Sites)
- Site plan
- Field logbook
- Ink pens
- Socket set and/or well keys
- Lock with lock keys (if stickups are installed)

2.2 TRAINING AND HEALTHY AND SAFETY

Credere employees must have received 40-hour Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) training pursuant to 29 CFR 1910.120 and be current on their 8-hour refresher. Employees must have 24 hours of training specifically in well installation procedures described herein and proven competence of the SOP in compliance with OSHA 29 CFR 1910.120(e)(4).

Employees will follow the Credere generic health and safety plan when implementing well installation procedures, and the site-specific HASP for the project.

2.3 WELL LOCATING

Well locating is highly site-specific. Additionally, well locations laid out in a work plan may require adjustment if field conditions present issues (e.g., bedrock encountered shallower than expected, new contamination encountered, etc.). Generally, well locations should be selected based on the project objectives for the well and to most efficiently assess overall project objectives. Objectives for a well may include:

- Plume delineation through groundwater sampling
- Hydraulic testing
- Water level monitoring

Well locations may target hydraulically up- or downgradient position, greatest areas of contamination, the extents of contamination, potential migration pathways, assessment of certain

receptors, spacial distribution across a site, or certain geologic units. These factors may be combined to best locate a well to achieve more than one objective with the same well.

Screen interval and target depths may be based on the depth to groundwater, specific geologic features, or other factors. These screen intervals may target a distinct bedrock fracture or thin overburden seam, and attention to fine details is important when installing wells with certain target depth objectives.

Before selecting well locations, a comprehensive review of any prior environmental data, local geology, hydrology, topography, and chemical characteristics of the subsurface is advantageous.

2.4 WELL INSTALLATION METHOD

Monitoring wells are typically installed within boreholes drilled using direct push probes, hollow-stem auger, sonic drilling, or air hammer. Drive and wash and mud rotary drilling for installation of monitoring wells are not recommended due to introduction of outside fluids into the aquifer, which impacts chemical and hydraulic conditions surrounding the well.

Well Installation Preparation

1. Upon arrival at the Site, begin a field logbook entry recording the Credere staff, scope of work for the day, and weather conditions. Also record the drilling contractor, foreman, drill rig make and model, and tooling details.
2. Calibrate PID according to product specifications if applicable. Keep record of calibration in the field logbook including PID make and model, serial number, calibration gas and concentration, and span gas check.
3. Locate wells in accordance with the prepared work plan or project objectives. Note any adjustments to well locations based on field conditions in the log book.
4. Begin drilling to target depth using standard techniques for the respective drilling method.
5. During drilling, determine the depth of the apparent water table as the depth of wet soil. Wet soil is defined as visible water. Use caution to prematurely identify the water table in area where groundwater may be perched (e.g., silt or clay aquifers) or to miss the water table in certain fine grained soils. If the water table is not obvious, it may be helpful to leave the borehole open for a period of 10 to 20 minutes and measure the depth to water using a water level meter in the open borehole.
6. If installing bedrock wells, determine the depth to bedrock noting weathered bedrock thicknesses if present, depth of prominent fractures, and changes in dominant rock types.

Well Materials

Once the borehole has been drilled, well materials will be selected based on the observed geology and know chemical conditions of the site groundwater. Well materials must also be selected to meet regulatory requirements, data quality objectives and accommodate required downhole equipment.

Casing, Riser and Screen

Polyvinyl Chloride (PVC) is the typical well construction material for risers and screens. PVC is generally inert and does not react with most common contaminants. PVC has shown evidence of absorption and desorption of organics; however, sufficient purging of a well using low flow methods minimizes effects from this issue by drawing in fresh groundwater. Schedule 40 is the standard PVC thickness; however, schedule 80 is available for deeper wells requiring more structural stability. Stainless steel materials are more expensive and are impacted by acidity; but are more structurally durable in deep wells exceeding 300 feet.

In New Hampshire and Massachusetts wells should be installed with a minimum 2-inch inner diameter (ID). Maine permits installation of 1-inch ID monitoring wells for preliminary assessment; however, 2-inch diameter wells should be considered or may be required for Site with the potential to initiate long term monitoring.

The well screen in conjunction with the surrounding filter pack is intended to filter out sediment that may flow into the well with groundwater. Varying sand and slot size pairings are available for site-specific factors. The following should be considered when selecting sand and slot size:

- Native soil type: native soil should be courser than the filter pack and slot opening
- Sand pack size: the slot size should be smaller than the selected grain size for sand pack
- Types of contaminant: slot size should not inhibit flow of product (e.g., creosote and No. 6 fuel oil) into the well thereby clogging the screen

Typical well screens are PVC slotted 10 or 20 slot with 0.010 or 0.020 inch openings, respectively; however, if fine grained native soils are present, 5 slot screen may be considered.

Annulus

The well annulus consists of a filter pack surrounding the screen, a seal (typically bentonite), backfill or grouting to the surface, and well finishing at the surface (e.g., road box and concrete pad or collar, or stand pipe).

The filter pack typically consists of washed sand, graded silica sand, or glass beads to prevent collapsing of the native materials, provide fines filtration of the water entering the well, and prevent screening clogging. The most commonly used filter pack is graded #2 silica sand, but finer #1 and #0 silica sands are also available for fine grained native soils such as silt and clay.

Seals are used to prevent migration of surface water through the disturbed borehole. Seals are typically installed above screens, where casing enters bedrock, or between nested well depths to prevent vertical migration through the borehole within the aquifer. Typical sealant materials include cement, hydrated bentonite chips, or a grout of percentages of both. In deep wells, grout may need to be tremied (i.e., pumped to depth with a pipe) to the desired depth to avoid blocking of the borehole shallower than desired.

Types of Wells

Piezometer

Piezometers are installed to measure the water level or head of a particular section of an aquifer. Piezometers may be installed across the water table to strictly measure the depth to water at lower cost that a complete monitoring well, or they may be installed to a target depth within an aquifer to monitor hydraulic changes of conditions relative to other areas of an aquifer. Piezometers are typically 0.5 to 1-inch ID. **Piezometers are not intended to be used for collection of environmental samples**. Piezometers should be installed according to the following steps:

1. Determine the piezometer's screen target depth. If the piezometer is to be installed to a target depth, simply install the piezometer to the predetermined depth. If the piezometer is being installed at the water table, the screen should straddle the water table such that the water table is within the screened interval during all seasons. A good rule of thumb is to have 5 feet of water within a screen during the winter, 6 feet of water within the screen during the summer and fall, and 7 feet of water within the screening during the spring.
2. Place the screen at the desired depth. Screen length can vary from a standard 10 foot screen or can be modified to screen only a specific target interval. Add enough riser to reach the surface. Well pieces should be threaded connection and should at no time be installed with solvent based cements.
3. Fill the annulus with an appropriate filter pack and install a 2-foot thick bentonite seal 1 foot above the top of the screened interval.
4. Backfill the borehole to the surface and finish the piezometer at the surface as a temporary measuring point (bentonite seal at the surface), with a road box, or standpipe.
5. Record piezometer construction details in the field logbook.

Overburden Wells

Overburden wells are installed across the water table to assess hydraulic and environmental conditions in groundwater most likely to be encountered by humans. Overburden wells are useful during preliminary site assessments and to assess light non-aqueous phase liquid (LNAPL) concentration and migration that may "float" on the water table surface. Overburden wells should be installed according to the following steps:

1. Determine the depth to the groundwater table and the screen depth such that the water table is within the screened interval during all seasons. A good rule of thumb is to have 5 feet of water within a screen during the winter, 6 feet of water within the screen during the summer and fall, and 7 feet of water within the screening during the spring.
2. Place the screen at the desired depth. Screen length can vary from a standard 10-foot screen or can be modified if the saturated thickness or drilling refusal are encountered shallower than will permit installation of a 10-foot screen. Add enough riser to reach the surface. Well pieces should have threaded connections with O-rings and should at no time be installed with solvent based cements.

3. Fill the annulus with an appropriate filter pack and install a 2-foot thick bentonite seal 1 foot above the top of the screened interval.
4. Backfill the borehole to the surface and finish the well at the surface with a road box and concrete pad or collar, or standpipe.
5. Record well construction details in the field logbook.

Deep Overburden Wells

Deep overburden wells are installed in areas of thick overburden where differing surficial geology may warrant assessment of different depths or preferential pathways (e.g., confining clay layers, sand seams, or separate aquifers). Deep overburden wells are useful during more detailed Site assessment coupled with a series of shallower wells or for assessment of dense non-aqueous phase liquid (DNAPL) that may have sunk and be perched on bedrock or certain confining layers (e.g., any soil with a lower ability to flow than overlying soil [sand to silt, silt to clay]). Deep overburden wells should be installed according to the following steps:

1. Determine the target screen interval based on observed geology or nature of contamination. Since deeper wells are screened below the water table, the entire screen will be within the saturated zone.
2. Place the screen at the desired depth. Screen length can vary from a standard 10 foot screen or can be modified to target certain intervals. Add enough riser to reach the surface. Well pieces should have threaded connections with O-rings and should at no time be installed with solvent based cements.
3. Fill the annulus with an appropriate filter pack and install a 2-foot thick bentonite seal 1 foot above the top of the screened interval. If the deeper wells are installed in combination with a shallower/deeper series of wells (i.e., nested wells) the bentonite seal above each screen should be carefully installed to ensure an adequate seal between target zones. In a series, the deepest well should be installed first.
4. Backfill the borehole to the surface and finish the well at the surface with a road box or standpipe. If a series of nested wells were installed in a borehole, be sure to label the tops of each well as to which riser is for which well.
5. Record well construction details in the field logbook.

Bedrock Wells

Bedrock wells are installed into bedrock to assess hydraulic and environmental conditions deeper than in overburden. Bedrock wells can be installed with screens or be an open hole well. Open hole wells are sufficient when the bedrock aquifer in general is targeted. If bedrock is incompetent (i.e., weathered or highly fractured) a screen may be required to maintain the structure of the borehole for sampling. If a particular water bearing fracture or zone is targeted these areas can be screened with PVC wells and the remainder of the borehole sealed off. (Details on alternative methods of sealing certain zones in bedrock wells are not included in this SOP). Bedrock wells should be installed according to the following steps:

1. Perform drilling through overburden until bedrock is encountered.

2. Set enough steel casing driven sufficiently into the bedrock surface and to reach the ground surface. Proper setting of casing is essential to prevent leakage of overburden or surface water into the bedrock aquifer. If a weathered surface is present at the top of bedrock, it should be attempted to drill through and loosen the weathered material prior to setting casing in an effort to set the casing into competent bedrock.
3. Continue drilling through bedrock to the desired depth noting water bearing fractures and rock quality while drilling. Drill to the desired well completion depth.
4. If the well is to be an open hole well, tremie grout the casing and bedrock interface and to the surface.
5. If the well is to be a screened/multi-screened well, place the screen at the desired intake depth and place a seal above the screen. If the screened wells are installed in combination with a shallower/deeper series of wells (i.e., nested wells) the bentonite seal above each screen should be carefully installed to ensure an adequate seal between target zones (Packers, etc. may also be used to seal off certain zones). In a series, the deepest well should be installed first.
6. Fill the well annulus to the surface and finish the well at the surface with a road box or stand pipe.
7. Record well construction details in the field logbook.

Post Installation Activities

1. After construction of the well, the well should be allowed to set for a period sufficient to allow for setting of annulus materials, particularly hydrated bentonite seals and surface finishing materials.
2. After the materials have set, the well should be developed in accordance with Credere SOP CA-9: Monitoring Well Development.
3. After installation of monitoring wells, a relative elevation survey should be completed to determine to relative elevation of the ground surface at a well and the top of the riser or casing to be used as a water level reference point during future monitoring/sampling activities. The relative elevation survey should be conducted in accordance with Credere SOP CA-25: Relative Elevation Surveys.

3. QUALITY ASSURANCE/QUALITY CONTROL

Quality assurance (QA) and quality control (QC) will be obtained through strict adherence to work plans, appropriate communication with project managers, and proper field documentation.

Field documentation will be reviewed to assure it is compliant with Credere SOP CA-1: Field Activity Documentation and methods used for well installation are compliant with the protocol herein.

4. FIELD DOCUMENTATION

The following information should be recorded in the field logbook in compliance with Credere SOP CA-1:

- Date of field activity
- Credere personnel
- Scope of work
- Weather
- Contractor/foreman/equipment make and model
- Changes in scope
- Correspondence with Project Managers
- Well materials
- Well construction details (depths of installation, thickness, etc.)
- General timeline of field activities
- Well location measurements (i.e., swing ties from permanent site features)

5. REFERENCES

ASTM D 5092-04 (2010) *Standard Practice for Design and Installation of Groundwater Monitoring Wells*, ASTM International, West Conshohocken, PA, 2010, www.astm.org

ASTM D 5979-96 (2014) *Guide for Conceptualization and Characterization of Groundwater Systems*, ASTM International, West Conshohocken, PA, 2014, www.astm.org

ASTM D 6724-04 (2010) *Guide for Installation of Direct-Push Groundwater Monitoring Wells*, ASTM International, West Conshohocken, PA 2010, www.astm.org

Credere Associates, LLC, *Standard Operating Procedure CA-1 Field Activity Documentation*, Revision 0, dated August 4, 2015.

Credere Associates, LLC, *Standard Operating Procedure CA-9 Monitoring Well Development*, Draft, dated TBD.

Credere Associates, LLC, *Standard Operating Procedure CA-25 Relative Elevation Survey*, Draft, dated TBD.

Commonwealth of Massachusetts Department of Environmental Protection, *Standard References for Monitoring Wells*, WSC-310-91, dated January 1991.

U.S. DoD, *Environmental Field Sampling Handbook, Revision 1.0*, dated April 2013.

U.S. Environmental Protection Agency, *Compendium of Superfund Field Operations Methods*, dated December 1987.

U.S. Environmental Protection Agency, *Monitoring Well Installation*, SOP #2048, Revision 0, dated March 18, 1996.

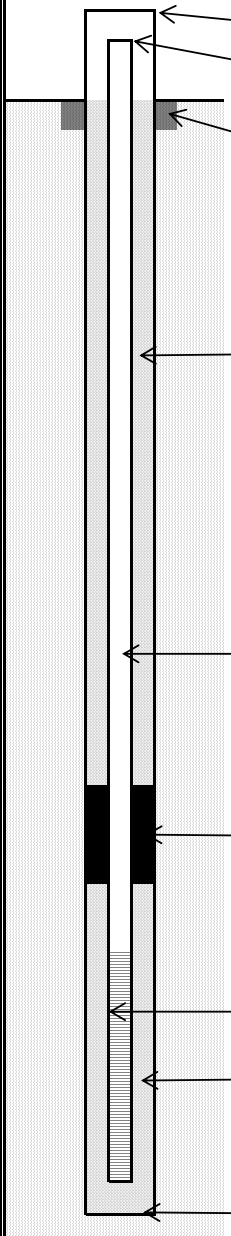
U.S. Environmental Protection Agency, Region 4, Science and Ecosystem Support Division, Guidance, *Design and Installation of Monitoring Wells*, SESDGUID-101-R1, dated January 29, 2013.

Credere Associates LLC

Boring/Well ID:

SITE INFORMATION	DRILLING EQUIPMENT
Project Number/Client:	**Equipment (make/model):**
Site Location:	**Casing/Auger/Core Diameter:**
Date Start/Finish:	**Casing Material:**
Credere Representative:	**CONTRACTOR**
	Drilling Contractor/Foreman:

OVERBURDEN WELL CONSTRUCTION DETAILS



Top of stickup/roadbox elevation: _____
Stickup height (in): _____
Top of well/PVC elevation: _____
Ground elevation: _____
Concrete thickness (if roadbox; ft): _____

Borehole diamter (in): _____

Backfill material: _____
Backfill depth (ft): _____

Well riser material: _____
Well riser length (ft): _____
Well riser diameter (in): _____

Bentonite depth interval (ft): _____

Screen material: _____
Well diameter (in): _____
Screen length: _____
Annulus material: _____
Annulus depth interval (ft): _____
Depth to water (ft bgs): _____

Depth of borehole (ft): _____

Well completion: stickup roadbox temporary
Lock installed? Yes No

Generalized description of overburden: _____

Development method: _____
Total well volume: _____
Total purge volume (g): _____
No. of well volumes purged: _____
Final turbidity (NTU): _____

Field Parameters

DO (mg/L): _____
pH: _____
Sp. Conductivity (mS/cm): _____
ORP (mV): _____
Temperature (°C): _____

Annulus material size: #00 #1 #2 G

Remarks and Well Details

Well ID:

CREDERE ASSOCIATES, LLC
776 Main Street
Westbrook, Maine 04092
Phone: 207-828-1272
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Standard Operating Procedure CA-9
Well Development

Effective Date: October 20, 2017
Revision: 1

Allison Drouin 10/20/2017
Allison Drouin, Author Date

Theresa Patten 10/20/2017
Theresa Patten, Technical Review Date

Judd R. Newcomb 10/20/2017
Judd Newcomb, Technical Review Date

Revision	Date	Reason for Revision
1	10/20/2017	Clarification on limitations of pumps for certain well types.

TABLE OF CONTENTS

1. OBJECTIVE AND APPLICABILITY

1.1 OBJECTIVE

The purpose of this Standard Operating Procedure (SOP) is to provide guidance for well development. If followed correctly this SOP will allow for the following:

- Safety of employees performing the sampling
- Collection of a sample representative of the target groundwater aquifer
- Shorten groundwater purging times during low-flow sampling

Credere Associates, LLC (Credere) SOPs are a guidance, and state requirements, site-specific plans, and special conditions may require alternative approaches.

1.2 APPLICABILITY

This SOP should be used during well development of monitoring wells installed in accordance with Credere SOP CA-8: Groundwater Monitoring Well Installation.

2. PROCEDURE

2.1 NECESSARY EQUIPMENT

The following is a list of required equipment for groundwater sampling:

- Appropriate personal protection equipment (PPE)
- Photoionization detector (PID) (if applicable)
- Water level meter or interface probe (for NAPL Sites)
- Peristaltic pump and associated tubing (if developing 1 inch wells less than 25 feet deep)
    - Backup batteries
    - 1 foot silicone tubing per well
    - Polyethylene or Teflon lined (if VOCs are a COC) 3/8 inch ID tubing
- Submersible/centrifugal pump (if developing 2 inch or larger wells selected from below table)
    - Marine battery or generator
    - Polyethylene or PVC tubing appropriately sized for pump
- Appropriate drilling subcontractor equipment for mechanical surge development (if developing deep or bedrock wells
- Multi-parameter water quality meter (if required by work plan)
- Turbidity meter
- Buckets
- Drums (if purge water requires containerization)
- Site plan
- Field logbook
- Ink pens
- Socket set and/or well keys
- Metal detector (for locating wells)
- Tubing cutter
- Large screw driver (for prying)
- Lock keys (if stickups are locked)
- Paper towels

Pump	Depth Limitation	Power Source	Other limitations
Whale Pump (single stage)	35 feet	Marine battery	Low to moderately high turbidity
Whale Pump (two stage)	60 feet	Marine battery	
Grunfos	300	Generator	
Waterra/check valve	Any depth	Generator	Very high turbidity and mud
Bailer	Any	None	Time consuming, last resort

2.2 TRAINING AND HEALTHY AND SAFETY

Credere employees must have received 40-hour Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) training pursuant to 29 CFR 1910.120 and be current on their 8-hour refresher. Employees must have 24

hours of training specifically in well development procedures described herein and proven competence of the SOP in compliance with OSHA 29 CFR 1910.120(e)(4).

Employees will follow the Credere generic health and safety plan when implementing well development procedures and the site-specific HASP for the project.

2.3 WELL DEVELOPMENT METHOD

When developing wells the general objective is to remove any accumulated fine sediment from the well bottom and the screened portion of the well annulus, mechanically rearrange the sand pack to improve filtration, begin drawing in fresh groundwater, and improve the hydraulic connectivity of the well with the aquifer after the disruption of the well installation.

When developing 1-inch wells over-pumping and agitation is implemented using a peristaltic pump. If the well is to be sampled with a peristaltic pump after development, tubing should be selected such that it is appropriate for collection of samples for the planned analyses (i.e., Teflon lined for VOC/VPH wells and polyethylene for other analyses only) so the tubing can be left in the well as dedicated tubing. When developing shallow (less than 40 feet below ground surface) 2 inch or larger wells, a submersible/centrifugal pump is used for over-pumping with large diameter tubing that cannot be left in the well for later sampling. A submersible whale pump is sufficient for 2 inch wells; however, a higher volume Grundfos or similar make may be needed for larger wells. Deep or bedrock well development may be facilitated by the drilling subcontractor through use of mechanical surge development that reduces the need for long lengths of tubing and expensive high power pumps. This type of development uses a surge block that forces water out of the well screen and then draws the water back through using a piston type mechanical motion.

Wells should be permitted to stabilize after development for a minimum of 14 days (7 days is allowable by the Maine DEP) prior to sampling; however, wells not intended for sampling such as piezometers may not require development.

A general idea of the level of contamination in a monitoring well or location should be obtained prior to development to properly manage purge water. If groundwater is known to be contaminated from previous investigations, purge water will be containerized for treatment or disposal. If no previous investigation has been conducted, groundwater will be assumed suitable for discharge to the Site's surface unless obvious evidence of contamination is observed such as measurable non-aqueous phase liquid (NAPL), elevated PID headspace readings, water sheens, or odor.

Development Preparation

1. Upon arrival at the Site, begin a field logbook entry recording the Credere staff, scope of work for the day, and weather conditions.
2. Calibrate field instruments according to Credere SOP CA-11 and product specifications. Keep record of calibration in the field logbook.

3. Using the appropriate socket or well key, open the well making sure to place the bolts/lock in a safe location away from the well mouth. Remove the expansion plug. (Note: temporary wells will not have a road box or standpipe)
4. If volatiles are a contaminant of concern (COC) and concentrations are unknown, or concentrations are known to be at concentrations that may volatilize and create an atmospheric hazard, the well mouth should be screened using a PID for health and safety concerns and/or evidence of contamination in groundwater that may require containerization of purge water. In the case of groundwater or vapor extraction system installation at a site, VOC measurements in the well mouth may also prove useful data.
5. Using the water level meter or interface probe, measure the depth to water and depth to bottom to the nearest 0.01 and in accordance with Credere SOP CA-10. Calculate the length of the water column by subtracting the depth to water (DTW) from the depth to bottom (DTB).

   Length of Water Column = DTB - DTW
6. Calculate the total well volume according to the following:

   Well Volume = gal/foot from table below x length of water column (feet)

Well Diameter	Gallons of water per foot
1 inch	0.04
2 inch	0.16
4 inch	0.65
6 inch	1.47

7. Calculate minimum gallons to purge according to the following

   Minimum gallons to purge = well volume x 3

Purging

Peristaltic Development by Agitation and Over-pumping

Peristaltic development will be used in 1 inch wells.

1. Install the pump tubing into the well approximately 6 inches from the bottom. Connect the tubing to the peristaltic pump and begin pumping at the pump's maximum speed. Collect purge water into a bucket.
2. Agitate the water in the well by vigorously moving the tubing and gradually lowering the tubing to the bottom of the well to agitate any accumulated sediment. Avoid lowering too quickly, which may clog the tubing. Lift tubing to approximately 6 inches from the bottom and allow sediment to visually clear from purge water.
3. Agitate again surging the tubing up through the entire water column several times to draw sediment in from the filter pack. Lift tubing to approximately 6 inches from the bottom and allow sediment to visually clear from purge water. On subsequent agitations, lift the tubing further up in the water column to purge the sand pack (e.g., 1.5 feet from bottom, 2.5 feet from bottom).

4. Continue agitation alternating between steps 2 and 3 listed above until agitation no longer produces significant visible sediment in the purge water.
5. Once agitation produces visibly less sediment or purge water appears clear, continue to purge and begin monitoring turbidity in the well until turbidity is reduced to below 10 nephalometric turbidity units (NTUs). Record the series of turbidity readings in the field notes.
6. If evidence of contamination is identified in groundwater (e.g., odor, sheen, measurable NAPL), obtain one sample for jar headspace screening to assess volatile concentrations. Screen the groundwater jar headspace sample in accordance with Credere SOP CA-7: Headspace Field Screening and compare the results to the 100 parts per million by volume (ppm_v) threshold for containerization.

Submersible/Centrifugal Development

Submersible/centrifugal development will be used when developing 2 inch or larger diameter wells. The appropriate pump to be used should be selected based on planned well depth and expected turbidity. **A backup bailer should always be available as a last resort for development if pumps fail.**

1. Install the submersible/centrifugal pump into the well approximately 6 inches from the bottom with enough tubing to reach the purge bucket. Connect the power cord to the appropriate positive or negative terminal and the pump will immediately begin purging. Collect purge water into a bucket.
2. Agitate the water in the well by vigorously moving the pump and gradually lowering the pump to the bottom of the well to agitate any accumulated sediment. Avoid lowering too quickly into bottom sediment, which may clog the pump. Lift the pump to approximately 6 inches from the bottom and allow sediment to visually clear from purge water. On subsequent agitations, lift the pump further up in the water column to purge the sand pack (e.g., 1.5 feet from bottom, 2.5 feet from bottom).
3. Agitate again surging the tubing up through the entire water column several times to draw sediment in from the filter pack. Lift tubing to approximately 6 inches from the bottom and allow sediment to visually clear from purge water.
4. Continue agitation alternating between steps 2 and 3 above until agitation no longer produces significant sediment increases in the purge water.
5. Once agitation produces less sediment, continue to purge and begin monitoring turbidity in the well until turbidity is reduced to below 10 NTUs.
6. If evidence of contamination is identified in groundwater (e.g., odor, sheen, measurable NAPL), obtain one sample for jar headspace screening to assess volatile concentrations. Screen the groundwater jar headspace sample in accordance with Credere SOP CA-7: Headspace Field Screening and compare the results to the 100 ppm_v threshold for containerization.

7. Thoroughly decontaminate the pump prior to use at the next well by decontaminating the exterior and running potable water through the interior.

Mechanical Surging

Mechanical surging is used to develop deep or bedrock wells that cannot be efficiently developed with a submersible/centrifugal setup. Mechanical surging can also be used to improve the connectivity of the formation with the well as the reversing flow of water in two directions prevents sand bridging. This type of development is completed primarily by the drilling subcontractor using drill rig equipment attachments; however, the following steps should be observed and coordinated with the driller to prevent well damage and successfully develop a well.

1. Pre-clean the well by instructing the driller to direct purge any accumulated sediment in the well. This will prevent surging of fines out into the sandpack once the surge action begins.
2. Determine the soil type surrounding the well screen and filter pack. If a fine grained formation is present, begin surging action at a slow pace. Too rapid surging in a fine formation can result in screen collapse due to substantial pressure building and may also result in washing out of the formation outside the filter pack resulting in filter pack collapse. Sand and coarse grain formation should also be surged at a moderate pace; however, should be controlled based on the formation yield.
3. Develop the screened interval at lengths equal to the length of the surge block considering the surrounding soil type at each interval in case of a mid-screen change. Begin just above the top of the well screen.
4. After surging the entire well screen, begin monitoring turbidity in the well until turbidity is reduced to below 10 NTUs.
5. If evidence of contamination is identified in groundwater (e.g., odor, sheen, measurable NAPL), obtain one sample for jar headspace screening to assess volatile concentrations. Screen the groundwater jar headspace sample in accordance with Credere SOP CA-7: Headspace Field Screening and compare the results to the 100 ppm_v threshold for containerization.
6. Ensure thorough decontamination of the surge block and rods prior to use in the next well.

Post Development

1. Determine the total purge volume in gallons by estimating the fullness of the 5 gallon bucket/drum and number of buckets filled. A graduated bucket is useful in estimating this volume.
2. If groundwater is not known to be contaminated and showed no evidence of contamination, discharge purge water to a permeable ground location upgradient of the well. If evidence of contamination resulted in field screening with results that exceeded

100 ppm_v or if groundwater is known to be contaminated, containerize groundwater for treatment or offsite disposal.

8. Disassemble the development setup.
9. Insert the expansion plug into the well mouth, and replace the well cap and bolts or lock. If the well is completed with a flush mounted road box, ensure the bolts are tightly screwed to minimize potential damage to the well (particularly during the winter months from snow plows).

3. QUALITY ASSURANCE/QUALITY CONTROL

Quality assurance (QA) and quality control (QC) will be obtained through daily self-review of field notes for completion. Field documentation will be reviewed to assure it is compliant with Credere SOP CA-1: Field Activity Documentation and methods used for well redevelopment are compliant with the protocol herein.

4. DEVELOPMENT DOCUMENTATION

The following information should be recorded in the field logbook in compliance with Credere SOP CA-1:

- Date of well development
- Credere personnel
- Scope of work
- Weather
- Type of development/equipment used
- Type of dedicated tubing left in well (if applicable)
- Well volume calculations
- Total purge volumes
- NTU readings
- Site conditions
- Decontamination procedure (if applicable)

5. REFERENCES

ASTM D 5521-13 *Guide for Development of Groundwater Monitoring Wells in Granular Aquifers,* ASTM International, West Conshohocken, PA, 2013, www.astm.org

Credere Associates, LLC, *Standard Operating Procedure CA-1 Field Activity Documentation*, Revision 0, dated August 4, 2015.

Credere Associates, LLC, *Standard Operating Procedure CA-7 Headspace Field Screening*, Draft, dated TBD.

Credere Associates, LLC, *Standard Operating Procedure CA-8 Monitoring Well Installation*, Draft, dated TBD.

Credere Associates, LLC, *Standard Operating Procedure CA-10 Monitoring Well Gauging*, Draft, dated TBD.

Credere Associates, LLC, *Standard Operating Procedure CA-11 Water Quality Field Instrument Calibration*, Draft, dated TBD.

Commonwealth of Massachusetts Department of Environmental Protection, *Standard References for Monitoring Wells*, WSC-310-91, dated January 1991.

US DoD, *Environmental Field Sampling Handbook, Revision 1.0,* dated April 2013.

US Environmental Protection Agency, *Compendium of Superfund Field Operations Methods*, dated December 1987.

US Environmental Protection Agency, *Monitor Well Installation*, SOP# 2048, dated March, 18 1996.

U.S. Environmental Protection Agency, Region 4, Science and Ecosystem Support Division, Guidance, *Design and Installation of Monitoring Wells*, SESDGUID-101-R1, dated January 29, 2013.

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Standard Operating Procedure CA-10
Monitoring Well Gauging

Effective Date: August 29, 2016
Revision: 0

Allison Drouin, Author — 8/29/2016 Date

Theresa Patten, Technical Review — 8/29/2016 Date

Judd Newcomb, Technical Review — 8/29/2016 Date

Revision	Date	Reason for Revision

TABLE OF CONTENTS

1. OBJECTIVE AND APPLICABILITY

1.1 OBJECTIVE

The purpose of this Standard Operating Procedure (SOP) is to provide guidance for the collection of groundwater level data. If followed correctly this SOP will allow for the following:

- Safety of employees performing the measurements
- Accurate measurement of groundwater levels and product thicknesses
- Assessment of groundwater elevations and contours

Credere Associates, LLC (Credere) SOPs are a guidance and state requirements, site-specific plans, and special conditions may require alternative approaches.

1.2 APPLICABILITY

This SOP should be used during gauging of groundwater levels and product thicknesses in monitoring wells installed in accordance with Credere SOP CA-8: Groundwater Monitoring Well Installation and developed in accordance with Credere SOP CA-9: Groundwater Monitoring Well Development.

2. PROCEDURE

2.1 NECESSARY EQUIPMENT

The following is a list of required equipment for groundwater sampling:

- Appropriate personal protection equipment (PPE)
- Water level meter or interface probe (for NAPL Sites)
- Photoionization detector (PID), if needed
- Site plan
- Field logbook
- Ink pens
- Socket set and/or well keys (power drill with socket attachment may be useful at Sites with many wells to be gauged)
- Metal detector (for locating wells)
- Large screw driver (for prying)
- Lock keys (if stickups are locked)
- Decontamination fluids
- Paper towels
- Torch (winter freezing conditions)

2.2 TRAINING AND HEALTHY AND SAFETY

Credere employees must have received 40-hour Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) training pursuant to 29 CFR 1910.120 and be current on their 8-hour refresher. Employees must have 24 hours of training specifically in groundwater measurement procedures described herein and proven competence of the SOP in compliance with OSHA 29 CFR 1910.120(e)(4).

Employees will follow the Credere generic health and safety plan and the site-specific HASP when implementing groundwater gauging procedures.

2.3 METHOD

Groundwater monitoring wells should be measured to nearest hundredth of a foot (0.01) to provide sufficient detail in changing water levels or product thicknesses over time. Measurement equipment should be selected based on well construction details and potential for product. Equipment details to considered include appropriate tape length, indicator diameter, interface or water level probe, etc.

When completing gauging on multiple wells in conjunction with a groundwater sampling program, gauging should be completed at the beginning of the program in a single day if possible to best correlate the water levels for hydraulic interpretation. Water levels can fluctuate day to day based on precipitation, atmospheric pressure, and tidal influence in certain cases. Wells should be gauged in order of increasing contamination.

Gauging Methodology

1. Upon arrival at the Site, begin a field logbook entry recording the Credere staff, scope of work for the day, and weather conditions in accordance with Credere SOP CA-1. Set up a table in the field logbook similar to the following:

Time	Well ID	Depth to Water (feet)	Depth to Product (feet)	Depth to Bottom (feet)	Notes

2. Using the appropriate socket or well key, open the road box or protective standpipe being sure to place the bolts/lock in a safe location away from the well mouth. It may be necessary to pry open the well using the large screw driver or melt away excessive ice using the torch. Remove the expansion plug. Record the physical condition and construction materials on the field log, and note any other pertinent observations such as odors, PID results of the well mouth (if applicable), frost heaving, damage to the monitoring well or protective system, etc.
3. If volatiles are a contaminant of concern (COC), the well mouth should be screened using a PID if required in the Site-Specific Health and Safety Plan.
4. Identify the well's measuring point. The measuring point should be marked on the riser or will be slightly angled with the highest point being the measuring point. If no measuring point is marked and there is no clear highest point, measure at any point around the riser and designate the location with an indelible marker for future gauging events.
5. Lower the water level or interface probe into the well listening for the unit's distinctive indicator tone(s). If measuring for separate phase liquid (light or dense non-aqueous phase liquid [LNAPL or DNAPL]), the interface probe will provide two distinctly different tones for water and NAPL (e.g., solid vs. intermittent tones). **Note: LNAPL will be measured at the top of the water column and DNAPL will be measured at the bottom of the water column/well**. Record the depth to the first encountered liquid (water or LNAPL) as indicated by the tone to the nearest 0.01 foot. If LNAPL is encountered advance the probe until the tone indicates a transition to water. These measurements will indicate LNAPL thickness.
6. Lower the probe to the bottom of the well. It is helpful to know the general depth of the well prior to gauging to know if the probe is approximately at the bottom or on an obstruction. The bottom of the well measurement will require feeling through the tape when the bottom has been reached. When the tape no longer advances, lift the tape and gently bounce the probe on the bottom of the well to feel the bottom while also pulling the tape taught up the well. Record the depth to bottom to the nearest 0.01 foot. If measuring for DNAPL listen for the interface probe's solid tone then lift the probe up through the DNAPL until the tone indicates a transition to water (i.e., a beeping tone). The depth to bottom and depth to product measurements will indicate DNAPL thickness.
7. As the water level or interface probe tape is removed from the well, appropriately decontaminate the tape using a decontamination fluid wetted rag. If gross contamination

is present on the tape (e.g., coal tar or heavy oil), more aggressive decontamination may be required. It is important to decontaminate the tape as it is rolled from the well to avoid contact with the ground surface. Decontaminate the probe and thoroughly clean the probe tip prior to use at the next well.

8. Insert the expansion plug into the well mouth, and replace the well cap and bolts or lock. If the well is completed with a flush mounted road box, ensure the bolts are tightly screwed to minimize potential damage to the well (particularly during the winter months from snow plows).

Post Gauging Activities

After collection of field data, gauging measurements will be tabulated into a table of historical (if applicable) water levels for calculation of groundwater elevations and product thicknesses according to the following:

1. Calculate the groundwater elevation by subtracting the depth to water from the measuring point elevation (usually the top of riser elevation)
2. Calculate the product thickness by:
    a. Subtracting the depth to product from the depth to water for LNAPL; or
    b. Subtracting the depth to product from the depth to bottom for DNAPL.

The following table is an example tabulation table:

Well ID	Gauging Date	Elevation (top of riser)	Depth to Water (feet)	Depth to Product (feet)	Depth to Bottom (feet)	Product Thickness (feet)	Groundwater Elevation (ft amsl)
MW-1	1/1/2016	100.00	2.50	2.00	11.60	0.50 (L)	97.50
	2/1/2016		2.55	1.98	11.40	0.57(L)	97.45
MW-2	1/1/2016	101.00	3.00	11.75	12.00	0.25 (D)	98.00
	2/1/2016		3.10	11.65	12.00	0.35 (D)	97.90
MW-3	1/1/2016	98.00	2.00	NM	13.00	None	96.00
	2/1/2016		1.80	1.78	13.00	0.02 (L)	96.20

(L)/(D) – indicates LNAPL (L) or DNAPL (D)
NM – not measured

3. QUALITY ASSURANCE/QUALITY CONTROL

Quality assurance (QA) and quality control (QC) will be obtained through daily self-review of field notes for completion. Field documentation will be reviewed to assure it is compliant with Credere SOP CA-1: Field Activity Documentation and methods used for water level and product measurement are compliant with the protocol herein.

4. DOCUMENTATION

The following information should be recorded in the field logbook in compliance with Credere SOP CA-1:

- Date of gauging event
- Credere personnel
- Scope of work
- Weather
- Gauging data
- Site conditions
- Decontamination procedure (if applicable)

5. REFERENCES

Commonwealth of Massachusetts Department of Environmental Protection, *Standard References for Monitoring Wells*, WSC-310-91, dated January 1991.

Credere Associates, LLC, *Standard Operating Procedure CA-1 Field Activity Documentation*, Revision 0, dated August 4, 2015.

Credere Associates, LLC, *Standard Operating Procedure CA-8 Monitoring Well Installation*, Draft, dated TBD.

Credere Associates, LLC, *Standard Operating Procedure CA-9 Monitoring Well Development*, Draft, dated TBD.

New Hampshire Department of Environmental Services, *Water Level Measurement,* SOP No. HWRB-1, Revision 2, dated December 2011.

US DoD, *Environmental Field Sampling Handbook, Revision 1.0,* dated April 2013.

US Environmental Protection Agency, *Compendium of Superfund Field Operations Methods*, dated December 1987.

US Environmental Protection Agency Environmental Response Team, *Manual Water Level Measurements*, SOP #2043, dated February 11, 2010.

US Environmental Protection Agency, *Low Stress (low flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells*. EQASOP-GW-001, Revision 3, dated January 19, 2010.

U.S. Environmental Protection Agency, Region 4, Science and Ecosystem Support Division, Operating Procedure, *Groundwater Level and Well Depth Measurements,* SESDPROC-105-R2, dated January 29, 2013.

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Standard Operating Procedure CA-11
Water Quality Field Instrument Calibration

Effective Date: September 18, 2017
Revision: 0

Allison Drouin, Author — 9/18/2017 Date

Theresa Patten, Technical Review — 9/18/2017 Date

Judd Newcomb, Technical Review — 9/18/2017 Date

Revision	Date	Reason for Revision

TABLE OF CONTENTS

ATTACHMENTS

1. OBJECTIVE AND APPLICABILITY

1.1 OBJECTIVE

The purpose of this Standard Operating Procedure (SOP) is to provide guidance for the collection of groundwater samples. If followed correctly, this SOP will allow for the following:

- Document performance objectives are met for equipment used during data collection/sampling
- Collection of sample representative of the target groundwater aquifer
- Collection of reliable field parameter data that is reproducible and comparable to existing data sets

Credere Associates, LLC (Credere) SOPs are a guidance and state requirements, site-specific plans, and special conditions may require alternative approaches.

1.2 APPLICABILITY

This SOP should be used during use of water quality field instrumentation during collection of groundwater, surface water, or residential drinking water samples collected in accordance with Credere SOPs CA-12: Low-Flow Groundwater Sampling, CA-13: Surface Water & Sediment Sampling, and CA-3: Residential Well Sampling, respectively.

2. PROCEDURE

2.1 NECESSARY EQUIPMENT

The following is a list of required equipment for groundwater sampling:

- Appropriate personal protection equipment (PPE)
- Water quality parameter instrument(s) (e.g., YSI, Horiba, etc.) with calibration cup
- Turbidity meter
- Calibration standards (check expiration dates)
    - pH 4, pH 7, and pH 10
    - Specific conductivity (NHDES requires 0.718 and 1.413 mS/cm solutions)
    - Oxidation reduction potential (ORP)/Zobell solutions
    - Zero mg/L dissolved oxygen (DO) solution
    - Turbidity standards
- Deionized water
- Rinse cup
- Bulb thermometer
- Calibration log (**Attachment A**)
- Field logbook
- Ink pens
- Paper towels

2.2 TRAINING AND HEALTHY AND SAFETY

Credere employees should have received 40-hour Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) training pursuant to 29 CFR 1910.120 and be current on their 8-hour refresher. Employees must have 24 hours of training specifically in water quality field instrument calibration procedures described herein and proven competence of the SOP in compliance with OSHA 29 CFR 1910.120€(4).

Employees will follow the Credere generic health and safety plan when calibrating water quality field instruments, and any site-specific HASP for any project.

2.3 CALIBRATION METHOD

Water quality field equipment will be calibrated at the Site at the beginning of each sampling day. The probe will be regularly maintained and documented by the Credere Equipment Manager. The manager is responsible for maintaining a log of the date of maintenance, cleaning, and repairs. Frequency of maintenance will be based on the frequency of equipment use; however, maintenance will occur at least every 3 months.

Calibration should occur at the Site to ensure the instrument is calibrated at the same temperature and pressure as where use of the instrument will occur.

Calibration Preparation

1. Upon arrival at the Site, begin a field logbook entry recording the Credere staff, scope of work for the day, and weather conditions. Indicate the equipment being calibrated and reference the calibration log for calibration details.
2. Begin calibrating the instruments according to the following parameter specific procedures.

YSI Calibration

The following procedure assumes use of a YSI 556 or 600XL, or similar. Other instrument specifics may vary, but the general procedure will be the same.

1. Turn on the display and connect the sonde using the cable. Allow the instrument to warm up according to the manufacturer's instructions.
2. By viewing the display in the read mode, ensure the instrument is programed to measure temperature in degrees Celcius (C°), pH in units, DO in percent (%) and milligrams per liter (mg/L), specific conductivity in milliSiemens per centimeter (mS/cm), and ORP in millivolts (mV).

Temperature

1. Set the instrument to read mode.
2. Fill the rinse cup with DI water and insert the probe and bulb thermometer.
3. Allow the probe and thermometer to stabilize and record the readings for both on the field log.
4. Compare results for consistency. Results should be within ±0.15 to 0.2 °C.

Three Point pH

1. Record the pH solution numbers and expiration dates on the calibration log.
2. Set the instrument to calibration mode and select the pH three point calibration.
3. ¾ fill the calibration cup with pH 4 solution and carefully screw the cap onto the sonde. Enter 4 in the screen requesting the respective standard. Allow the pH reading to stabilize. Record the pre-calibration stabilized pH reading.
4. Select calibrate and record the post-calibration reading on the calibration log.
5. Hit enter to advance to the next calibration standard.
6. Remove the calibration cup, discard or return the solution to the bottle, thoroughly rinse the probe in the rinse cup, and rinse the calibration cup. Shake excess water from both.
7. ¾ fill the calibration cup with pH 10 solution and carefully screw the cap onto the sonde. Enter 10 in the screen requesting the respective standard. Allow the pH reading to stabilize. Record the pre-calibration stabilized pH reading.
8. Select calibrate and record the post-calibration reading on the calibration log.
9. Hit enter to advance to the next calibration standard.

10. Remove the calibration cup, discard or return the solution to the bottle, thoroughly rinse the probe in the rinse cup, and rinse the calibration cup. Shake excess water from both.
11. ¾ fill the calibration cup with pH 7 solution and carefully screw the cap onto the sonde. Enter 7 in the screen requesting the respective standard. Allow the pH reading to stabilize. Record the pre-calibration stabilized pH reading.
12. Select calibrate and record the post-calibration reading on the calibration log.
13. Hit enter to return to the calibration screen, and back out of the pH calibration.
14. Remove the calibration cup, discard or return the solution to the bottle, thoroughly rinse the probe in the rinse cup, and rinse the calibration cup. Shake excess water from both.

Specific Conductivity

1. Record the specific conductivity solution number(s) and expiration date(s) on the calibration log. For work in New Hampshire, a two point calibration is required using 0.718 and 1.413 mS/cm standards. For work elsewhere, a one point calibration typically using 1.413 mS/cm solution is acceptable.
2. Set the instrument to calibration mode and select the specific conductivity calibration.
3. ¾ fill the calibration cup with the specific conductivity solution and carefully screw the cap onto the sonde.
4. Enter the respective solution concentration and hit enter. Allow the specific conductivity reading to stabilize. Record the pre-calibration stabilized specific conductivity reading.
5. Select calibrate and record the post-calibration reading on the calibration log.
6. Remove the calibration cup, discard or return the solution to the bottle, thoroughly rinse the probe in the rinse cup, and rinse the calibration cup. Shake excess water from both.
7. Hit enter to return to the calibration screen, if a second point calibration is need, repeat steps 3 through 6, otherwise, back out of the specific conductivity calibration.

Dissolved Oxygen Calibration

1. Record the zero DO solution number and expiration date on the calibration log.
2. Set the instrument to calibration mode and select the dissolved oxygen percent saturation calibration.
3. Lightly screw on the empty calibration cup.
4. Record the instruments internal reading of barometric pressure or record and enter the barometric pressure as reported by www.weather.gov. Press enter.
5. Allow the % DO reading to stabilize. Record the pre-calibration stabilized % DO reading.
6. Select calibrate and record the post-calibration reading on the calibration log.
7. Obtain the adjusted % saturation value from the Pressure vs. DO % Saturation table in **Attachment B**. Record the adjusted calibration value on the calibration log and compare to the value the instrument calibrated to.

8. Hit enter to return to the calibration screen and return to read mode.
9. ¾ fill the calibration cup with the zero DO solution and carefully screw the cap onto the sonde. Allow the instrument to read the zero DO solution. It may take several minutes for the instrument to read down to 0 and it may stabilize above 0 if the solution has been used previously. Record the reading on the calibration log.

ORP

1. Set the instrument to read mode.
2. ¾ fill the calibration cup with the ORP solution and carefully screw the cap onto the sonde.
3. Allow the temperature to stabilize and record the solution's temperature on the calibration log.
4. Obtain the adjusted ORP reading using the recorded temperature from the ORP temperature vs. mV table in **Attachment B**. Record the adjusted value on the calibration log.
5. Set the instrument to calibration mode and select the ORP calibration.
6. Enter the adjusted value obtained from **Attachment B** per above and hit enter.
7. Allow the ORP reading to stabilize. Record the pre-calibration stabilized ORP reading.
8. Select calibrate and record the post-calibration reading on the calibration log.
9. Remove the calibration cup, discard or return the solution to the bottle, and thoroughly double-rinse the probe and cup. ORP solution can be difficult to rinse. Shake excess water from both.
10. Hit enter to return to the calibration screen and back out of the ORP calibration.
11. Proceed to setup the instrument for use on the respective sampling job.

Turbidity Calibration

Turbidity meter calibration specifics will vary depending on the unit used. Since turbidity meter models used vary regularly, specific for a specific model are not provided herein; therefore, it is important to reference the instrument's user manual during calibration.

1. Turn on the turbidity meter and allow the unit to warm up per the manufacturer's recommendations.
2. Enter the calibration mode for the respective unit.
3. Insert the calibration standard and read the standard.
4. Make appropriate calibration adjustments per the user manual, and record adjustments on the calibration log (**Attachment A**).
5. Repeat steps 3 and 4 for each standard.

Post Calibration

1. Thoroughly rinse the YSI probes and flow through cell (if needed) with DI water and assemble the unit for the respective sampling job.
2. Ensure several paper towels are present in the turbidity meter case (especially important when using the Lamotte 2020 meter).

Evening Drift Check

The evening drift check will be completed at the close of a sampling day to assess the drift of the instrument throughout the day. A mid-day drift check may also be warranted depending on the data quality objectives of a project.

1. Set the instrument to read mode.
2. Using the same standards as were used to calibrate the respective instrument that morning (i.e., match up the bottle numbers), ¾ fill the calibration cup with a solution and carefully screw the cap onto the sonde.
3. Allow the respective reading to stabilize and record the reading under the evening drift check column on the calibration log.
4. Repeat steps 2 and 3 for each standard.
5. Complete and sign the calibration log for the day.

3. QUALITY ASSURANCE/QUALITY CONTROL

Quality assurance (QA) and quality control (QC) will be obtained through daily self-review of field notes for completion. Field documentation will be reviewed to assure it is compliant with Credere SOP CA-1: Field Activity Documentation and methods used for calibration compliant with the protocol herein.

The effectiveness of calibration and maintenance of the calibration by the instrument throughout a sampling day is measured through performance of the evening drift check. For instruments that appear to be behaving suspiciously mid-sampling day, a bump check can be performed using one or more standards to assess the accuracy of the instrument at that point in the day.

4. SAMPLING DOCUMENTATION

The following information should be recorded in the field logbook in compliance with Credere SOP CA-1:

- Date of calibration
- Credere personnel
- Equipment being calibrated
- Reference to the calibration log
- Scope of work
- Weather (particularly temperature, barometric pressure, and humidity)

5. REFERENCES

Credere Associates, LLC, *Standard Operating Procedure CA-1 Field Activity Documentation*, Revision 0, dated August 4, 2015.

Credere Associates, LLC, *Draft Standard Operating Procedure CA-3 Residential Well Sampling*, Draft, dated TBD.

Credere Associates, LLC, *Standard Operating Procedure CA-12 Low-Flow Groundwater Sampling*, Revision 0, dated August 11, 2015.

Credere Associates, LLC, *Draft Standard Operating Procedure CA-13 Surface Water & Sediment*, Draft, dated TBD.

New Hampshire Department of Environmental Services, *Calibration of Field Instruments*, SOP No. HWRB-17, Revision 4, dated January 2012.

US DoD, *Environmental Field Sampling Handbook, Revision 1.0*, dated April 2013.

US Environmental Protection Agency, *QA Bulletin Calibration of Dissolved Oxygen Meters*, EQAGUI-DO, Revision 0, dated February 2006.

US Environmental Protection Agency, Region 1, *Standard Operating Procedure Calibration of Field Instruments*, EQASOP-FieldCalibrat, Revision 2, dated January 19, 2010.

US Environmental Protection Agency, *Low Stress (low flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells*. EQASOP-GW-001, Revision 3, dated January 19, 2010.

Attachment A

Calibration Field Log

INSTRUMENT CALIBRATION LOG

Credere Associates, LLC, 776 Main Street, Westbrook, ME 04092

PROJECT NAME: _____ DATE: _____

PROJECT NUMBER: _____

WEATHER: _____
(barometric pressure, temperature, precipitation)

WATER QUALITY INSTRUMENT SN: _____

TURBIDITY METER SN: _____

FIELD STAFF ASSIGNED TO EQUIPMENT: _____

TEMPERATURE

Bulb thermometer: _____ °C

Instrument: _____ °C

pH 3 POINT CALIBRATION

ph 4 solution #: _____ pH 7 solution #: _____ pH 10 solution #: _____

Expiration: _____ Expiration: _____ Expiration: _____

	Morning		Evening Drift Check		
	before	after	before	after (if needed)	Comment
pH 4 calibration					
pH 7 calibration					
pH 10 calibration					

CONDUCTIVITY CALIBRATION *(718 µS/cm calibration required only for work in New Hampshire)*

0.718 mS/cm Conductivity solution #: _____ 1.413 mS/cm Conductivity solution #: _____

Expiration: _____ Expiration: _____

	Morning		Evening Drift Check		
	before	after	before	after (if needed)	Comment
Conduc. Calibration 0.718 mS/CM					
Conduc. Calibration 1.413 mS/CM					

DO Calibration

Barometric Pressure: _____ mm HG Calibration value*: _____

0 DO Solution #: _____ Expiration: _____

	Morning		Evening Drift Check		
	before	after	before	after (if needed)	Comment
DO Calibration					
0 DO Check	NA				

ORP Calibration

ORP solution #: _____ Expiration: _____

Solution temperature: _____ Adjusted solution reading*: _____

	Morning		Evening Drift Check		
	before	after	before	after (if needed)	Comment
ORP Calibration					

TURBIDITY CHECK

	Morning		Evening Drift Check		
	before	after (if needed)	before	after (if needed)	Comment
NTU check					
NTU check					
NTU check					
NTU check					

CALIBRATOR SIGNATURE

* see second page attachment for adjustment tables

Signature _____

Attachment B

Parameter Adjustment Tables

Value Adjustment Tables
Credere Associates. LLC. 776 Main Street. Westbrook. ME 04092

ORP temperature vs. mV Table	
Solution Temperature (°C)	Corrected ORP Reading (mV)
-5	270.0
-4	268.7
-3	267.4
-2	266.4
-1	264.8
0	263.5
1	262.2
2	260.9
3	269.6
4	258.3
5	257.0
6	255.7
7	254.4
8	253.1
9	251.8
10	250.5
11	249.2
12	247.9
13	246.6
14	245.3
15	244.0
16	242.7
17	241.7
18	240.1
19	238.8
20	237.5
21	236.2
22	234.9
23	233.6
24	232.3
25	231.0
26	229.7
27	228.4
28	227.1
29	225.8
30	224.5
31	223.2
32	221.9
33	220.6
34	219.3
35	218.0
36	216.7
37	215.4
38	214.1
39	212.8
40	211.5

Pressure vs. DO % Saturation	
Pressure (mm HG)	% Saturation
768	101
760	100
725	99
745	98
737	97
730	96
722	95
714	94
707	93
699	92
692	91
684	90
676	89
669	88
661	87
654	86
646	85
638	84
631	83
623	82
616	81
608	80
600	79
593	78
585	77
578	76
570	75
562	74
555	73
547	72
540	71
532	70
524	69
517	68
509	67
502	66

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Standard Operating Procedure CA-12
Groundwater Sampling

Effective Date: May 2, 2017
Revision: 2

Allison Drouin, Author — 5/2/2017 Date

Theresa Patten, Technical Review — 5/2/2017 Date

Judd Newcomb, Technical Review — 5/2/2017 Date

Revision	Date	Reason for Revision
1	8-2-2016	EPA Comments for SOP use with Generic QAPP
2	5-2-2017	Compliance with NHDES Sampling for Per- & Poly-Fluorinated Alkyl Substances

TABLE OF CONTENTS

1. OBJECTIVE AND APPLICABILITY

1.1 OBJECTIVE

The purpose of this Standard Operating Procedure (SOP) is to provide guidance for the collection of groundwater samples. If followed correctly this SOP will allow for the following:

- Safety of employees performing the sampling
- Collection of a sample representative of the target groundwater aquifer

It should be noted that Credere Associates, LLC (Credere) SOPs are a guidance and state requirements, site-specific plans, and special conditions may require alternative approaches.

1.2 APPLICABILITY

This SOP should be used during the collection of groundwater samples collected from monitoring wells installed in accordance with Credere SOP CA-8: Groundwater Monitoring Well Installation and developed in accordance with Credere SOP CA-9: Groundwater Monitoring Well Development.

2. PROCEDURE

2.1 NECESSARY EQUIPMENT

The following is a list of required equipment for groundwater sampling:

- Appropriate personal protection equipment (PPE)
- Photoionization detector (PID) (if applicable)
- Water level meter or interface probe (for NAPL Sites)
- Water quality parameter instrument(s) (e.g., YSI, Hach, etc.)
- Polyethylene or Teflon lined (if sampling for VOCs) 3/8 inch ID tubing
- Peristaltic pump (if sampling in Maine or New Hampshire)
    - Backup batteries
    - 1 foot silicone tubing per well
- Submersible bladder pump (if sampling in Massachusetts for volatiles)
    - QED MP10 Control and compressor; or
    - QED MP15 with extra CO2 tank for backup
    - String
    - Appropriately sized air line tubing
    - 2 feet of silicone tubing
- Turbidity meter
- Cooler with ice and sample protection material (bubble wrap)
- Laboratory provided sample containers plus appropriate trip blank(s)
- Polyethylene graduated cylinder
- Buckets (3 per setup)
- 0.45 micron filters (if analyzing for dissolved metals, at least one per well)
- Groundwater monitoring logs (**Attachment A**)
- Site plan
- Chain of custody (COC)
- Field logbook
- Field logs from previous sampling rounds (when available)
- Ink pens
- Socket set and/or well keys
- Metal detector (for locating wells)
- Tubing cutter
- Large screw driver (for prying)
- Lock keys (if stickups are locked)
- Timer
- Paper towels

2.2 TRAINING AND HEALTHY AND SAFETY

Credere employees should have received 40-hour Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) training pursuant to 29 CFR 1910.120 and be current on their 8-hour refresher. Employees must have 24 hours of

training specifically in groundwater sampling procedures described herein and proven competence of the SOP in compliance with OSHA 29 CFR 1910.120(e)(4).

Employees will follow the Credere generic health and safety plan when implementing groundwater sampling procedures, and any site-specific HASP for any project.

2.3 SAMPLE COLLECTION METHOD

When collecting samples from groundwater monitoring wells the general objective is to purge stagnant water at a low flow rate (100 to 400 milliliters per minute [mL/min]) and draw in fresh groundwater so as to be collecting a sample that is representative of the aquifer.

The Maine Department of Environmental Protection (DEP) and New Hampshire Department of Environmental Services (NHDES) SOPs permit the use of peristaltic pumps for collection of all groundwater samples; however, the Massachusetts DEP requires submersible bladder pumps for collection of volatile samples and the Rhode Island Department of Environmental Management (RIDEM) requires bailers for collection of volatile samples. State requirements/SOPs outside of Maine, New Hampshire, Massachusetts, and Rhode Island should be referenced prior to sampling to ensure this SOP is compliant.

Sampling should be conducted a minimum of 14 days (7 days is allowable by the Maine DEP) after completion of well development in accordance with Credere SOP CA-9.

Sampling Preparation

1. Upon arrival at the Site, begin a field logbook entry recording the Credere staff, scope of work for the day, and weather conditions.
2. Calibrate field instruments according to Credere SOP CA-11 and product specifications. Keep record of calibration in the field logbook.
3. Using the appropriate socket or well key, open the well being sure to place the bolts/lock in a safe location away from the well mouth. Remove the tension plug. (Note: temporary wells will not have a road box or standpipe) Record the physical condition and construction materials on the field log, and note any other pertinent observations such as odors, PID results of the well mouth (if applicable), frost heaving, damage to the monitoring well or protective system, etc.
4. If volatiles are a contaminant of concern (COC), the well mouth should be screened using a PID. Record both the well mouth and ambient air readings on the field log.
5. Using the water level meter or interface probe, measure the depth to water (and thickness of NAPL if present) to the nearest 0.01 and in accordance with Credere SOP CA-10. **Do not measure the depth to bottom at this time as not to stir up bottom sediment.** Record measurements on the field log. Using the historical depth to bottom, calculate the well volume and the middle of the present water column to be the target pump intake depth (unless other site-specific target pump intake depth has been established).

Purging

Low Flow

1. Determine the depth of pump intake according to the following option:
    a. Deep wells (screened below the water table) – mid-point of screen
    b. Overburden groundwater (screened across the water table) – mid-point of water column.
2. Install the pump tubing (peristaltic) or submersible bladder pump into the well at the desired pump intake. If using a submersible pump be sure to lower the pump using string in addition to the tubing to avoid losing the pump down the well. String should be replaced between wells during pump decontamination.
3. Connect the purge line to the bottom barb of the water quality instrument flow through cell and add a piece of tubing to the top barb to allow for drainage into a purge bucket.
4. After pump installation, re-measure the depth to water and start the pump on the lowest possible setting. Frequently measuring the depth to water, adjust the pump rate to establish a stable purge rate such that drawdown of more than 4 inches does not occur. Ideally the depth to water will stabilize with less than +/-0.01 change in depth to water.
5. After a stable purge rate is achieved, measure the flow rate using a graduated cylinder and stop watch. An ideal flow rate is between 100 and 400 mL/min. **If a stable flow rate cannot be achieved and draw down of greater than 4 inches occurs, discontinue purging and skip to the later described no-purge method. Do not allow the well to be pumped dry.** Record the stable flow rate on the field log.
6. Position the flow through cell at a 45 degree angle to minimize the accumulation of bubbles in the flow through cell, and allow the flow through cell to fill and begin recording field parameters measured by the YSI on the field log. Turbidity is measured using a separate turbidity meter and water is collected from before the flow through cell using a t-valve. Temperature, pH, specific conductivity, oxidation reduction potential (ORP), dissolved oxygen, turbidity, flow rate, and depth to water should be recorded every five minutes or at an interval to allow for complete exchange of water through the flow through cell calculated based on the volume of the flow through cell and flow rate. As field parameters and calibrations rely on water temperature or can have optical probes and heating tubing can cause volatilization of VOCs, avoid placing the flow through cell and tubing in direct sunlight and/or protect the flow through cell (e.g., place in the shadow of the purge bucket, etc.). Continue monitoring these field parameter until they have stabilized for a period of three consecutive readings according to the following criteria:
    - pH: ±0.1
    - Specific conductivity (mS/cm): 3%
    - Temperature (°C): 3%
    - Dissolved Oxygen (mg/L): 10% or less than 0.5mg/L
    - ORP (mV): ±10

- Turbidity (NTUs): less than 10 NTUs or within 10% if not sampling for unfiltered analyses

If parameters do not stabilize within a period of 2 hours or before a maximum purge volume of 5 well volumes, samples will be collected with field note justification of attempts to achieve stabilization and data will be reviewed for evidence of bias.

7. Once parameters have stabilized, begin sampling.

No Purge

If a stable flow rate could not be achieved during the low flow methodology, the following no-purge methodology will be implemented.

1. Disconnect the flow through cell and turn off the pump ensuring backflow into the well does not occur. Place a gloved finger over the end of the purge tubing. Allow the well to recharge while measuring the depth to water.
2. At the lowest possible flow rate, begin pumping again and purge the tubing length of one tubing volume to begin drawing in fresh well water.
3. After the tubing has been purged, begin sampling.
4. Continue monitoring the depth to water between sample containers to ensure the water in the well is not depleted. If the water level drops to within 2 feet of the well bottom or to 6 inches of the top of the well screen in deep wells with the water level above the screen, turn off the pump again and begin this method from the beginning until all sample containers have been filled.

Sample Collection

1. While maintaining the same flow rate as during purging, begin filling sample containers in order of decreasing volatility from immediately after the pump (i.e., before the flow through cell). Sample containers should be labeled with the time, sample ID, analyses, preservation, and sample date. Samples should be collected in the following order according to the following methods:
    a. VOCs and VPH: Fill hydrochloric acid (HCl) preserved volatile organic analysis (VOA) viles until a meniscus forms at the top. Be sure not to allow the tubing to contact the sample container and do not overflow the container as to dilute the preservative. Gently screw on the Teflon lined cap and invert the vile gently tapping your hand to check for air bubbles. If bubbles are present, unscrew the cap and reform the meniscus. Screw the cap back on and check again for bubbles. If bubbles are present again, discard the vial and begin with a new preserved vile. Although uncommon in our region, if a sample container effervesces after addition of a sample (i.e. carbonates are present that are reacting with the HCl), contact the laboratory for an alternative sampling method. Use a similar method for other VOAs container alternate preservatives (e.g., H_2SO_4 for low-level VOC analysis).

b. SVOCs and EPH: Fill a 1 liter HCl preserved (EPH) or unpreserved (SVOCs/PAHs) amber container to the neck. Insufficient volume may result in elevated laboratory reporting limits above the applicable regulatory criteria.

c. PCBs and unfiltered inorganic analyses: Fill a nitric acid (HNO_3) preserved (metals) or unpreserved (PCBs) container to the neck.

d. Filtered inorganic analyses: Apply a 0.45 micron filter to the end of the purge line and allow the filter to fill until water breaks through. Begin filling HNO_3 preserved polyethylene bottles to the neck.

2. Place all samples immediately on ice and prepare the chain of custody. Record the sample time, ID, analyses, sample volume and preservatives in the field log book and on the field log.

Post Sampling

1. Cease purging the well and using the water level meter or interface probe, measure the depth to the bottom of the well. If measurements significantly differ from historical data or sediment buildup can be felt in the well bottom, remove the sediment by agitation and overpumping methods until the sediment has been removed and the water runs clear or turbidity is similar to that of that time at well stabilization.
2. Disassemble the well setup.
3. Decontaminate non-dedicated equipment for use at the next well or for transport in accordance with Credere SOP CA-2. Insert the expansion plug into the well mouth, and replace the well cap and bolts or lock. If the well is completed with a flush mounted road box, ensure the bolts are tightly screwed to minimize potential damage to the well (particularly during the winter months from snow plows).

3. QUALITY ASSURANCE/QUALITY CONTROL

Quality assurance (QA) and quality control (QC) will be obtained through collection of additional samples. The types of samples to be collected are dependent on the data quality objectives and should be defined in the project's proposal/scope of work, Site-Specific Quality Assurance Project Plan (SSQAPP), regulatory/client guidance, or similar. Types of QA/QC samples that may be collected include:

- Field Duplicates: Field duplicate samples are two samples of the same matrix, which are collected, to the extent possible, at the same time, from the same location, using the same techniques and are analyzed at the same laboratory. Field duplicates will be handled, containerized, preserved, stored, and transported in the same manner. Sample volume for field duplicates should immediately follow the initial sample for each analysis (e.g., collect VOC sample, then VOCs duplicate, then SVOC sample, then SVOCs duplicate, etc.). Field duplicates are optional based on data quality objectives for a specific project.
- Matrix Spike/Matrix Spike Duplicates (MS/MSD): MS and MSD samples are collected as additional aliquots of sample to be used by the laboratory as QC samples. The samples are spiked in the laboratory by adding a predetermined concentration of target analytes into the sample prior to sample extraction/digestion and analysis. The concentrations of the target analytes determined during analysis are compared to the known concentration of the added spike compound (percent recovery) to provide a measure of the accuracy of the analysis in the site matrix. Precision is assessed by measuring the RPD between the two spiked samples.
- Trip Blanks**:** Trip blanks are prepared by the laboratory and consist of VOC-free water plus preservative (HCl) in a sealed VOA vial. A trip blank accompanies VOC field samples from the field to the laboratory to assess potential cross-contamination. One trip blank is to be included with each cooler of VOC field samples delivered to an analytical laboratory.
- Equipment Blanks: The field/equipment blanks are samples consisting of laboratory (analyte-free) water collected during a sampling event from a final rinse of sampling equipment after a decontamination procedure has been performed. The purpose of equipment blanks is to determine whether the sampling equipment is being adequately decontaminated and if it may be causing cross contamination of samples. Typically, equipment blanks are only applicable when non-dedicated equipment is being used to collect samples.
- Temperature Blanks: Some laboratories require temperature blanks to measure the cooler temperatures at the time of arrival at the laboratory. Some labs use a thermometer gun and do not require temperature blanks. Determine the applicable laboratory's requirements prior to field mobilization.

Additionally, field documentation will be reviewed to assure it is compliant with Credere SOP CA-1: Field Activity Documentation and methods used for sampling are compliant with the protocol herein.

4. SAMPLING DOCUMENTATION

The following information should be recorded in the field logbook in compliance with Credere SOP CA-1:

- Date of sampling event
- Credere personnel
- Scope of work
- Weather
- Purge rate/volume/duration
- Site conditions
- Sample IDs, time of collection, requested analyses, volume of sample collected, preservatives
- Decontamination procedure (if applicable)

5. REFERENCES

Credere Associates, LLC, *Standard Operating Procedure CA-1 Field Activity Documentation*, Revision 0, dated August 4, 2015.

Credere Associates, LLC, *Standard Operating Procedure CA-2 Equipment Decontamination*, Draft, dated TBD.

Credere Associates, LLC, *Standard Operating Procedure CA-7 Headspace Field Screening*, Draft, dated TBD.

Credere Associates, LLC, *Standard Operating Procedure CA-8 Monitoring Well Installation*, Draft, dated TBD.

Credere Associates, LLC, *Standard Operating Procedure CA-9 Monitoring Well Development*, Draft, dated TBD.

Credere Associates, LLC, *Standard Operating Procedure CA-11 Water Quality Field Instrument Calibration*, Draft, dated TBD.

Credere Associates, LLC, *Standard Operating Procedure CA-16 Chain of Custody Preparation*, Draft, dated TBD.

Credere Associates, LLC, *Standard Operating Procedure CA-17 Packaging and Shipping Samples*, Draft, dated TBD.

ASTM D 4448-01 (2013) *Guide for Sampling Groundwater Monitoring Wells*, ASTM International, West Conshohocken, PA, 2013, www.astm.org

ASTM D 6634-14 *Guide for Selection of Purging and Sampling Devices for Groundwater Wells*, ASTM International, West Conshohocken, PA, 2014, www.astm.org

ASTM D 6001-05 (2012) *Guide for Direct-Push Water Sampling for Geoenvironmental Investigations*, ASTM International, West Conshohocken, PA, 2012, www.astm.org

Commonwealth of Massachusetts Department of Environmental Protection, *Standard References for Monitoring Wells*, WSC-310-91, dated January 1991.

Maine Department of Environmental Protection, *Groundwater Sampling Using Low Flow Purging and Sampling for Long-Term Monitoring*, SOP No. RWM-DR-003, Revision 4, dated March 27, 2009.

Maine Department of Environmental Protection, *Groundwater Sample Collection for Site Investigation and Assessment Monitoring*, SOP No. RWM-DR-002, Revision 0, dated March 25, 2009.

New Hampshire Department of Environmental Services, *Low Flow Groundwater Purging and Sampling*, SOP No. HWRB-9, Revision 5, dated January 2012.

US DoD, *Environmental Field Sampling Handbook, Revision 1.0,* dated April 2013.

US Environmental Protection Agency, *Compendium of Superfund Field Operations Methods*, dated December 1987.

US Environmental Protection Agency Environmental Response Team, *Groundwater Well Sampling*, SOP# 2007, dated January 26, 1995.

US Environmental Protection Agency, *Low Stress (low flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells*. EQASOP-GW-001, Revision 3, dated January 19, 2010.

Attachment A

Groundwater Sampling Field Log
2015

LOW FLOW SAMPLING LOG
CREDERE ASSOCIATES, LLC

Credere Associates LLC

PROJECT NAME: _____ DATE: _____

PROJECT NUMBER: _____ LOCATION ACTIVITY

START: _____

SAMPLE LOCATION ID: _____ END: _____

WELL DATA:

WELL DEPTH (ft): _____ [ ] MEASURED [ ] HISTORICAL [ ] TOP OF WELL [ ] TOP OF CASING [ ] FROM GRADE [ ] _____

WATER DEPTH (ft): _____ [ ] MEASURED [ ] HISTORICAL

WATER LEVEL EQUIPMENT USED:
[ ] ELECT. COND. PROBE
[ ] FLOAT ACTIVATED PROBE
[ ] PRESSURE TRANSDUCER
[ ] _____
[ ] _____

DEPTH OF PUMP INTAKE (ft): _____ Stick-up (in): _____

WELL MATERIAL:	WELL LOCKED:	PROTECTIVE CASING SECURE:	CONCRETE COLLAR INTACT:
[ ] PVC	[ ] YES	[ ] YES	[ ] YES
[ ] SS	[ ] NO	[ ] NO	[ ] NO
[ ] _____			

AMBIENT AIR VOC: _____ PPM

WELL MOUTH VOC: _____ PPM

EQUIPMENT DATA:

PURGING	SAMPLING	Equipment	DECONTAMINATION FLUIDS USED:
[ ]	[ ] PERISTALTIC PUMP	_____	[ ] DISTILLED WATER
[ ]	[ ] SUBMERSIBLE	_____	[ ] DEIONIZED WATER
[ ]	[ ] BLADDER PUMP	_____	[ ] POTABLE WATER
[ ]	[ ] HAND PUMP	_____	[ ] TSP SOLUTION
[ ]	[ ] DEDICATED HDPE	_____	[ ] ALCONOX SOLUTION
[ ]	[ ] NEW HDPE	_____	[ ] NONE
[ ]	[ ] DEDICATED LDPE	_____	[ ] _____
[ ]	[ ] NEW LDPE	_____	[ ] _____
	[ ] FILTER		
[ ]	[ ] _____		

FIELD ANALYSIS DATA:

TIME	TEMP (°C)	pH	SP. COND. (mS/cm)	ORP (mV)	D.O. (mg/l)	TURBID. (ntu)	Flow Rate (mL/min)	DTW (ft)	Comments/Flow Rate (indicate stable flow rate)
	3%	±0.1	3%	±10	10%, <0.5	<10	100-400 mL/min		

SAMPLE DATA:

SAMPLE BOTTLE ID TIME	LOCATION	PRESERVATION METHOD	SAMPLE CONTAINER #	TYPE	LABORATORY ANALYSIS

PURGE DATA

[ ] 0.04 GAL/FT (1" DIAM.)
[ ] 0.16 GAL/FT (2" DIAM.)
[ ] 0.65 GAL/FT (4" DIAM.)
[ ] 1.47 GAL/FT (6" DIAM.)

x length of water column =
Total Well Volume: _____ g
Total Purge Volume: _____ g
# of well volumes:

Stable flow not achieved, sampled via no-purge: [ ]

_____
SAMPLER

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Standard Operating Procedure CA-13
Surface Water & Sediment

Effective Date: September 9, 2016
Revision: 0

Allison Drouin, Author — 9/9/2016 Date

Theresa Patten, Technical Review — 9/9/2016 Date

Judd Newcomb, Technical Review — 9/9/2016 Date

Revision	Date	Reason for Revision

TABLE OF CONTENTS

1. OBJECTIVE AND APPLICABILITY

1.1 OBJECTIVE

The purpose of this Standard Operating Procedure (SOP) is to provide guidance for the collection of surface water and sediment samples. If followed correctly, this SOP will allow for the following:

- Safety of employees collecting surface water and sediment samples
- Collection of representative samples

Credere Associates, LLC (Credere) SOPs are a guidance, and state requirements, site-specific plans, and special conditions may require alternative approaches.

1.2 APPLICABILITY

This SOP should be used during the collection of grab sediment and surface water samples from riverbanks, shallow lakes, ponds, streams, wetlands, tidal areas. This SOP does not cover collection of sediment via sediment coring or barge drilling, or collection of sediment or sludge from oil/water separators, catch basins or tanks as these collection procedures will be site-specific. Sediment should be logged in accordance with Credere SOP CA-4: Soil Description.

2. PROCEDURE

2.1 NECESSARY EQUIPMENT

The following is a list of required equipment for collection of sediment and/or surface water:

- Appropriate personal protection equipment (PPE)
  - Harness, rope, and spotter may be needed in certain conditions
  - Personal floatation device (PFD) may be needed in certain conditions
  - Waders or tall rain boots
  - Shoulder length neoprene gloves, insulated for cold temperatures (if necessary)
- Boat (if applicable)
- Sediment Sampling Equipment
  - Trowel/bulb corer/tube auger/Ponar grab/Ekman dredge/bucket auger
  - Volatile syringes
  - Stainless steel bowl
  - Stainless steel spoons
- Surface Water Sampling Equipment
  - Peristaltic pump
  - Backup battery
  - 1 foot of silicone tubing per sample
  - ¼ inch ID Teflon line (if sampling for VOCs) or polyethylene tubing
  - Extendable rod (pore water sampler) and zip ties
  - Dip bucket and rope
  - Beta/Van Dorn sampler (for sampling a distinct point in the water column)
  - Turbidity meter
  - Water quality parameter instrument(s) (e.g., YSI, Hach, etc.)
- Laboratory provided sample containers and appropriate trip blank(s)
- 0.45 micron filters (if analyzing for dissolved metals, one per sample location)
- Labels
- Chain of custody (COC)
- Sample cooler with ice
- Site plan
- Tubing cutter
- Field logbook
- Ink pens
- Decontamination supplies
- Measuring tape with weighted end (shallow water) or staff gauge (deeper water)
- Camera
- Paper towels
- GPS Unit

2.2 TRAINING AND HEALTHY AND SAFETY

Credere employees must have 40-hour Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) training pursuant to 29

CFR 1910.120 and be current on their 8-hour refresher. Employees must have 24 hours of training specifically in surface water and/or sediment sampling procedures described herein and proven competence of the SOP in compliance with OSHA 29 CFR 1910.120(e)(4).

Employees will follow the Credere generic health and safety plan when implementing sampling procedures, and any site-specific HASP for any project. **The buddy system should also be used when collecting sediment or surface water samples with one person in a safe location away from the water's edge to assist or get help in case of emergency. When using a boat for sample collection, the field team in the boat should maintain contact via cell phone with a landward team member or the Project Manager at least every two hours.**

Credere staff should never wade into water greater than 2 feet in depth to collect a sample, shallower if water is flowing swiftly or footing is unsafe (e.g., algae covered rocks). A PFD should be worn by any staff wading into any depth of swiftly flowing water or collecting samples in a boat. Certain conditions may warrant wearing a harness and tying off to a stationary onshore object prior to approaching the water's edge. Any such condition should be documented in the Site's Health and Safety Plan and donned as appropriate.

In the event of a water emergency where staff is drawn into flowing or deep water, attempt to swim to the nearest shore perpendicular with the flow of water. At no time attempt to stand up in swiftly flowing water and keep feet near the water surface relying on your PFD to stay afloat. The spotter should seek help from local emergency services immediately.

2.3 SURFACE WATER SAMPLING

The method for surface water sampling must be selected based on Site specific conditions. Surface water sampling stagnant water will require a different method than sampling surface water in swiftly flowing rivers. The following tools may be appropriate for different surface water environments:

Environment	Appropriate Sampling Method
Lake or pond, stagnant water	Peristaltic pump with intake below the surface
Flowing stream or river	Direct collection, peristaltic pump
Targeted depth in any water body	Beta sampler

The general objective when collecting surface water samples is to collect from a targeted depth interval within a water column or avoid unnecessary aeration of the sample. Surface water in stagnant water bodies can be overly aerated or contain surface debris such as pollen, leaves, aquatic vegetation, etc. Direct collection of stagnant water can result in biasing through collection of unmixed aerated surface water. Additionally, direct collection may be limited if pre-preserved sample containers are used, as submerging the container will result in over dilution of the preservative.

Surface water samples should be collected prior to sediment samples if being collected in the same location to avoid disturbance of bottom sediment prior to sample collection. Samples should be collected in the downstream most position first gradually working upstream to the most upstream sample location to avoid cross contamination of downstream samples. When

collecting samples from a motor boat, samples should be collected from the bow (front) of the boat to avoid contamination from the motor.

Pre-Sampling Procedure

Safety is of primary importance when working in and around water bodies. Regardless of one's confidence and experience in water, the safety procedures outlined herein will be followed. The sampling program should be well established and reviewed with proper management of sample containers and equipment prior to approaching the sampling locations. Additionally, the sampling locales should be observed from a safe distance prior to approaching with equipment to ensure the planned health and safety procedures are going to be adequate. The following procedures outline steps for preparation prior to sample collection:

1. Upon arrival at the Site, begin a field logbook entry recording the Credere staff, scope of work for the day, health and safety precautions, and weather conditions.
2. Observe sampling locations as conditions in some bodies can vary greatly with weather or engineering controls (e.g., dams, outflows, etc.); therefore, confirm the safety procedures in the Health and Safety Plan are adequate.
3. Calibrate field instruments according to Credere SOP CA-11 and product specifications. Keep record of calibration in the field logbook.
4. If sampling from a boat, perform an inspection of the boat and double check that all supplies needed for sampling a respective set of data points are aboard.

Types of Sampling Procedures

Direct Collection

Direct collection of surface water samples is the easiest and fastest method of surface water sampling. This method is best used in flowing water when pre-preserved laboratory sample bottles are not used. This method should avoid contact with bottom sediment. If water is not deep enough to submerge the largest collection container of an analysis suite, direct collection should not be used.

1. Mark sample locations along the shoreline using pin flags, stakes, or tree flagging.
2. Don shoulder length neoprene gloves when water temperatures are low since gloves and lower arm will be submerged repeatedly with this procedure.
3. Submerge unpreserved sample container approximately 6 inches below the surface of flowing water. Container should be submerged in the upstream position from the sampler with the container mouth pointed upstream. Replace cap and repeat with sample containers for other analyses. Samples should be collected in the following order according to the following methods:
    a. SVOCs/PAHs: Fill a 1 liter unpreserved (SVOCs/PAHs) amber container to the neck. Insufficient volume may result in elevated laboratory reporting limits above the applicable regulatory criteria.

b. PCBs and unfiltered inorganic analyses: Fill unpreserved (PCBs) container to the neck.

c. VOCs, VPH, EPH, metals, other filtered inorganic, or other preserved analyses cannot be collected by this method.

4. Dry outside of sample container and label container accordingly with the time, sample ID, analyses, and date.

5. Place all samples immediately on ice and prepare the chain of custody in accordance with Credere SOP CA-16. Record the sample time, ID, analyses, and sample volume in the field log book.

Peristaltic Pump Sampling

Surface water sampling with a peristaltic pump is most useful when targeting a certain shallow depth interval (e.g., below the surface) in environments or for analyses where direct collection is not desirable. This method also permits collection of field water quality parameters and inline filtration if required. **This method requires a team of two for collection: one person to hold the intake line in position and a second to fill sample containers.**

1. Mark sample locations along the shoreline using pin flags, stakes, or tree flagging. If sampling in open water, record the location position when over the respective sampling location with the GPS for improved locating in the future if needed.

2. Determine the appropriate sample depth below the surface and assess where the sampler will need to stand on the water's edge to reach the point using an extendable sampling rod or by hand. Waders may be warranted.

3. Considering the above information, determine the length of tubing need to reach the sample point and the peristaltic pump.

4. If the extendable rod is needed, attach tubing to the end of the rod using zip ties. Otherwise, connect the tubing to the peristaltic pump.

5. Begin purging at a moderate rate avoiding contact of the intake tubing with bottom sediments. The intake tube should be in the upstream position of the sampler. In lakes and ponds that require wading, it is helpful to wade into the water gently and stand still for 2-3 minutes prior to purging to allow disturbed bottom sediment to settle. Purge one tubing length to ensure water from the target interval is being drawn through the pump.

6. If field parameters are to be collected/monitored, connect the purge line to the bottom barb of the water quality instrument flow through cell and add a piece of tubing to the top barb to allow for drainage into a purge bucket. Alternatively, the probe protector may be installed on the water quality instrument and direct field readings can be taken from the surface water body. If sampling from a boat, it may be efficient to zip-tie the intake tubing to the water quality instrument and lower them together to the sample depth. **If taking direct measurements use a backup tether tied to the instrument's top loop to lower the instrument, do not rely on the instrument cable.** Record field parameters according to the Site specific work plan in the field logbook. When field parameter recording is complete, disconnect the flow through cell.

7. Begin filling sample containers in order of decreasing volatility from immediately after the pump (i.e., before the flow through cell). Sample containers should be labeled with the time, sample ID, analyses, preservation, and sample date. Samples should be collected in the following order according to the following methods:

    a. VOCs and VPH: Fill 40 milliliter (mL) hydrochloric acid (HCl) preserved volatile organic analysis (VOA) vile until a meniscus forms at the top. Be sure not to allow the tubing to contact the sample container and do not overflow the container as to dilute the preservative. Gently screw on the Teflon lined cap and invert the vile gently tapping your hand to check for air bubbles. If bubbles are present, unscrew the cap and reform the meniscus. Screw the cap back on and check again for bubbles. If bubbles are present again, discard the vial and begin with a new preserved vile. Although uncommon in our region, if a sample container effervesces after addition of a sample (i.e., carbonates are present that are reacting with the HCl), contact the laboratory for an alternative sampling method. Use a similar method for other VOAs container alternate preservatives (e.g., H_2SO_4 for low-level VOC analysis).

    b. SVOCs and EPH: Fill a 1 liter HCl preserved (EPH) or unpreserved (SVOCs/PAHs) amber container to the neck. Insufficient volume may result in elevated laboratory reporting limits above the applicable regulatory criteria.

    c. PCBs and unfiltered inorganic analyses: Fill a nitric acid (HNO_3) preserved (metals) or unpreserved (PCBs) container to the neck.

    d. Filtered inorganic analyses: Apply a 0.45 micron filter to the end of the purge line and allow the filter to fill until water breaks through. Begin filling HNO_3 preserved polyethylene bottles to the neck.

8. Place all samples immediately on ice and prepare the chain of custody in accordance with Credere SOP CA-16. Record the sample time, ID, analyses, preservative, and sample volume in the field log book.

Beta/Van Dorn Sampler Sampling

Surface water sampling with a beta/Van Dorn sampler is most useful when targeting a certain deep depth interval. Field parameters can be collected in combination with this sampling method through direct measurement in the surface water body. This method is less desirable for volatile samples as the sampled water is aerated during transfer from the sampler to the sample containers. This method requires no tubing and permits rapid collection of grab samples at multiple locations and/or depths. Inline filtration is not possible with this method. This method typically requires use of a boat to reach deeper water and recording of sample location on open water with a GPS.

1. When over the respective sampling location, record the location position with the GPS for improved locating in the future if needed.
2. Engage the sampler to the open position as shown below per the product specifications:

Van Dorn Sampler (ecoenvironmental.com)

3. Measure the depth to the bottom of the water body at the sample location to avoid contact with bottom sediment, record in the field logbook.
4. Lower the sampler over the side of the boat to the desired depth measured directly using an attached field tape. Allow sampler to rest at this depth for approximately 30 seconds to allow the surrounding water to equilibrate after the mixing caused by lowering the sampler.
5. If field parameters are to be collected/monitored, simultaneously lower the water quality instrument fitted with the probe protector and begin recording/monitoring directly from the surface water body. **If taking direct measurements, use a backup tether tied to the instrument's top loop to lower the instrument, do not rely on the instrument cable.** Record field parameters according to the Site specific work plan in the field logbook.
6. Release the weighted messenger to close the sampler and draw the sampler back into the boat.
7. Using the valve on one end of the sampler, begin filling sample containers in order of decreasing volatility. If additional volume is needed to fill sample containers, lower the sampler to the desired depth again, allow surrounding water to stabilize, and close the sampler. Sample containers should be labeled with the time, sample ID, analyses, preservation, and sample date. Samples should be collected in the following order according to the following methods:
    a. VOCs and VPH: Fill 40 milliliter (mL) hydrochloric acid (HCl) preserved volatile organic analysis (VOA) vile until a meniscus forms at the top. Be sure not to allow the tubing to contact the sample container and do not overflow the container as to dilute the preservative. Gently screw on the Teflon lined cap and invert the vile gently tapping your hand to check for air bubbles. If bubbles are present, unscrew the cap and reform the meniscus. Screw the cap back on and check again for bubbles. If bubbles are present again, discard the vial and begin with a new preserved vile. Although uncommon in our region, if a sample container effervesces after addition of a sample (i.e., carbonates are present that are reacting with the HCl), contact the laboratory for an alternative sampling method. Use a similar method for other VOAs container alternate preservatives (e.g., H_2SO_4 for low-level VOC analysis).

b. SVOCs and EPH: Fill a 1 liter HCl preserved (EPH) or unpreserved (SVOCs/PAHs) amber container to the neck. Insufficient volume may result in elevated laboratory reporting limits above the applicable regulatory criteria.

c. PCBs and unfiltered inorganic analyses: Fill a nitric acid (HNO_3) preserved (metals) or unpreserved (PCBs) container to the neck.

8. Place all samples immediately on ice and prepare the chain of custody in accordance with Credere SOP CA-16. Record the sample time, ID, analyses, preservative, and sample volume in the field log book.

Post Sampling

1. Decontaminate non-dedicated equipment for use at the next sample location or for transport in accordance with Credere SOP CA-2.

2.4 SEDIMENT SAMPLING

The method for sediment sampling must be selected based on Site specific conditions. Sediment sampling in a tidal area with no overlying water will require a different method than sampling sediment in swiftly flowing rivers. The following tools may be appropriate for different sediment environments:

Environment	Appropriate Tools
Tidal flat, no overlying water	Trowel or spoon
Lake or pond edge, stagnant water	Bulb corer or bucket auger
Slow flowing stream	Bulb corer or tube auger
Swiftly flowing river edge	Tube auger
Deep water – hard bottom	Ponar sampler
Deep water – soft bottom	Ekman grabber

The primary objective when selecting an appropriate sampling tool is to avoid loss of fine grained sediment from the sample during extraction through the overlying water.

Surface water samples should be collected prior to sediment samples if being collected in the same location to avoid disturbance of bottom sediment prior to sample collection. Samples should be collected in the downstream most position first gradually working upstream to the most upstream sample location to avoid cross contamination of downstream samples. When collecting samples from a motor boat, samples should be collected from the bow (front) of the boat to avoid contamination from the motor. If applicable in stagnant water conditions (e.g., sampling a pond due to a point source such as a discharge pipe), samples furthest from the source area should also be sampled first to avoid suspending potential contaminated sediment and further reduce the risk of cross-contamination.

Pre-Sampling Procedure

Safety is of primary importance when working in and around water bodies. Regardless of one's confidence and experience in water, the safety procedures outlined herein will be followed. The sampling program should be well established and reviewed with proper management of sample

containers and equipment prior to approaching the sampling locations. Additionally, the sampling locales should be observed from a safe distance prior to approaching with equipment to ensure the planned health and safety procedures are going to be adequate.

1. Upon arrival at the Site, begin a field logbook entry recording the Credere staff, scope of work for the day, health and safety precautions, and weather conditions.
2. Calibrate field instruments according to Credere SOP CA-11 and product specifications. Keep record of calibration in the field logbook.
3. If sampling from a boat, perform an inspection of the boat and double check that all supplies needed for sampling a respective set of data points are aboard.

Types of Sampling Procedures

Shallow Water Sampling

Shallow water sediment sampling involves the direct collection of sediment from tidal or shallow water environments using hand tools. The primary objective of this method of sampling is to obtain a representative sample of sediment that retains all particle sizes (primarily fines) while removing the sample through the overlying water column. The appropriate tools can be selected based on the rate of flow of the overlying water.

1. If sampling in a tidal zone where surface exposure of sediment occurs within the cycle, facilitate sampling by planning sampling at low tide when no overlying surface water is present.
2. In environments with overlying surface water, don shoulder length neoprene gloves when water temperatures are low since gloves and lower arm will be submerged repeatedly with this procedure.
3. Mark sample locations along the shoreline using pin flags, stakes, or tree flagging.
4. Using the appropriate sampling device from the above table to collect sediment from the sample location. Use care when removing the device to minimize the amount washed out by running water.
5. If sampling for volatiles, place the filled sampling device, in/over a decontaminated stainless steel bowl. Using a dedicated syringe, obtain 10 grams of sediment directly from the sampling device and transfer to a 40 mL methanol preserved VOA. Remove any sand grains or sediment from the cap threads and securely replace the VOA vile cap. Failure to remove sediment and sand from the cap threads can result in leaking of preservative out of or infiltration of cooler ice melt into the container, which both may impact the data. **If sampling for only volatiles, also collect a syringe full of sediment and cap the syringe for percent moisture analysis by the lab.** Place the filled syringe and VOA vile together in a bubble wrap sleeve. If other analyses are requested, this can be performed from other unpreserved sediment volume.
6. Empty the remaining sediment from the sampling device into the decontaminated stainless steel bowl. Removal any organic detritus and decant excess water off the surface of the sediment using care not to pour off fines. If additional volume is needed,

repeat step 3 above and empty the device into the stainless steel bowl. Homogenize the sediment and begin filling the remaining sample containers according to the following:

a. SVOCs, PAHs, EPH, and PCBs: Fill an amber 4 to 8 oz. glass jar. Wipe away sand from the cap threads and replace cap. Failure to remove sediment and sand from the cap threads can result in infiltration of cooler ice melt into the container, which may impact the data.

b. Metals: Fill a clear 4 to 8 oz. glass jar to the neck. Wipe away sand from the cap threads and replace cap. Failure to remove sediment and sand from the cap threads can result in infiltration of cooler ice melt into the container, which may impact the data.

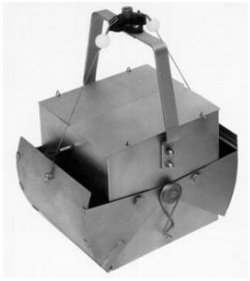
7. Place all samples immediately on ice and prepare the chain of custody in accordance with Credere SOP CA-16. Record the sample time, ID, analyses, preservative, and sample volume in the field log book.

Deep Water Sampling

Deep water sampling involves collection of sediment samples from depth below water greater than is reachable by hand. This type of sampling is done using a mechanical sampling device equipped with rapidly closing jaws that dig into the bottom sediment and capture a sample. This method typically requires use of a boat and recording of sample location on open water with a GPS.

1. When over the respective sampling location, record the location position with the GPS for improved locating in the future if needed.
2. Measure the depth to the bottom at the sampling location and record in the field logbook.
3. Engage the sampling device to the open position as shown below:

Ponar Sampler (www.envirotools.hu)



Ekman Sampler (www.damarus.com)

4. Lower the sampling device to approximately 1 foot from the bottom, then drop the sampler the remaining 1 foot to lodge in the sediment, and immediately drop the release messenger to obtain a sample.
5. Retrieve the sampler and inspect the contents ensuring water is present above the sediment indicating no leakage and the targeted depth has been achieved.
6. If sampling for volatiles, using a dedicated syringe, obtain 10 grams of sediment directly from the open top doors of the sampling device and transfer to a 40 mL methanol

preserved VOA. Remove any sand grains or sediment from the cap threads and securely replace the VOA vile cap. Failure to remove sediment and sand from the cap threads can result in leaking of preservative out of or infiltration of cooler ice melt into the container, which both may impact the data.

7. Empty the remaining sediment from the sampling device into a decontaminated stainless steel bowl. Removal any organic detritus. If additional volume is needed, repeat steps 3 through 5 above and empty the device into the stainless steel bowl. Homogenize the sediment and begin filling the remaining sample containers according to the following:
    a. SVOCs, PAHs, EPH, and PCBs: Fill an amber 4 to 8 oz. glass jar. Wipe away sand from the cap threads and replace cap. Failure to remove sediment and sand from the cap threads can result in infiltration of cooler ice melt into the container, which may impact the data.
    b. Metals: Fill a clear 4 to 8 oz. glass jar to the neck. Wipe away sand from the cap threads and replace cap. Failure to remove sediment and sand from the cap threads can result in infiltration of cooler ice melt into the container, which may impact the data.
8. Place all samples immediately on ice and prepare the chain of custody in accordance with Credere SOP CA-16. Record the sample time, ID, analyses, preservative, and sample volume in the field log book.

Post Sampling

1. Decontaminate non-dedicated equipment for use at the next sample location or for transport in accordance with Credere SOP CA-2.

3. QUALITY ASSURANCE/QUALITY CONTROL

Quality assurance (QA) and quality control (QC) will be obtained through collection of additional samples. The types of samples to be collected are dependent on the data quality objectives and should be defined in the project's proposal/scope of work, Site-Specific Quality Assurance Project Plan (SSQAPP), regulatory/client guidance, or similar. Types of QA/QC samples that may be collected include the following:

- Field duplicates (optional)
- Matrix spike/matrix spike duplicates (MS/MSD) (optional)
- Trip blanks (should be included with volatile analyses)
- Equipment blanks (e.g., after decontaminating an auger or sampler)
- Temperature blanks (laboratory specific, should be included in sample coolers for certain labs)

Additionally, field documentation will be reviewed to assure it is compliant with Credere SOP CA-1: Field Activity Documentation and methods used for sampling are compliant with the protocol herein.

4. FIELD DOCUMENTATION

The following information should be recorded in the field logbook in compliance with Credere SOP CA-1:

- Date of sampling
- Credere personnel
- Scope of work
- Weather
- Equipment used
- Changes in scope
- Correspondence with Project Managers
- Sample depths and depths to bottom
- Sample location information (unless GPS is used)
- Sample IDs, time of collection, requested analyses, volume of sample collected, preservatives
- Decontamination procedures
- General timeline of field activities

5. REFERENCES

Credere Associates, LLC, *Standard Operating Procedure CA-1 Field Activity Documentation*, Revision 0, dated August 4, 2015.

Credere Associates, LLC, *Standard Operating Procedure CA-4 Soil Description*, Draft, dated TBD.

Credere Associates, LLC, *Standard Operating Procedure CA-11 Water Quality Field Instrument Calibration*, Draft, dated TBD.

Credere Associates, LLC, *Standard Operating Procedure CA-16 Chain of Custody Preparation*, Draft, dated TBD.

Maine Department of Environmental Protection, *Surface Water and Sediment Sampling*, RWM-DR-004, dated March 27, 2009

New Hampshire Department of Environmental Services, *Sediment Sampling Procedure*, HWRB-13, Revision 2, dated January 2012.

New Hampshire Department of Environmental Services, *Surface Water Sampling Procedure*, HWRB-10, Revision 2, dated January 2012.

U.S. DoD, *Environmental Field Sampling Handbook, Revision 1.0*, dated April 2013.

U.S. Environmental Protection Agency Environmental Response Team, *Sediment Sampling*, SOP #2016, dated November 17, 1994.

U.S. Environmental Protection Agency Environmental Response Team, *Surface Water Sampling*, SOP #2013, dated November 17, 1994.

U.S. Environmental Protection Agency, Region 4, Science and Ecosystem Support Division, Operating Procedures, *Surface Water Sampling*, SESDPROC-201-R3, dated February 28, 2013.

U.S. Environmental Protection Agency, Region 4, Science and Ecosystem Support Division, Operating Procedures, *Fluvial Sediment Sampling*, SESDPROC-500-R3, dated July 19, 2013.

U.S. Environmental Protection Agency, Region 4, Science and Ecosystem Support Division, Operating Procedures, *Sediment Sampling*, SESDPROC-200-R3, dated August 21, 2014.

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Standard Operating Procedure CA-16
Chain of Custody

Effective Date: November 29, 2017
Revision: 0

Allison Drouin, Author — 11/29/2017 Date

Theresa Patten, Technical Review — 11/29/2017 Date

Revision	Date	Reason for Revision

TABLE OF CONTENTS

ATTACHMENTS

1. OBJECTIVE AND APPLICABILITY

1.1 OBJECTIVE

The purpose of this Standard Operating Procedure (SOP) is to provide a standard for the completion of chain of custody forms while collecting samples to be submitted to a laboratory by Credere Associates, LLC (Credere) employees. Sample custody begins immediately after a sample is collected. The sampler who collected the sample is responsible for the preservation and integrity of the sample(s) until that responsibility is transferred to someone else, and documented with the chain of custody form. The chain of custody form then travels with the sample(s) and is used to document any other transfers of custody. If followed correctly this SOP will allow for the following:

- Proper documentation of the custody of samples from collection to analysis and the maintenance of the integrity of the samples during the entire chain of custody
- Accurate communication of sample analysis requirements and sample details to the analytical laboratory
- Samples are protected from loss or damage
- Defensibility of data collected for a project in the court of law

1.2 APPLICABILITY

This SOP should be used during collection of any samples to be submitted to an analytical laboratory. This SOP should be used in conjunction with the following sample collection SOPs:

- CA-3: Residential Well Sampling
- CA-5: Environmental Soil Sampling
- CA-12: Low-Flow Groundwater Sampling
- CA-13: Surface Water & Sediment Sampling
- CA-14: Sub-Slab Soil Gas and Indoor Air Sampling
- CA-15: Asbestos Containing Materials (ACM) Surveys and Asbestos Abatement Air Monitoring and Clearances
- CA-23: Collection of PCB-Containing Building Material and Substrate Samples
- CA-26: Incremental Sampling Methodology

Additionally, this SOP should be followed in conjunction with the procedures outlined in Credere SOP CA-1: Field Activity Documentation and custody elements of Credere SOP CA-17 Packaging and Shipping Samples.

2. PROCEDURE

2.1 REQUIRED EQUIPMENT

The following is a list of required equipment:

- Laboratory specific or generic chain of custody
- Ink pen (all weather pens are best)
- Field sampling data forms and field notes for associated samples
- Associated properly labeled samples

2.2 DEFINITIONS

- **Chain of Custody Form** – A document detailing who is legally responsible for samples at any point in time from collection until the sample is received by the laboratory.
- **Custody** – A sample is "in custody" when: 1) the sample is in the sampler's possession, or 2) the sample was in the sampler's possession and then secured by the sampler to prevent tampering, or 3) the sample is placed in a designated secure area.
- **Secure Area** – An area in which entry is limited by keyed lock to a designated population.

2.3 CHAIN OF CUSTODY FORM

Chain of custody forms should, in most cases, be carbon copy forms received directly from the laboratory where samples will be submitted. In certain instances, prepopulated generic chain of custodies may be used (e.g., larger projects, FUDSCHEM set up projects). An example chain of custody form for reference is provided as **Attachment A**. The following outlines the steps for completing chain of custody forms:

- Complete the heading information (company, project, project required analyses/methods) prior to entering any sample information.
- At the time of sample collection, properly label the sample container according to the applicable sampling SOP (see **Section 1.2**). Immediately enter the sample details into the field logbook and/or field forms according to SOP CA-1 and enter the sample information onto the chain of custody form. Samples should be entered immediately in order of time collected. The chain of custody should not be completed at the end of a sampling day or anytime thereafter (e.g., the next day or at demobilization), otherwise the custody will not have been properly documented. In the event that multiple field samplers are submitting samples on a single chain of custody, communication should be maintained between samplers and the field manager (i.e., the person completing the chain of custody) to ensure no two samples have the same time and that the proper order of samples on the chain of custody can be maintained. Generally, laboratory provided chain of custody forms will provide fields for all required information. All fields must be populated prior to submittal to the laboratory.

The following is a list of required information on chain of custody forms:

- Credere name and address
- Project manager name and contact information
- Project number and project name
- Required level of reporting
- Sample IDs
- Preservatives
- Number of containers collected (include MS/MSD volume if collected)
- Date of sample collection
- Time of sample collection
- Type of sample
- Requested analyses
- Custody signatures documenting the timeline of custody from collection to analysis.
- Any notes for special handling or criteria for certain samples (e.g., MS/MSD for metals only, hold samples pending criteria)

- Ensure the chain of custody, field notes and sample container have matching field IDs, required analyses, and sample times. (This will be QC'ed again at the close of the field day per SOP CA-1 and other sampling SOPs.)
- It is not required to "sign over" samples from one Credere employee to another or into Credere cold storage within Credere's office; as Credere's is considered one entity, and the samples are still in Credere's custody.

Error Corrections

All error corrections will be crossed out with a single line to maintain legibility of the original entry, and the correction will be entered, dated and initialed. At no time should an entry be blacked out or made illegible.

2.4 STORAGE

Samples should be stored in Credere's possession or in a secure location accessible only to authorized Credere personnel (e.g., personal vehicles, Credere equipment room cold storage) until transferred to laboratory/courier custody. It is not necessary to sign away custody from the sampler to a storage location as long as the storage location meets the above criteria as the above storage is still considered to be in Credere's custody.

2.5 COURIER SERVICE AND LAB DELIVERY

At the time of transfer of custody to the lab or courier, signatures with the date and time of transfer will document the change in custody. If samples are being sent by mail, it will not be possible for the carrier to sign the chain and the receiver should be indicated to be "via carrier" (e.g., via Fedex, via USPS).

3. QUALITY ASSURANCE/QUALITY CONTROL

Logbooks, field data forms, and chains of custody will be checked for consistency. (Note: see SOP CA-1 for specifics on preparation of a field forms/logbook). In general, the following shall be checked at the close of a field task, sampling event, or project:

- Level of detail on COC is sufficient to communicate sample details and required analyses
- Completeness and accuracy of COCs
- Consistency of data between logbook, field forms and COC
- COC completion compliance with this SOP

4. FIELD DATA MANAGEMENT

Copies of COCs will be uploaded to the Credere server for easy access during report preparation and for reference prior to receipt of the laboratory reports. Carbon copies of COCs will be kept in the permanent project central file with the sampling field sheets.

5. REFERENCES

Credere Associates, LLC, *Standard Operating Procedure CA-1: Field Activity Documentation*, Revision 1, dated August 2, 2016.

Credere Associates, LLC, *Standard Operating Procedure CA-3: Residential Well Sampling*, Revision 1, dated March 17, 2016.

Credere Associates, LLC, *Standard Operating Procedure CA-5: Environmental Soil Sampling*, revision 0, dated May 27, 2017.

Credere Associates, LLC, *Standard Operating Procedure CA-12: Low-Flow Groundwater Sampling*, revision 2, dated May 2, 2017.

Credere Associates, LLC, *Standard Operating Procedure CA-13: Surface Water & Sediment Sampling*, revision 0, dated September 9, 2016.

Credere Associates, LLC, *Standard Operating Procedure CA-14: Sub-Slab Soil Gas and Indoor Air Sampling*, Draft, dated TBD.

Credere Associates, LLC, *Standard Operating Procedure CA-15: Asbestos-Containing Materials (ACM) Surveys and Asbestos Abatement Air Monitoring and Clearances*, Draft, dated TBD.

Credere Associates, LLC, *Standard Operating Procedure CA-17: Packaging and Shipping Samples*, revision 0, dated August 22, 2017.

Credere Associates, LLC, *Standard Operating Procedure CA-23: Collection of PCB-Containing Building Material and Substrate Samples*, revision 0, dated October 25, 2017.

Credere Associates, LLC, *Standard Operating Procedure CA-26: Incremental Sampling Methodology*, revision 0, dated October 31, 2017.

Maine DEP, *Standard Operating Procedure Chain of Custody*, RWM-DR-012, Revision 05, dated April 3, 2009.

US DoD, *Environmental Field Sampling Handbook, Revision 1.0,* dated April 2013.

US Environmental Protection Agency, *Compendium of Superfund Field Operations Methods*, dated December 1987.

PAGE 1 OF 1

Absolute Resource associates

124 Heritage Avenue #16
Portsmouth, NH 03801
603-436-2001
absoluteresourceassociates.com

CHAIN-OF-CUSTODY RECORD AND ANALYSIS REQUEST

Company Name: Credere Associates, LLC
Company Address: 776 Main St, Westbrook, Maine
Report To: Project Manager
Phone #: 207-828-1272 x 100
Invoice to Email: jenn@crederellc.com
☐ Hard Copy Invoice Required

Project Name:
Project #: 17001501
Project Location: NH MA (ME circled) VT NY ___ Other
Protocol: RCRA SDWA NPDES MCP NHDES OTHER
Reporting Limits: (QAPP circled) GW-1 S-1 EPA DW Other___
Quote # Credere ☐ NH Reimbursement Pricing
PO # ___

Lab Sample ID (Lab Use Only)	Field ID	# CONTAINERS	Matrix: WATER	Matrix: SOLID	Matrix: OTHER	Preservation: HCl	Preservation: HNO_3	Preservation: H_2SO_4	Preservation: NaOH	Preservation: MeOH	Sampling: DATE	Sampling: TIME	Sampling: SAMPLER
	CA-SB-1 (6-8)	2		X						X	1/1/17	0810	ASD
	CA-DUP1	2		X						X	—	—	↓
	CA-SS-6 (0-2)	3		X							1/1/17	0830	↓
	CA-MW-1	3	X			X	X				↓	1310	↓
	Trip Blank	1		X						X			
	Trip Blank	1	X			X							

ANALYSIS REQUEST

Field ID	☒ VOC 8260 ☐ VOC 8260 NHDES ☐ VOC 8260 MADEP	☐ VOC 624 ☐ VOC BTEX ☐ MtBE, only ☐ VOC 8021VT	☒ VPH MADEP ☐ GRO 8015 ☐ 1,4-Dioxane	☐ VOC 524.2 ☐ VOC 524.2 NH List ☐ Gases-List:	☒ TPH ☐ DRO 8015 ☐ EPH MADEP ☐ TPH Fingerprint	☒ 8270PAH ☐ 8270ABN ☐ 625 ☐ EDB	☐ 8082 PCB ☐ 8081 Pesticides ☐ 608 Pest/PCB	☐ O&G 1664 ☐ Mineral O&G SM5520F	☐ pH ☐ BOD ☐ Conductivity ☐ Turbidity	☐ TSS ☐ TDS ☐ TS ☐ TVS ☐ Alkalinity	☒ RCRA Metals ☐ Priority Pollutant Metals ☐ TAL Metals ☐ Hardness	☐ Total Metals-list:	☐ Dissolved Metals-list:	☐ Ammonia ☐ COD ☐ TKN ☐ TN ☐ TON ☐ TOC	☐ T-Phosphorus ☐ Phenols ☐ Bacteria P/A ☐ Bacteria MPN	☐ Cyanide ☐ Sulfide ☐ Nitrate + Nitrite ☐ Ortho P	☐ Nitrate ☐ Nitrite ☐ Chloride ☐ Sulfate ☐ Bromide ☐ Fluoride	☐ Corrosivity ☐ Reactive CN ☐ Reactive S- ☐ Ignitibility/FP	☐ TCLP Metals ☐ TCLP VOC ☐ TCLP SVOC ☐ TCLP Pesticide	Subcontract: ☐ Grain Size ☐ Herbicides ☐ Formaldehyde				(Grab (G) circled) or Composite (C)
CA-SB-1 (6-8)	X				X																			
CA-DUP1	X				X																			
CA-SS-6 (0-2)						X					X			(MS/MSD)										
CA-MW-1	X										X													
Trip Blank	X																							
Trip Blank	X																							

TAT REQUESTED
Priority (24 hr)* ☐
Expedited (48 hr)* ☐
Standard (10 Business Days) ☒
*Date Needed 1/9/17

See absoluteresourceassociates.com for sample acceptance policy and current accreditation lists.

SPECIAL INSTRUCTIONS
MS/MSD for metals only, metals water samples are field filtered

REPORTING INSTRUCTIONS ☒ PDF (e-mail address) pmanager@credenellc.com
☐ HARD COPY REQUIRED ☐ FAX (FAX#)___

RECEIVED ON ICE ☒ YES ☐ NO
TEMPERATURE <6 °C

CUSTODY RECORD

Relinquished by	Date	Time	Received by	Date	Time
Relinquished by Sampler: Ann Sampler (credere)	1/2/17	0900	Received by: Brian Labertory Cain (courier)	1/2/17	0900
Relinquished by: Brian Laburo Cain (courier)	1/2/17	1400	Received by: Labertory Cain (check-in)	1/2/17	1400
Relinquished by:			Received by Laboratory:		

QSD-01 Revision 1/27/16

CREDERE ASSOCIATES, LLC
776 Main Street
Westbrook, Maine 04092
Phone: 207-828-1272
Fax: 207-887-1051

Standard Operating Procedure CA-17
Packaging and Shipping Samples

Effective Date: August 22, 2017
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Allison Drouin, Author — 8/22/2017 Date

Theresa Patten, Technical Review — 8/22/2017 Date

Judd Newcomb, Technical Review — 8/22/2017 Date

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1. OBJECTIVE AND APPLICABILITY

1.1 OBJECTIVE

The purpose of this Standard Operating Procedure (SOP) is to provide guidance for packaging and shipping environmental samples. If followed correctly, this SOP will allow for the following:

- Safety of employees performing the sampling
- Transport of environmental samples after relinquished by Credere to laboratories while maintaining adequate cooler temperature and sample integrity

Credere Associates, LLC (Credere) SOPs are guidance, and state requirements, site-specific plans, and special conditions may require alternative approaches.

1.2 APPLICABILITY

This SOP should be used when preparing environmental samples for shipment other than by laboratory courier. This SOP does not cover procedures for actual sample collection, which are provided in the following Credere SOPs:

- Credere SOP CA-3: Residential Well Sampling
- Credere SOP CA-5: Environmental Soil Sampling
- Credere SOP CA-12: Groundwater Sampling
- Credere SOP CA-13: Surface water and Sediment Sampling
- Credere SOP CA-14: Sub-Slab Soil Gas and Indoor Air Sampling
- Credere SOP CA-15: Asbestos-Containing Material Sampling
- Credere SOP CA-23: Collection of PCB-Containing Building Material Samples

2. PROCEDURE

2.1 NECESSARY EQUIPMENT

The following is a list of required equipment:

- Samples
- Chain of custody
- Cooler
- Ice
- Ziploc gallon bags
- Bubble wrap or bags
- Packing tape
- Duct tape/fiber tape
- Custody seals
- Permanent marker
- Box/envelope for uncooled samples
- Zip ties (if shipping in plastic container)
- Appropriate personal protection equipment (PPE)

2.2 TRAINING AND HEALTHY AND SAFETY

Credere employees must have received 40-hour Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) training pursuant to 29 CFR 1910.120 and be current on their 8-hour refresher. Employees must have adequate training in the procedures herein and proven competence of the SOP.

Employees will follow Credere's generic health and safety plan (HASP) and the site-specific HASP when packaging any samples.

There are restrictions on shipping dangerous goods. In the case of certain waste characterization, performance evaluation, methanol preserved samples totaling more than 500mL, or polychlorinated biphenyl (PCB) samples with known elevated concentrations, additional requirements may be warranted for shipping according to 49 CFR Part 172.

2.3 SHIPPING PLANNING

Samples must be shipped to the laboratory in a reasonable time period after sample collection to allow for laboratory receipt and processing within the relevant sample hold times. In most instances/Site locations, samples for shipping should be packaged in the field and shipped the day of collection. It is pertinent to know the local drop-off facility closing time to plan the sampling day accordingly. Certain remote locations will not allow for this timeline and appropriate planning should be considered prior to mobilization. Special consideration should be made for Friday sampling and Saturday lab availability for sample receiving. Additionally, expedited sampling may be required in the summer months when trucks and warehouse are hotter to minimize melting ice and maintain sample temperature.

2.4 COOLER PACKAGING

Most soil and water samples require cooling to less than 6° Fahrenheit for storage. Asbestos, radon, and air samples do not require cooling and should be packaged according to **Section 2.6**. Samples should remain on ice within the cooler until immediately before packaging for shipping. Cooler packing should be completed while wearing clean nitrile gloves and safety glasses. The following procedure outlines the steps to properly prepare the cooler for shipping.

1. Gather all necessary materials for packaging prior to removing the samples from the cooler.
2. Place loose ice in heavy-duty Ziploc or similar bags to minimize water leakage into the cooler. Double bagging may be warranted in the summer.
3. If the cooler has a drain, securely tape over the drain with at least three layers of duct tape to minimize leakage. (The cooler will be returned if it leaks).
4. Complete the chain of custody per Credere SOP CA-16 and compare against field notes for consistency.
5. While removing samples from the cooler, check the samples against the chain of custody for time, sample IDs, and analyses. Place samples on clean poly sheeting or clean Ziploc bags or directly into a separate cooler. When shipping samples, it is recommended to write the sample ID in permanent marker on the sample lid in case the label becomes wet and illegible.
6. Relinquish the samples by signing and writing the shipping method on the following receipt line (e.g., via Fedex, via UPS, via postal service).
7. Ensure the cooler and chain of custody contain necessary QC samples (e.g., trip blank and temperature blank).
8. Place the chain of custody in a Ziploc or similar bag and tape to inside of cooler lid.
9. Line the cooler with bubble wrap and place bagged ice along bottom of cooler. When shipping larger 1-Liter bottles, a different configuration of ice may be necessary to keep the bottles upright.
10. Wrap each sample individually in bubble wrap or place in bubble wrap bags and return to the cooler in a single layer on top of the ice. All samples should be upright. If two layers of samples are required, place two layers of bubble wrap between the sample layers. Add additional bagged ice on either end of the cooler if a larger standard size cooler (not needed for small coolers).
11. Fill the cooler to the lid with packing material such as bubble wrap such that samples are tightly packed to minimize movement while shipping.
12. Close the lid and place duct/fiber tape around the cooler on either end. DO NOT TAPE OVER HANDLES. Wrap tape around the cooler 5 times.
13. Place signed custody seal over opening point of the cooler on the front and over back hinge on opposite sides of the cooler. Write THIS SIDE UP in permanent marker on duct tape or other label.

14. Use clear packing tape to tape over the custody seal and wrap around cooler twice.

2.5 COOLER SHIPPING

The following provides guidance for shipping options for environmental samples requiring cooling:

1. Complete the shipping label and adhere to the top of the cooler/box. Ensure a cell phone number for the project manager is provided in case of issues during shipping.
2. Samples should be shipped priority overnight for delivery by 8 or 10 am the following morning. Other shipping options will not ensure delivery prior to the ice melting. Receipt of samples not on ice by the laboratory may affect the sample integrity. Uncooled samples without holding times may allow for standard shipping methods.
3. Special consideration and coordination with the laboratory is required for samples shipped on Fridays for Saturday delivery. For some labs, Saturday delivery is not available and samples requiring shipping cannot be collected on Fridays depending on hold times.
4. When dropping off samples, ensure the acknowledgment of receipt of the samples. (E.g., do not leave samples at an unattended counter or belt).

2.6 OTHER PACKING AND SHIPPING

Asbestos bulk samples, radon samples, and air canisters do not require cooling during storage and can be shipped by more typical methods. Asbestos samples can be shipped in a standard envelope or box. Air canisters and radon samples can often be return to the lab in the box/tote/container they were provided in. The following procedure should be following for shipping uncooled samples:

1. Package samples such that they are tightly packed using bubble wrap. Avoid shipping overly heavy samples in cardboard boxes. A plastic tote or cooler may be more appropriate.
2. Complete the chain of custody per Credere SOP CA-16 and compare against field notes for consistency. Relinquish the samples by signing and writing the shipping method on the following receipt line (e.g., via Fedex, via UPS, via postal service).
3. Place the chain of custody in a Ziploc or similar bag and tape to inside of cooler lid or place in box.
4. Seal the container with packing tape. Plastic totes should be closed with zip ties.

5. Write THIS SIDE UP on the top of the package, where applicable.
6. Apply signed custody seals over opening of tote or flaps of box on top and bottom. Place additional packing tape over custody seal.

3. FIELD DOCUMENTATION

The following information should be recorded in the field logbook in compliance with Credere SOP CA-1:

- Note samples were packaged for shipping
- List the shipping company used
- List the date and time of drop-off at the shipping facility
- List the requested delivery time

4. REFERENCES

Credere Associates, LLC, *Standard Operating Procedure CA-1: Field Activity Documentation*, Revision 1, dated August 2, 2016.

Credere Associates, LLC, *Standard Operating Procedure CA-3: Residential Well Sampling*, Revision 0, dated March 17, 2016.

Credere Associates, LLC, *Standard Operating Procedure CA-5: Environmental Soil Sampling*, Revision 0, dated May 27, 2016.

Credere Associates, LLC, *Standard Operating Procedure CA-12: Groundwater Sampling*, Revision 3, dated May 3, 2017.

Credere Associates, LLC, *Standard Operating Procedure CA-13: Surface Water and Sediment Sampling*, Revision 0, dated September 9, 2016.

Credere Associates, LLC, *Standard Operating Procedure CA-14: Sub-Slab Soil Gas and Indoor Air Sampling*, DRAFT, dated TBD.

Credere Associates, LLC, *Standard Operating Procedure CA-15: Asbestos Containing materials (ACM) Surveys, and Asbestos Abatement Air Monitoring and Clearances*, DRAFT, dated TBD.

Credere Associates, LLC, *Standard Operating Procedure CA-23: Collection of PCB-Containing Building Material Samples*, DRAFT, dated TBD.

New Hampshire Department of Environmental Services, *Chain-of-Custody, Sample Handling, and Shipping*, SOP No. HWRB-18, Revision 2, January 2012.

U.S. DoD, *Environmental Field Sampling Handbook, Revision 1.0*, dated April 2013.

U.S. Environmental Protection Agency, *Standard Operating Procedure for Soil, Sediment and Solid Waste Sampling*, SOP EIASOP_SOILSAMPLING2, Revision 2, dated February 13, 2004.

U.S. Environmental Protection Agency, Region 4, Science and Ecosystem Support Division, Operating Procedure, *Soil Sampling*, SESDPROC-300-R3, Revision 3, dated August 21, 2014.

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Standard Operating Procedure CA-23 Collection of PCB-Containing Building Material Samples

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Allison Drouin, Technical Review 10/25/2017 Date

Theresa Patten, QC Review 10/25/17 Date

Revision	Date	Reason for Revision

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ATTACHMENT

1. OBJECTIVE AND APPLICABILITY

1.1 OBJECTIVE

The purpose of this Standard Operating Procedure (SOP) is to provide guidance for the collection of polychlorinated biphenyl (PCB)-containing building material samples. If followed correctly, this SOP will allow for the following:

- Safety of employees performing the sampling
- Collection of representative samples with reproducible results

Credere Associates, LLC (Credere) SOPs are guidance, and state requirements, site-specific plans, and special conditions may require alternative approaches.

1.2 APPLICABILITY

This SOP should be used for the collection of building material samples during PCB-containing building materials surveys or delineation surveys.

Applicable building materials to be sampled during a PCB-containing building materials survey include, but are not limited to, caulk/sealants, wall and floor paint, and mastics/adhesives that were manufactured between approximately 1930 and 1980.

Applicable porous materials/surfaces to be sampled during delineation surveys include any surface that allows PCBs to penetrate or pass into itself including but not limited to, painted or unpainted concrete or cement; paint or coating on metal; corroded metal; fibrous glass or glass wool; unglazed ceramics; ceramics with porous glaze; porous building stone such as sandstone, travertine, limestone, or coral rock; low density plastics such as Styrofoam and low density polyethylene; coated (varnished or painted) or uncoated wood; plaster; plasterboard; wallboard; rubber; caulking; fiberboard; chipboard; asphalt; or tar paper.

PCB sample results will be compared to the 40 CFR 761.3 definition of PCB bulk product waste (50 mg/kg). Materials that have been analyzed to contain total PCBs at a concentration of equal to or greater than 1 mg/kg but less than 50 mg/kg are not regulated by the Toxic Substance Control Act (TSCA) for disposal as long as they remain in use. However, if these materials are removed from use, such as through demolition or renovation, they must be disposed of at a facility that is licensed to accept this waste. Building materials which have been analyzed to contain total PCBs at a concentration of less than 1 mg/kg are unrestricted for future use and/or disposal.

2. PROCEDURE

2.1 NECESSARY EQUIPMENT

The following is a list of required equipment:

PCB-Containing Building Materials Survey

- Various tools for the collection of paint chips
  - Wire brushes
  - Small aluminum pans to collect paint chips
  - Metal scraper
  - Chip brush
  - 5-in-1 tool and a power supply
- Various tools for collection of other building materials (caulks, sealants, mastics, adhesives, etc.)
  - Chisel
  - Hammer
  - Utility knife
  - Pliers
- Digital scale to measure grams
- Ladder
- Measuring tape
- Laser measuring device
- Flash light
- Repair materials such as caulk, silicon, etc. as necessary
- Laboratory provided glass sample containers, cooler and ice
- Decontamination supplies
- Contractor trash bags
- Paper towels
- Appropriate personal protection equipment (PPE)
  - Dust mask
  - Full face appropriately fitted respirator with P100 filter cartridges
  - Safety glasses
  - Nitrile gloves
  - Coveralls
- Dedicated vacuum cleaner with a disposable filter or a vacuum pump with a dust filter
- Building plan(s)
- Field logbook
- Chain of custody
- Ink pens/markers
- Digital camera

Delineation Survey (Porous Material Sampling)

- Rotary impact hammer variable speed drill
- Generator (if no power in the building) and fuel
- Steel chisel or sharp cutting knife
- Hammer
- Brush and cloths to clean area
- Extension cords
- Digital scale for measuring grams
- An adequate number of sample collection supply bags, which are sandwich-sized plastic bags filled with the following:
    - (1) Small aluminum pan to collect powder sample
    - (1) ¾ and ½ -inch diameter carbide tip drill bits
    - (1) Chip brush
    - (2) Pairs of nitrile gloves
    - (1) Laboratory provided sample container
- Appropriate personal protection equipment (PPE)
    - Full-body Tyvek suit(s) (optional) and duct tape
    - Steel-toe boots
    - Nitrile gloves
    - Full face appropriately fitted respirator with P100 filter cartridges (optional)
- Pasteur pipette
- Decontamination supplies
    - Hexane
    - Two small buckets
    - Scrub brush
    - Alconox or liquid pink detergent
    - Deionized water
    - Hexane laboratory squirt bottle
    - Paper towels
- Contractor trash bags
- Flash light
- Cooler and ice
- Dedicated vacuum cleaner with a disposable filter or a vacuum pump with a dust filter
- Building plan(s)
- Field logbook
- Chain of custody
- Ink pens/markers
- Digital camera

2.2 TRAINING AND HEALTHY AND SAFETY

Credere employees must have received 40-hour Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) training pursuant to 29 CFR 1910.120 and be current on their 8-hour refresher. Employees must have 24

hours of training specifically in PCB-containing building materials sampling procedures described herein and proven competence of the SOP in compliance with OSHA 29 CFR 1910.120(e)(4).

Employees will follow the Credere generic health and safety plan (HASP) and the site-specific HASP when collecting PCB-containing building materials samples.

2.3 PCB-CONTAINING BUILDING MATERIALS SURVEY

Building materials and areas to be surveyed for PCB-containing building materials are based on Site-specific objectives. Depending on the needs of the client, only certain materials for replacement, only certain rooms for renovation, or the entire building may be targeted for survey. It is crucial to understand these Site-specific objectives prior to mobilizing to the Site to complete the survey.

The following is a step-by step procedure to complete a PCB-containing building materials survey:

1. Upon arrival at the Site, begin a field logbook entry recording the Credere staff, scope of work for the day, and health and safety briefing details in accordance with Credere SOP CA-1. Keep a timeline of the day and be sure to record building locations/unit IDs/room designations etc. as the survey progresses through the Site.
2. Don appropriate PPE (safety glasses, nitrile gloves, etc.)
3. The Site/Building should first be visually inspected to identify potential PCB-containing building materials to be sampled, related homogeneous samples, and destructive or non-destructive sample locations to meet the project objectives. In conjunction with this inventory likely sample locations should be evaluated for the need for potential repairs after sampling. During this initial walk-through, Credere staff should draw out a floor plan for each Site building floor surveyed, noting potential areas for sample collection. Materials that typically contain PCBs include caulk/sealants, paint, and mastics/adhesives that were manufactured between 1930 and 1980 and are most commonly found in areas that endure high wear, weather, high heat, or moisture. There are no regulations dictating which materials must be sampled, therefore, sample collection is largely based on Credere experience and discretion. Example typical materials and locations that PCBs are encountered include, but are not limited to:
    - exterior caulks and sealants around doors and windows or within expansion joints
    - wall paints in high heat or moisture areas such as boiler rooms, equipment rooms, or basements
    - floor paints in high traffic areas such as hallways, stairways, or building entrances
    - mastics beneath floor tiles
    - certain colors of paints, including yellows and greens
4. Once potential PCB-containing building materials to be sampled per project objectives have been identified, the samples are to be collected by manual removal methods.

a. When collecting paint chip samples, use a metal scraper or 5-in-1 tool to scrape off the paint with one hand while holding a small aluminum pan underneath to collect the paint chips that fall. After the paint chips are collected into the aluminum pan, tare the digital scale with a similar empty aluminum pan. Then, weigh the aluminum pan that contains the sample. Continue sample collection to a target of 15 to 20 grams.

b. When collecting samples from building materials other than paint (mastic, caulk, glazing, etc.) use the various tools (pliers, utility knife, chisel, hammer, etc.) to collect the materials. Measure the mass of sample, ensuring that at least 15 to 20 grams of sample is submitted for analysis.

5. Samples will be placed in appropriately labeled laboratory-provided glassware. Each sample should be given a designated numeric ID (e.g., PCB-1, PCB-2, etc.).
6. Record sample time, location details (i.e. building number, room, location in room), material description (color, observed layers of paint, etc.), media condition, and quantity on the Bulk Building Material Sampling Log included as **Attachment A**. Record the sample time, volume of sample collected, and sample ID in the field logbook. Ensure the sample analysis for PCBs is indicated at least once in the entry as it will apply to all samples.
7. Perform additional inventory of homogenous materials sampled to identify all the units, rooms, and quantities of a respective material. It may also be helpful to make notes of correlations of certain materials as similar between multiple buildings/rooms at certain sites.
8. Repeat steps 4 through 7 for additional samples.
9. Complete the chain of custody in accordance with Credere SOP CA-16 using the field notes and maintain the chain of custody with the samples until provided to the laboratory.
10. Submit samples to an accredited laboratory for analysis of PCBs by EPA Method 8082 using soxhlet extraction method 3540C.

2.4 DELINEATION SURVEY AND SUBSTRATE SAMPLING

After an initial identification of TSCA regulated Bulk Product Waste (concentrations exceeding 50 milligram per kilogram [mg/kg]), the collection of delineation samples from the underlying porous materials is used to assess if PCBs have migrated into these adjacent materials. Delineation should only be performed if removal of the overlying bulk product waste is feasible, if the entire product (e.g., paint and underlying concrete) is planned to be demolished, delineation sampling is not warranted. Porous materials/surfaces include any surface that allows PCBs to penetrate or pass into itself including but not limited to, paint or coating on metal; corroded metal; fibrous glass or glass wool; unglazed ceramics; ceramics with porous glaze; porous building stone such as sandstone, travertine, limestone, or coral rock; low density plastics such as Styrofoam and low density polyethylene; coated (varnished or painted) or uncoated wood; painted or unpainted concrete or cement; plaster; plasterboard; wallboard; rubber; caulking; fiberboard; chipboard; asphalt; or tar paper. The procedure discussed herein may also

be employed during delineation surveys involving non-building material contamination source areas (e.g., liquid releases).

Credere will conduct all porous materials PCBs characterizations in accordance with the May 5, 2011, EPA Region I *Standard Operating Procedure for Sampling Porous Surfaces for PCBs*. The following is a step-by step procedure to complete a porous material PCBs characterization, which incorporates the procedures in the above referenced EPA SOP:

1. Prior to mobilization to the Site, Credere should create a figure depicting the designated sample locations for the different porous materials for each floor of the Site building(s). This figure should be included in the Site-specific sampling plan. After the total number of samples and duplicates is known, Credere staff should prepare an adequate number of sample collection supply bags (see **Section 2.1**).
2. Upon arrival at the Site, begin a field logbook entry recording the Credere staff, scope of work for the day, and health and safety briefing details in accordance with Credere SOP CA-1. Keep a timeline of the day and be sure to record building locations/unit IDs/room designations etc. as the survey progresses through the Site.
3. Don appropriate PPE (i.e., steel-toe boots, nitrile gloves, etc.).
4. Visually assess the building to identify the designated sample locations.
5. Sample collection from hard porous surfaces (e.g., concrete and brick) includes the following procedure:
    a. Lock a ¾-inch carbide drill bit into the impact hammer drill and plug the drill into an appropriate power source (using extension cords as necessary). If using a generator, ensure the exhaust is properly ventilated outside or that the generator is located away from doors and windows if positioned outside.
    b. Locate designated sample location and remove any debris with a clean brush or cloth prior to drilling.
    c. For floor samples, puncture a hole through the center of an aluminum pan to collect the powder. A team of two samplers will be required for wall and ceiling sampling. The first person will operate the hammer drilling in the designated location, applying steady and even pressure, while letting the drill do the work. The drill will provide a finely ground powder. The second person will hold a clean catch surface (small aluminum pan) below the drill to collect the falling powder. For ceilings, the drill bit can be fitted through a custom hole in the aluminum pan and the drill bit can be drilled into the ceiling at an angle while the assistant steadies the pan to catch the falling powder.
    d. Advance the drill bit 0.5 inches. A 0.5-inch deep hole generates about 10 grams (20 mL) of powder. Use a pipet to remove dust particles from the hole for inclusion in the sample. Multiple holes, located closely adjacent to each other, may be needed to generate at 15 to 20 gram sample.
    e. After the powder is collected into the aluminum pan, tare the digital scale with an empty aluminum pan. Weigh the aluminum pan that contains the sample dust.

Continue adding additional volume until 15 to 20 grams of sample are collected. Use the designated chip brush to brush all of the powder in the aluminum pan into the appropriate laboratory-provided glassware.

f. Wipe the holes of residual dust with a damp cloth and allow to dry.

g. Replace the ¾ inch drill bit with the small ½ inch drill bit and return to the prior holes to advance a second 0.5 to 1-inch sample using a clean/new aluminum pan. Use care to avoid contact with the side walls of the hole so as to sample only 0.5 to 1 inch. Return to other holes until 15 to 20 grams of sample are collected per (e) above.

h. Place samples on ice and repeat steps a through f above for additional samples.

6. Sample collection from soft porous surfaces (e.g., wood) includes the following procedure:

   a. The procedure for hard porous surfaces (described in **Section 2.4,** step 4) may be used for certain soft porous surfaces, such as wood.

   b. For other soft porous surfaces, such as caulking and rubber, samples should be collected at no more than 0.5-inch intervals using a metal chisel or sharp cutting knife. Thus, the initial surface sample should be collected from 0 - 0.5 inches.

   c. Weigh the sample using the digital scale, ensuring that at least 15 to 20 grams of sample is collected.

7. Properly dispose of used disposable equipment and PPE used for the collection of the sample (nitrile gloves, aluminum pan, chip brush). All carbide drill bits should be collected in a separate designated "decon" bag. A new sample collection supply bag will be used for each individual sample.

8. After the collection of all of the samples, samples will be continuously stored in an environment less than or equal to 4 degrees centigrade.

9. Prior to leaving the Site and loading equipment into vehicles, ensure there are no holes, tears, or puncture marks in any of the "waste" or "decon" contractor trash bags that are being removed from the Site. Also, ensure that they are tightly closed. If a hole, puncture mark, or tear is observed, double wrap the bag with another contractor trash bag and tie tightly closed. If available on-Site, dispose of the "waste" contractor trash bag(s) in an on-Site dumpster.

10. Decontamination for equipment in the "decon" contractor trash bags will take place in the field room at the office. Decontamination procedures will be in accordance with Credere SOP CA-2.

11. Complete the chain of custody in accordance with Credere SOP CA-16 using the field notes and maintain the chain of custody with the samples until provided to the laboratory.

12. Submit samples to an accredited laboratory for analysis of PCBs by EPA Method 8082 using soxhlet extraction method 3540C.

3. QUALITY ASSURANCE/QUALITY CONTROL

Quality assurance (QA) and quality control (QC) will be obtained through collection of additional samples. The types of samples to be collected are dependent on the data quality objectives and should be defined in the project's proposal/scope of work, Site-Specific Quality Assurance Project Plan (SSQAPP), regulatory/client guidance, or similar. Types of QA/QC samples that may be collected include the following:

- Field duplicates (optional)
- Matrix spike/matrix spike duplicates (MS/MSD) (optional)
- Equipment blanks (e.g., after decontaminating a drill bit)
- Temperature blanks (laboratory specific, should be included in sample coolers for certain labs)

Additionally, field documentation will be reviewed to assure it is compliant with Credere SOP CA-1: Field Activity Documentation and methods used for sampling are compliant with the protocol herein.

4. FIELD DOCUMENTATION

The following information should be recorded in the field logbook in compliance with Credere SOP CA-1:

- Date of field activity
- Credere personnel
- Scope of work
- Weather (particularly precipitation)
- Health and safety precautions
- Changes in scope or deviations from the work plan
- Correspondence with Project Managers and/or Clients
- Sketches of sample locations relative to Site features and measurements (i.e., "swing ties") from permanent landmarks where location accuracy is required
- Sample details including IDs, time of collection, requested analyses, volume of sample collected, and preservatives
- Decontamination procedures
- General timeline of field activities

5. REFERENCES

Credere Associates, LLC, *Standard Operating Procedure CA-1: Field Activity Documentation*, Revision 0, dated August 4, 2015.

Credere Associates, LLC, *Standard Operating Procedure CA-2: Equipment Decontamination*, Revision 0, dated March 17, 2016.

Credere Associates, LLC, *Standard Operating Procedure CA-16: Chain of Custody Preparation*, Draft, dated TBD.

U.S. Environmental Protection Agency, *Standard Operating Procedure for Sampling Porous Surfaces for Polychlorinated Biphenyls (PCBs)*, SOP EIASOP_POROUSSAMPLING, Revision 4, dated May 5, 2011.

BULK BUILDING MATERIAL SAMPLING LOG
CREDERE ASSOCIATES, LLC

PROJECT NAME: _____ DATE: _____

PROJECT NUMBER: _____ LOCATION ACTIVITY

On Site : _____

SITE ADDRESS: _____ Off Site: _____

Site Notes & Information:

_____

_____

_____

_____

_____

SAMPLING INFORMATION:

SAMPLE IDENTIFICATION	MATERIAL DESCRIPTION (material, color, condition, quantity)	QUANTITY	MATERIAL LOCATION (building ID, Unit)	SAMPLE DATE	SAMPLE TIME

INSPECTOR LICENSE No. _____ SAMPLER _____

BULK BUILDING MATERIAL SAMPLING LOG, FIELD FIGURE
CREDERE ASSOCIATES, LLC

Community Economy Environment
Credere Associates LLC

PROJECT NAME: _____ DATE: _____

PROJECT NUMBER: _____ LOCATION ACTIVITY

SITE ADDRESS: _____ On Site : _____

Off Site: _____

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Standard Operating Procedure CA-24 Bedrock Drilling, Well Installation, and Packer Operation

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Richard S. Vandenberg, CG, PG, Author — 10/31/2017 Date

Judd Newcomb, PG, CG, Technical Review — 10/31/2017 Date

Allison Drouin, CG, PG., Technical Review — 10/31/2017 Date

Theresa Patten, PE., QC Review — 10/31/2017 Date

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1. OBJECTIVE AND APPLICABILITY

1.1 OBJECTIVE

The purpose/objective of this Standard Operating Procedure (SOP) is to provide guidance for directing bedrock drilling and monitoring well installation. If followed correctly, this SOP will allow for the following:

- Safety of employees performing well drilling
- Collection of useful and accurate data about the bedrock formation including description of the rock encountered and calculation of Rock Quality Designation (RDQ) during well drilling
- Identification of water yielding fractures, when applicable
- Proper installation of groundwater monitoring wells within boreholes (as needed)

Credere Associates, LLC (Credere) SOPs are guidance for field staff to utilize to ensure proper methods are being employed. However, specific state and federal requirements, site-specific plans, and special conditions may require alternative approaches or amendments to this SOP.

1.2 APPLICABILITY

This SOP should be used by Credere directed staff during the drilling of bedrock boreholes, construction of bedrock monitoring wells, and/or packer testing. This SOP also provides guidance regarding the description of samples collected during drilling and coring. This SOP should be used in conjunction with Credere SOP CA-4 for overburden soil description, and documentation of field activities will be completed according to Credere SOP CA-1.

This SOP does not cover overburden soil sampling that may be required prior to encountering bedrock. See Credere SOP CA-5 for these procedures. This SOP also does not cover installation of overburden monitoring wells that may be associated with bedrock installed wells. See Credere SOP CA-8 and CA-9 for these procedures. Lastly, this SOP does not include procedures for collection of groundwater samples from the wells once installed. See Credere SOP CA-12 for these procedures.

2. PROCEDURE

2.1 NECESSARY EQUIPMENT

The following is a list of equipment for performing this work:

- Subcontractor drilling equipment
- A Straddle Packer (optional, per project objectives)
- Folding table (optional, for convenience)
- Sweeping brush
- Measuring tape/ruler
- Wooden stakes, marking paint, or pin flags
- Well construction materials
- Well completion materials (concrete, road box and/or or steel stand pipe)
- Water level meter or interface probe (for NAPL Sites)
- Decontamination fluids
- Deionized or distilled water
- One 5-gallon buckets with water and detergent, one 5-gallon bucket with water, and scrub brush (if using shovel or hand auger) or a steam cleaner
- Paper towels
- Appropriate personal protection equipment (PPE)
- Site plan
- Field logbook
- Chain of custody (if sampling)
- Ink pens
- Digital camera

2.2 TRAINING AND HEALTHY AND SAFETY

If working at a contaminated site, Credere employees must have 40-hour Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) training pursuant to 29 CFR 1910.120 and be current on their 8-hour refresher. Employees will follow the Credere generic health and safety plan and any site-specific HASP for any project, and employees must have 24 hours of training related to handling contaminated water and proven competence of the SOP and use of associated equipment (e.g. submersible pumps, generator, etc.) in compliance with OSHA 29 CFR 1910.120(e)(4).

Personnel completing bedrock drill and installing monitoring wells should have a basic understanding of geology, including, rock types, procedures for rock identification and description, and bedrock features.

2.3 TOP HOLE DRILLING & CASING GROUTING

Bedrock wells are installed into bedrock to assess hydraulic and environmental conditions deeper than in overburden. Bedrock wells can be installed with screens or be an open hole well. Open hole wells are sufficient when the bedrock aquifer in general is targeted. If bedrock is

incompetent (i.e., weathered or highly fractured) a screen may be required to maintain the structural integrity of the borehole for sampling. If a water bearing fracture or zone is targeted, it can be screened with a PVC well and the remainder of the borehole sealed off using bentonite cement grout. Targeted depths will be site-specific and will be outline in the applicable work plan.

In general, bedrock wells should be installed according to the following steps:

1. Perform drilling through overburden until bedrock is encountered. This may include split spoon soil sampling procedures as outlined in Credere SOP CA-5. Drive the temporary steel casing sufficiently into any weathered bedrock to prevent downward flow of overburden groundwater or sloughing of overburden into bedrock "socket" or "top hole" and the loss of drilling fluids or air during permanent casing installation.
2. Clear the temporary casing until free of overburden material and continue drilling into the bedrock for a minimum of five feet or until other project criteria are met.
3. Set enough permanent steel casing into the bedrock surface and to extend at least 18-inches but not more than 3 feet (unless filling around the well location is planned) above ground surface to allow for proper protection of the well and to facilitate practical sampling. Proper setting of casing is essential to prevent leakage of overburden or surface water into the bedrock aquifer. If weathered bedrock is encountered just below the overburden, it should be drilled through until 5-10 feet of competent bedrock is encountered or other project criteria are met. In this scenario, the socket or top hole should be sufficiently larger than the permanent casing to allow for the installation of a weather tight grout seal. An example would be a 7 to 8" top hole for a 6" casing. The seal is created by grouting the bottom of casing into the competent bedrock with bentonite cement grout by tremie methods until the level of the grout is confirmed to be at a level that is stable and above the top of the socket or top hole. In some cases, the entire length of casing can be grouted. Any grouted casing shall be allowed to harden based on the product specifications (i.e., fast setting grout may be used), or if unknown, for a period of at least 24-hours before continuing any drilling activities. See **Figure A** for an example of casing installation construction.

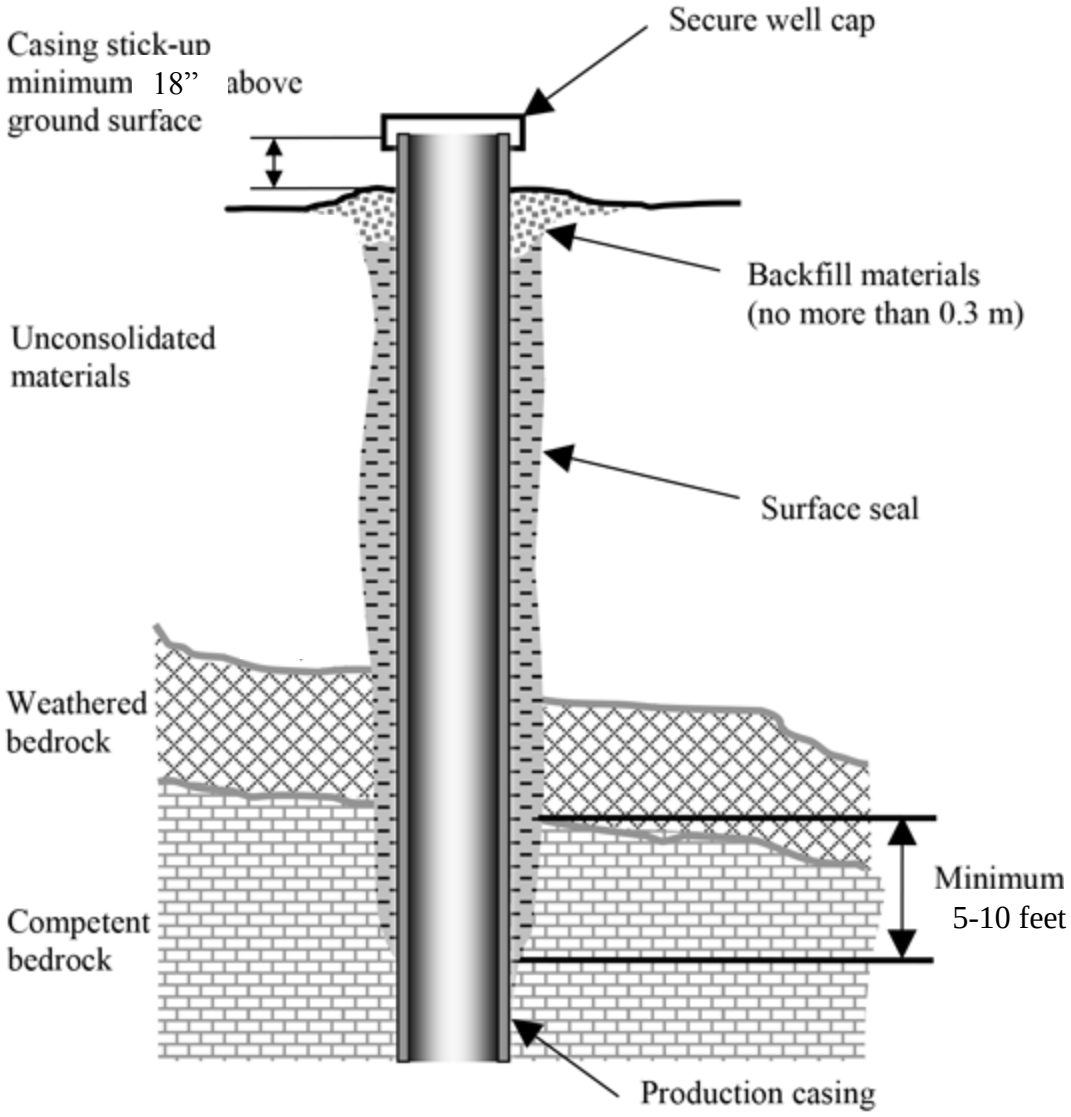


Figure A: Casing Construction Diagram

4. Continue drilling through bedrock using a method described in **Section 2.4** to the desired depth. Continuously describe/characterize bedrock on the boring log included as **Attachment A** according to the bedrock description card included as **Attachment B.** Note water bearing fractures and rock quality while drilling. Drill to the desired well completion depth.

5. If the monitoring well is to be a screened or multi-screened well, place the deepest screen at the desired intake depth, fill the annulus with the specified filter media (i.e., silica sand, gravel, etc.), and place a grout or bentonite seal above it. This will ensure an adequate seal and prevent communication and cross-contamination between target zones. Cement grout is to be tremie piped between wells in addition to any bentonite. This will help ensure a good seal. If the borehole is to contain multiple screens (i.e., nested wells), continuing adding silica sand or bentonite to the bottom depth of the next targeted screened interval. Place the second screen at the desired depth, fill the annulus with the specified filter media, and place a grout or bentonite seal above it. Repeat this process of building toward the surface until all the desired screens are installed. See **Figure B** for an example of basic nested well construction.

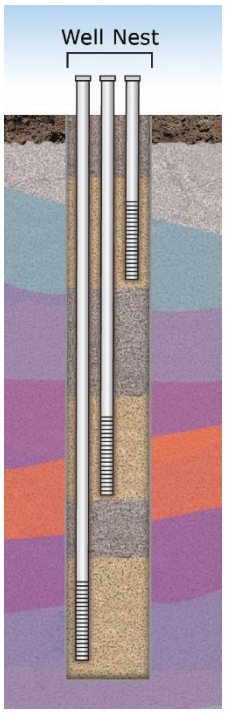


Figure B: Nested Wells

2.4 ACCEPTABLE BEDROCK DRILLING METHODS

Once the casing is installed, there are several methods of drilling that are acceptable. The most typical options employed on Credere projects can be divided between rotary methods and coring

Rotary Methods

Rotary methods consist of a drill pipe or drill stem coupled to a drilling bit that rotates and cuts through the soil/rock. The cuttings produced from the rotation of the drilling bit are transported to the surface by drilling fluids which generally consist of water, drilling mud, or air. The drilling fluid is forced down through the drill pipe and out through the bottom of the drilling bit. The cuttings are then lifted to the surface between the borehole wall and the drill pipe (or within a concentric drill stem in reverse rotary). The drilling fluid also keeps the drilling bit cool.

When considering this method, it is important to evaluate the potential for contamination when introducing material into the borehole. If the rotary method is selected, water rotary is the preferred method, followed by air rotary, and mud rotary as a last option. Water and mud rotary methods present the possibility of trace contamination of halogenated compounds when municipal water supplies are used as a potable water source. Air rotary drilling can introduce contamination using lubricants or entrained material in the air stream. In any of the rotary methods, care must be exercised in the selection and use of compounds to prevent galling of drill stem threads.

Air Rotary Drilling Method

Air rotary drilling uses air as the drilling fluid, requiring high air velocities and consequently using large air volumes and an air-compressor. In conventional air rotary method, the drill bit is rotated rapidly (similar to water and mud rotary drilling) to cut the formation of material and advance the borehole. The drill bit is attached to hollow drilling rods that transfer power from the rig to the bit. In a "hammer" or "down-the-hole" air rotary method, the bit is pneumatically driven rapidly against the rock in short strokes while the drilling string slowly rotates. In both types of air rotary drilling, filtered air is forced down the drill rods by an air compressor, escapes out of the bit and returns to the surface in the annular space between the hole wall and the drill string. Cuttings are moved out of the hole by the ascending air and collect around the rig. Cuttings are mixed and may not always be representative of the depth currently being drilled.

The use of air rotary methods is generally limited to consolidated and semi-consolidated formations. Casing is often used in semi-consolidated formations and through the weathered portion of consolidated formations to prevent hole collapse.

Water Rotary Drilling Method

Water rotary drilling is the same as conventional air rotary, except water is the drilling fluid. The drill bit is rotated rapidly to cut the formation of material and advance the borehole. Cuttings are removed by pumping water down through the drill rods, through the bit, and up the annulus between the borehole and the drill rods. The drilling fluid flows into a re-circulating bucket where the cuttings settle out and is then pumped back down the drill rods. The drilling fluid also cools the bit and prevents the borehole from collapsing in unconsolidated formations.

Sampling may be done from the cuttings, but samples are generally mixed, comingled with water, and the amount of fine material may not be accurately represented. Once the well is constructed, extensive well development may be necessary to removed drilling water from the formation. A water quality meter will be used to monitor dissolved oxygen, oxidation reduction potential, pH, specific conductivity, and temperature until stable.

Mud Rotary Drilling

Mud rotary drilling is not a preferred drilling method for environmental drilling of bedrock because it is difficult to remove the drilling mud from the borehole after drilling and during well development. This is an acceptable methodology for drilling the overburden above bedrock. The viscus mud tends to keep the borehole open during casing installation.

In mud rotary drilling, the drill bit is rotated rapidly to cut the formation of material and advance the borehole. The drill bit is attached to hollow drilling rods that transfer power from the rig to the bit. Cuttings are removed by pumping drilling mud (potable water mixed with pure [no additives] bentonite) down through the drill rods, through the bit, and up the annulus between the borehole and the drill rods. The drilling mud flows into a re-circulating tank where the cuttings settle out and is then pumped back down the drill rods. The drilling mud also cools the bit and prevents the borehole from collapsing in unconsolidated formations.

Sampling may be done from the cuttings, but samples are generally mixed, comingled with drilling mud, and the amount of fine material may not be accurately represented. Once the well is constructed, extensive well development may be necessary to remove drilling mud from the formation and turbidity is the best indicator of development success. Also, a water quality meter will be used to monitor dissolved oxygen, oxidation reduction potential, pH, specific conductivity, and temperature until stable.

Core Drilling Methodology

In certain circumstances, it will be desirable to drill and install the casing into bedrock using rotary techniques and then drill a small open hole into bedrock by coring. Coring allows for the continuous collection of representative samples of the bedrock and provides an open borehole that can serve as a monitoring well that can be fitted with a PVC/steel monitoring well if conditions warrant (i.e., soft bedrock that is prone to collapse or targeted screened depths).

Bedrock coring involves the advancement of a rotary diamond core barrel into the rock accompanied by a drilling fluid such as water or viscous high-gel strength muds. In New England, muds are usually only needed when the native bedrock is soft and can be eroded via injection of high pressure water or when the native rock is too permeable and excessive water is lost to the formation. The diamond core barrel is generally attached to the drill pipe and spun into the rock until a seal is formed. At this point, drill rig down pressure and rotational speed are increased. The optimal down pressure and rotational speed are dependent on the bedrock being cored and the size of core barrel being used and will generally be managed by the driller. The preferred size of the core barrel is NX but other sizes may be specified to meet the objectives of the project. The core barrel/drill rods should be marked in 1-foot increments and the time to advance each foot should be recorded on the boring log.

The core barrel is then tripped out of the hole, taken apart, and the bedrock core removed. Use a of wire line system can reduce rod trip out during coring. Cores are generally extruded from the barrel into a half round trough or open wooden box for description. Reassemble the core pieces and draw match lines on the core using permanent marker once the core is dry.

Coring can continue in the borehole until the target depth is achieved. In some cases, the borehole may require reaming (i.e., widening) to clear obstructions caused by poor rock quality or to create a larger diameter well. The borehole can either be left as an open hole (assuming that the casing is already grouted into place) or a smaller diameter well can be constructed in the borehole.

2.5 ROCK DESCRIPTION & ROCK QUALITY DESIGNATION

The bedrock will be viewed with a hand lens and descriptively logged by the on-Site Credere field staff. The type of rock will be described according to **Attachment B** and the rock type will be determined through use of an external rock identification guide. The angle of joints and fractures in the sample will be noted. Logs will detail any significant lithologic changes in the rock.

When the project requires rock coring, Rock Quality Designation (RQD) logging techniques will be used to assess the quality of the bedrock for each core sample. The RQD was first introduced in the mid-1960s to provide a simple and inexpensive general indication of rock mass quality to predict tunneling conditions and support requirements. The recording of RQD has since become virtually standard practice in drill core logging for a wide variety of geotechnical explorations. The use of RQD values are used to define the quality of the rock encountered and is used to provide a basis for making preliminary design and constructability decisions involving excavation for foundations of structures, or tunnels, open pits, and many other applications. The RQD values also can serve to identify potential problems related to bearing capacity, settlement, erosion, or sliding in rock foundations. The RQD can provide an indication of rock quality in quarries for issues involving concrete aggregate, rockfill, or large riprap. Specific to this SOP, the RQD can serve as a factor in determining if a well can be left as an open hole or if a screen is required to maintain well integrity.

Credere staff should follow ASTM D6032/D6032M-17 to calculate the RQD of each rock core that is deemed appropriate for measurement. This test method covers the determination of the RQD as a standard parameter in drill core logging. The procedure, formula, and quality ranges are included on the bedrock description card in **Attachment B**.

2.6 WELL DEVELOPMENT

Well development will be performed following Credere SOP CA-9. In general, this may include first developing the well by surging and purging the borehole with the drill rig, then by pumping the open hole or installed wells with a submersible or other pump in accordance with the project specifications until certain parameters are met. See Credere SOP CA-9: Well Development for more detailed instructions for well development.

2.7 MONITORING WELL INSTALLATION (AS NEEDED)

Where a bedrock monitoring well is required to be installed with the open borehole, well materials should be chosen based on the goals and objectives of the proposed monitoring program and the geologic conditions at the site(s). However, Schedule 40 PVC has been determined to be appropriate for nearly all applications to depths of up to approximately 100 feet and Schedule 80 PVC when deeper wells are required. It will be documented in the work plan when any non-PVC well construction materials are specified. The diameter of the PVC will be selected based on the specific goals of the project, anticipated depth of the well, and specific requirements of various entities overseeing the project (i.e., state and federal agencies). Credere's preferred bedrock monitoring well diameter is between 2 and 4-inches. However, there are some circumstances where less than 2-inch monitoring wells may be appropriate. The diameter of bedrock monitoring wells will be specified in the work plan.

When monitoring wells are constructed within open boreholes, a filter pack will be placed to fill the annulus and be positioned to coincide with the screened interval. The filter pack materials should consist of clean, rounded to well-rounded, hard, insoluble particles of siliceous composition. The required grain-size distribution or particle sizes of the filter pack materials will be selected to pass between 0 and 1% during development. A 2 to 3-foot bentonite seal will

be placed above the filter pack and at least 1-foot above the screened interval. Depending on the depth of the well, the work plan may require that the seal include cement grout that is tremie piped above the bentonite.

Credere personnel are required to provide the following measurements for each monitoring well (where applicable) on the bedrock well construction log provided as **Attachment C**:

- Total depth of the borehole
- Diameter of the borehole
- Diameter of the casing
- Total depth of the casing
- Screened interval depth
- Depth interval of the filter pack
- Depth interval of bentonite/grout seals
- Well casing stickup height
- Depth to water measured in the well immediately following well construction

2.8 WELL DECOMMISSIONING

When a decision is made to decommission (abandon) a bedrock monitoring well, the borehole should be sealed in such a manner that the well cannot act as a conduit for migration of contaminants from the ground surface to the water table or between aquifers. To properly decommission a well, the preferred method is to completely remove the well casing and screen from the borehole, clean out the borehole, and tremie with a cement or bentonite grout, neat cement, or concrete.

To comply with state well decommissioning requirements, the appropriate state agency should be notified (if applicable) of monitoring well decommissioning. However, some state requirements are not explicit, so a technically sound well abandonment method should be designed based on the site geology, well casing materials, and general condition of the well(s). Absent any specific state requirements, any abandonment work should follow these general guidelines:

The preferred method is to completely grout the entire length of the open borehole with a Portland cement and bentonite grout mixture. The well casing can be removed toward the end of this operation if possible. If it cannot be removed, it should be cut off approximately 18 inches below grade and buried with native material. Wells constructed in concrete or asphalt will be backfilled with structurally appropriate fill and cold patched with asphalt or restored with concrete to match the surrounding features.

For bedrock monitoring wells with PVC or metal screens and casing in the borehole, an attempt should be made to remove this material prior to abandoning the well. It may be possible to use drill rods to break the bottom of the casing (with PVC) and then tremie in the grout using the monitoring well casing as the tremie pipe. As an alternative, a smaller diameter casing can be used inside the monitoring well casing. The casing and screen (and tremie pipe) can be pulled

out during the grouting process by wrapping a chain around the casing and using the drill rig to pull it out. If the casing breaks off during removal, the remaining casing and annulus should be filled with cement/grout and any casing cut off approximately 18 inches below grade and buried with native material.

Any deviations from this general guideline shall be recorded in the field notebook in accordance with CA-1.

2.9 PACKER DEPLOYMENT AND TESTING

It may become necessary to assess the hydraulic characteristics or collect samples of groundwater from a specific zone or zones within an open hole bedrock well prior to actual construction of a final well. A borehole straddle packer is generally the tool of choice to isolate portions of a borehole for testing. Please note that the use of packers is not recommended for overburden wells that employ highly transmissive filter packs.

This type of packer has upper and lower air-deployed inflatable bladders mounted on PVC or steel piping with screened piping in the interval between the packers. In the center of the system is an air-driven or electric submersible pump to collect water from between the packers from the center piping. The type and number of packers used will be specified in the project work plan. The packer system may also be equipped with a variety of instruments to measure the characteristics of the aquifer and/or packer system performance (e.g., pressure transducers to assess packer leakage).

The packers are generally deployed by a geophysicist or by a licensed well driller using winch system, drill rods, and in some cases a well pump truck. Once the packers are deployed to the appropriate depth, the bladders are inflated per the manufacturer's specifications and the necessary hydraulic testing or environmental sampling. For groundwater sampling, please see Credere's SOP CA-12 recommended low flow purging methods.

3. INVESTIGATION DERIVED WASTE

Investigation derived waste (IDW) generated during drilling includes drill cuttings, development water, and personal protective equipment. All investigation derived waste should be managed in accordance with the project contract documents, site-specific quality assurance project plan (SSQAPP), or other similar client or agency approved document. In the case of mildly impacted sites, IDW management typically includes the disposal of expendable materials as general trash and the spreading of drill cuttings and discharge of groundwater to the area around or upgradient of the wellhead within the source area. However, in cases where non-aqueous phase liquid (NAPL) is present or high concentrations of contaminants have been identified, IDW may be treated onsite or containerized and sampled for off-site disposal.

4. QUALITY ASSURANCE/QUALITY CONTROL

Quality assurance (QA) and quality control (QC) will be maintained through the collection of manual data to ensure that is matches with the scope of work/site-specific quality assurance project plan. Additionally, field documentation will be reviewed to assure it is compliant with Credere SOP CA-1: Field Activity Documentation and methods used for sampling are compliant with the protocol herein.

5. FIELD DOCUMENTATION

The following information should be recorded in the field logbook in compliance with Credere SOP CA-1:

- Date of drilling
- Credere personnel
- Pertinent details of scope of work
- Changes in field conditions
- Weather
- Equipment used
- Changes in scope
- Correspondence with Project Managers
- Drilling location and monitoring well designation
- Total depth of the borehole
- Diameter of the borehole
- Diameter of the casing
- Total depth of the casing
- Screened interval depth
- Depth interval of the filter pack
- Depth interval of bentonite/grout seals
- Well casing stickup height
- Depth to water measured in the well immediately following well construction
- RQD description of bedrock encountered
- Field screening results, as needed or required
- Volumes of water removed during development
- Decontamination procedures
- IDW information (e.g. sampling, volume, container size and storage location, etc.)
- General timeline of field activities

Attachment A and **Attachment C** field forms will be used to facilitate collection of the above information.

6. REFERENCES

Credere Associates, LLC, *Standard Operating Procedure CA-1 Field Activity Documentation*, Revision 0, dated August 4, 2015.

Credere Associates, LLC, *Standard Operating Procedure CA-2 Equipment Decontamination Procedure*, Revision 0, dated March 17, 2016.

Credere Associates, LLC, *Standard Operating Procedure CA-4 Soil Description*, Revision 0, dated March 17, 2016.

Credere Associates, LLC, *Standard Operating Procedure CA-10 Monitoring Well Gauging*, Revision 0, dated August 29, 2016.

Credere Associates, LLC, *Standard Operating Procedure CA-12 Groundwater Sampling*, Revision 2, dated October 3, 2017.

US Army Corps of Engineers, *Rock Quality Designation (RQD) after Twenty Years*, Rocky Mountain Consultants, Inc. Contract Report GL-89, dated February 1989.

ASTM D2113-14, Standard Practice for Rock Core Drilling and Sampling of Rock for Site Exploration, ASTM International, West Conshohocken, PA, 2014, www.astm.org

ASTM D6032 / D6032M-17, *Standard Test Method for Determining Rock Quality Designation (RQD) of Rock Core*, ASTM International, West Conshohocken, PA, 2017, www.astm.org.

USEPA, E*nvironmental Investigations Standard Operating Procedures and Quality Assurance Manual*, USEPA Region 4, dated November 2004.

United States Geological Survey, *Techniques of Water-Resource Investigations of the United States Geological Survey Chapter F-1 Application of Drilling, Coring, and Sampling Techniques to Test Holes and Wells*, dated 1989.

Driscoll, F.G. 1986. Groundwater and Wells. 2nd ed. Johnson Division, UOP Inc., St. Paul, MN. p. 1089.

Credere Associates LLC

SITE INFORMATION	WELL SPECIFICATIONS
Project Number/Client:	**Well Depth (feet bgs):**
Site Location:	**Screen Length (feet):**
Date Start/Finish:	**Annulus materials:**
Credere Representative:	**DRILLING EQUIPMENT**
CONTRACTOR	**Equipment (make/model):**
Drilling Contractor:	**Casing/Auger/Core Diameter:**
Foreman:	**Casing Material:**

Boring/Well ID:

TOC Elevation:
TOR Elevation:
GS Elevation:

Depth	Sample Information: Sample No./Type	Pen/Rec (inches)	Depth (feet)	Blows /6" or time/ft	PID (ppm) (RF=1.0)	Soil/Bedrock Description and Classification
5						
10						
15						
20						
25						

Remarks and Well Details

Boring No:

CREDERE ASSOCIATES, LLC FIELD BEDROCK IDENTIFICATION

Blue-gray
Light-gray
Gray
Dark-gray
Olive-gray
Greenish-gray
Reddish-brown
Yellowish-red
Brownish-yellow
Dark-brown
Brown
Light-brown
Beige
Tan

MODIFIERS

Parting	0 to 1/16 inch thick
Seam	1/16 to ½ inch thick
Layer	½ to 12 inches thick
Stratum	Greater than 12 inches thick
Pocket	Small, erratic deposit, <1’
Varve	Alternating seams of sand, silt and clay
Occasional	One or less/foot of thickness
Frequent	More than one/foot of thickness
Stratified	Alternating layers of varying material or color with layers at least ¼” thick
Laminated	Alternating layers of varying material or color with the layers less than ¼” thick
Mottled	Blocky color variation
Homogeneous	Same color and appearance throughout

EXAMPLE ROCK DESCRIPTION

25-50’ Hardness, weathering, color, texture ROCK TYPE, structure, modifiers (RQD).

TEXTURE

Aphanitic	too small for naked eye
Fine grained	visible, 1/16 inch
Medium grained	1/16 to ¼ inch
Coarse grained	>1/4 inch

ADVANCE RATE

Number of minutes spinning rock core takes to advance

ROCK SAMPLE TYPES

C Core sampler

HARDNESS SCALE

Mohs Scale	Mineral	Common Object	Rock Hardness	Description
1	Talc	-	Very Soft	Can be carved with knife or fingernail. Can be excavated readily with point of pick. Pieces >1 inch can be broken with finger pressure.
2	Gypsum	Fingernail (2.2)		
3	Calcite	Copper penny (3.5)	Soft	Can be gouged or grooved readily with knife or pick. Can be excavated in chops to pieces several inches in size. Small thin pieces can be broken with finger pressure.
4	Fluorite	-		
5	Apatite	Hammer (5.1) Knife (5.2) Glass (5.5)	Medium	Can be grooved 1/16 inch deep with pressure on knife. Can be excavated in small chips to pieces about 1-inch max size by hard blows with pick.
6	Feldspar	-	Moderately Hard	Can be scratched with knife or pick. Gouges or grooves ¼ inch deep with hard blows with pick. Hand specimen can be detached with hard blow.
7	Quartz	-		
8	Topaz	-	Hard	Can be scratched with knife or pick only with difficulty. Hard blow required to detach hand specimen.
9	Corundum	-	Very Hard	Cannot be scratched with knife or pick. Hand specimen requires several hard blows with pick.
10	Diamond	-		

STRUCTURE

Bedding	Layering in sedimentary rocks (limited applicability in low grade metamorphic and volcanic rocks
Foliation	Planar fabric homogenously distributed, typical of metamorphic/secondary rocks
Fracture	Natural break in the rock, note angles
Crack	Partial or incomplete fracture, note angles
Joint	Fracture without displacement, note angles
Shear	Fracture with parallel minor displacement
Fault	Fracture with significant displacement
Fault/shear zone	Zone containing numerous faults and shears
Void	Loss of rock due to weathering or dissolution
Secondary mineralization	Occurs on weathered fractures

ROCK QUALITY DESIGNATION (RQD)

Realign the rock core and draw match points with a permanent marker. Using a 2 inch core, sum the length in inches of rock core pieces ≥4 inches (sound pieces) by natural processes (do not consider mechanical/handling breaks). Divide by the total recovered length of the core.

$$RQD = \frac{\Sigma \text{ length of sound pieces (in)}}{\text{total core length (in)}} \times 100\%$$

0-25% Very poor, very highly fractured
25-50% Poor, highly fractured
50-75% Fair, moderately fractured
75-90% Good, slightly fractured
90-100% Excellent, slightly fractured to massive

WEATHERING

Fresh: No visible sign of decomposition or discoloration. Rings when struck by hammer

Slightly Weathered : Slight discoloration inwards (up to 1 inch) from open fractures, may contain clay.

Moderately Weathered: Discoloration throughout, weaker minerals decomposed, core cannot be broken by hand, texture preserved. Dull sound under hammer.

Highly Weathered: Most minerals somewhat decomposed. Specimens can be broken by hand with effort or knife. Texture less distinct, fabric preserved. “Clunk” when struck.

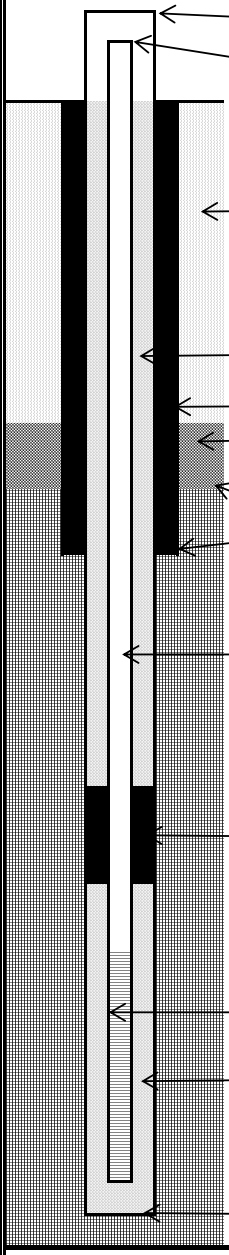
Completely Weathered: Advance state of decomposition resulting in plastic soil. Fabric and structure destroyed. Strong soil (saprolite).

Credere Associates LLC

Boring/Well ID:

SITE INFORMATION	DRILLING EQUIPMENT
Project Number/Client:	**Equipment (make/model):**
Site Location:	**Casing/Auger/Core Diameter:**
Date Start/Finish:	**Casing Material:**
Credere Representative:	**CONTRACTOR**
	Drilling Contractor/Foreman:

BEDROCK WELL CONSTRUCTION DETAILS



Top of casing elevation: _____

Stickup height (in): _____ Lock installed? Yes No

Top of well elevation: _____

Ground elevation: _____

Generalized description of overburden: _____

Overburden thickness (ft): _____

Casing type: steel

Casing diamter (in): _____

Borehole diameter (in): _____

Backfill material: _____

Backfill depth (ft): _____

Grout material: _____

Weathered bedrock thickness (feet): _____

Generalized decription of rock type (fracture depths, etc.): _____

Depth to competent bedrock (feet bgs): _____

Depth of casing into bedrock (ft): _____

Well riser material: _____ Development method: _____

Well riser length: _____ Total well volume: _____

Total purge volume (g): _____

No. of well volumes purged: _____

Depth to water (ft bgs): _____ Final turbidity (NTU): _____

Field Parameters

Bentonite depth interval (ft): _____ DO (mg/L): _____

pH: _____

Borehole diamter (in): _____ Sp. Conductivity (mS/cm): _____

ORP (mV): _____

Screen material: _____ Temperature (°C): _____

Well diamter (in): _____

Screen length: _____

Annulus material: _____ Annulus material size: #00 #1 #2 G

Annulus depth interval (ft): _____

Depth of open borehole (ft): _____

Remarks and Well Details

Well ID:

CREDERE ASSOCIATES, LLC
776 Main Street
Westbrook, Maine 04092
Phone: 207-828-1272
Fax: 207-887-1051

Standard Operating Procedure CA-26
Incremental Sampling Methodology

Effective Date: October 31, 2017
Revision: 0

Richard S. Vandenberg, Author — 10/31/2017 Date

Allison Drouin, Technical Review — 10/31/2017 Date

Theresa Patten, Technical Review — 10/31/2017 Date

Revision	Date	Reason for Revision

TABLE OF CONTENTS

1. OBJECTIVE AND APPLICABILITY

1.1 OBJECTIVE

The primary objective of this Standard Operating Procedure (SOP) is to detail the methods to be employed for the collection of soil samples using Incremental Sampling Methodology (ISM). Successful implementation of ISM in the field requires execution of several planning elements and sample preparation procedures with the analytical laboratory prior to entering the field. As a result, this document does provide some general background/guidance in these areas. If followed correctly, this SOP will allow for the following:

- Collection of representative samples with reproducible results that represent a predefined decision unit.

Credere Associates, LLC (Credere) SOPs are guidance for field staff to utilize to ensure proper methods are being employed. However, specific state and federal requirements, site-specific plans, and special conditions may require alternative approaches or amendments to this SOP.

1.2 APPLICABILITY

This SOP shall be used during soil sampling activities employing ISM. ISM will be implemented at sites where a structured composite methodology is needed to provide reasonably unbiased, reproducible estimates of the mean concentration of analytes in the specified volume of soil. ISM can also be used for the sampling of shallow and deep sediment. As such, this SOP is only applicable for projects that employ this method of surface and subsurface soil sampling. This approach is not applicable for sampling other environmental media such as groundwater or soil vapor.

Grab and standard composite sampling are summarized in SOP CA-5. This SOP does not cover procedures for description of soil or field screening, which are provided in Credere SOPs CA-4: Soil Description and CA-7: Headspace Field Screening, respectively.

Portions of this SOP have been taken directly from the Interstate Technology Regulatory Council (ITRC) Technical and Regulatory Guidance for ISM dated February 2012 (ITRC Guidance).

2. BACKGROUND, DEFINING INCREMENTAL SAMPLING METHODOLOGY & OTHER TERMS

Incremental sampling methodology (ISM) is a structured composite sampling and processing protocol that reduces data variability and provides a reasonably unbiased estimate of mean contaminant concentrations in a volume of soil targeted for sampling. ISM provides representative samples of specific soil volumes defined as decision units (DUs) by collecting numerous increments of soil (typically 30–100 increments) that are combined, processed, and subsampled according to specific protocols. More information on ISM can be obtained through ITRC's website at http://www.itrcweb.org/ism-1/Executive_Summary.html.

Variability in measured contaminant concentrations between discrete soil samples has been determined to be primarily due to the particulate nature of soil and heterogeneric distribution of contaminants resulting from releases or filling. The elements of ISM that control data variability are incorporated into (a) the field collection of soil samples and (b) laboratory processing and subsampling procedures. If used correctly, a single aliquot is obtained from each defined area/volume of soil. This sample theoretically has all constituents in the same proportions. Properly executed, the methodology provides reasonably unbiased, reproducible estimates of the mean concentration of analytes in the specified volume of soil.

Like all sampling approaches, ISM should be applied within a systematic planning framework. One of the first steps in such a framework is to have the investigation project team establish a working conceptual site model (CSM). Once the CSM has been agreed to, the project team defines the data quality objectives (DQOs) and determines the appropriate decision unit (DU) size(s) and location(s). DUs are based on project-specific needs and site-specific DQOs; both considerations specify and constrain the appropriate end use of the data. The size of a DU is site-specific and represents the smallest volume of soil about which a decision is to be made (USEPA 1999, Ramsey and Hewitt 2005, HDOH 2008a, ADEC 2009). In some cases, a DU is composed of smaller units known as sampling units (SUs). The requirement to explicitly and appropriately define the DU that each incremental sample represents is a key component of ISM.

It is important to understand the meaning of the following terms. The definitions of these terms were taken directly from the 2012 ISM ITRC guidance.

Composite sample – A sample composed of two or more increments, which generally undergoes some preparation procedures designed to reduce the variance in the errors associated obtaining a measurement from the combined sample. An ISM sample is a composite sample whose collection and preparation steps are designed using the general suggestions of Gy's sampling theory. In general, composite samples in environmental studies do not consist of a large volume, nor a large number of increments, and do not undergo the same preparation and subsampling steps suggested by Gy's sampling theory.

Decision unit (DU) – The smallest volume of soil (or other media) for which a decision will be made based upon ISM sampling. A DU may consist of one or more sampling units (SUs).

Exposure point concentration (EPC) – The value, based on either a statistical derivation of measured data or modeled data, that represents an estimate of the chemical or radionuclide concentration available from a particular medium or route of exposure.

Exposure unit (or exposure area) – For purposes of risk assessment, a defined area throughout which a potential receptor may be exposed to a contaminant. The receptor is assumed to move randomly across the area, being exposed equally to all parts of the area. The assumption of equal exposure to any and all parts of the exposure area is a reasonable approach (USEPA, 1992) that allows a spatially averaged soil concentration to be used to estimate the true average concentration contacted over time.

Field replicate samples – Samples collected following the same the process within the DU but from a different set of locations. The manner in which the replicate is collected is determined during systematic planning. The purpose of the collection of replicates is to provide multiple estimates of the mean.

Grid cell – A grid cell is a sub-division of the Sampling Unit (see below). Sampling units are divided into uniform-size grid cells, and one increment is collected from each cell, from the same relative location within each grid cell. The shape of the cells is not specified; the only criterion for cell shape selection is that the cells should be of equal size (they can be triangular, square, rectangular, etc.) so the increments collected from each cell are equally weighted over the SU.

Lot – An explicitly defined volume of soil from which incremental samples are collected to estimate the mean concentrations of analytes of interest present. The Lot changes during the ISM process; the initial Lot may be immovable, such as the soil of a residential plot, or movable, such as the incremental field sample being processed and sub-sampled in the laboratory.

Replicate (duplicate) sample – one of the two or more samples or subsamples obtained separately at the same time by the same sampling procedure or subsampling procedure.

Sampling unit (SU) – A user-defined volume of soil (or other media) from which increments are collected to determine an estimate of the mean concentration for that volume of soil (or other media).

3. SYSTEMATIC PLANNING AND DEVELOPING DECISION UNITS

ISM is useful when practical constraints limit the number of discrete samples that can be collected and, therefore, limit the precision with which the mean concentration in heterogeneous matrices may be estimated. Most action levels are derived from risk-based receptor models that assume a specific exposure scenario in a given area. In these cases, estimates of mean concentrations in volumes of media are generally the appropriate statistic to compare to action levels.

It is important to match the project objectives with the type of sampling employed. For some objectives, discrete sampling is appropriate (when sufficient numbers of discrete samples are used); for other's ISM sampling may be the best/most appropriate option. In certain situations, a hybrid of the discrete and ISM may be the most advantageous. For example, discrete samples might be used to make decisions on obviously contaminated volumes of soil in which contaminant concentrations are very likely to exceed action levels. Even though contaminant concentrations in this situation may be highly variable, this variation would not result in decision errors since any possible sample collected from the volume will likely have contaminant concentrations above the action level. Discrete samples may also be used to estimate the variability within a DU prior to ISM sampling. When field analytical methods (or other cost-saving analytical approaches) are available, sufficient numbers of discrete samples may be used to characterize some contaminants or DUs, while ISM may be appropriate for those contaminants for which these analytical approaches are not available.

3.1 ISM PLANNING

The use of ISM to characterize the soil within a DU can provide higher-quality data and fewer decision errors than conventional discrete or composite sampling designs which is lower density data. In combination with well-conceived investigation objectives and DU and SU designations, incremental samples will reduce the need for additional sample collection, will increase the certainty of decisions, and will reduce the time and money required to complete environmental projects. Although a project team may have an ISM strategy in mind during initial planning, a number of sampling and analysis options should still be considered, and the sampling strategy selected should be an outcome of the systematic planning process.

A systematic planning process will serve to identify the objectives of the site investigation and establish the type of information needed to make environmental decisions. The level of detail needed to adequately incorporate a systematic planning approach into a data collection effort varies from project to project; larger or more complex projects usually warrant more detailed planning than smaller, simpler projects. The nature of the ISM process is such that many decisions have to be made and detailed plans established in advance of sample collection. For these reasons, the principles of the systematic planning approach should be applied on every ISM project. Please note that the specifics of the systematic planning approach will be detailed in the project scope of work.

3.2 USE OF THE CONCEPTUAL SITE MODEL (CSM) IN ISM

CSMs are essential elements of the systematic ISM planning process. A comprehensive CSM serves to conceptualize the relationship between contaminant sources and receptors through consideration of migration and exposure pathways (potential and actual). The CSM also presents the current understanding of the site, helping to identify data gaps, and focus the data collection needs. The CSM should be maintained and updated as additional information is collected throughout the project. A Credere CSM describes site conditions, identifies the sources areas and contaminants of potential concern (COPCs), estimates the extent of contamination, and seeks to describe the exposure and migration pathways for current and assumed future site conditions. The sampling strategy should reflect the assumptions about the transport phenomena and exposure scenarios articulated in the CSM.

3.3 DEFINING DECISION UNITS

A clear understanding of the study objectives is important with all sampling strategies, but particularly so with ISM sampling. Different objectives will dictate the type, location, dimensions of DUs, and number of aliquots per decision unit. For example, small source-area DUs are important for highly mobile chemicals that can pose significant vapor intrusion or leaching risks. Larger exposure-area DUs or subsurface DUs can be appropriate to evaluate risks to specified receptors. The decision for additional investigation or remedial action might be made based on a comparison of ISM sample results to published screening levels. In other investigations the estimate of the mean contaminant concentration provided by ISM samples might be used to estimate the risk to human or ecological receptors. ISM results may also be used to estimate background concentrations, to assess sources, or to evaluate various stages of remedial activities.

There are various approaches to defining DUs. The approach selected should be consistent with the understanding of the site and support the objectives of the investigation. According to ITRC, DUs can be defined in regularly spaced and equal volumes as established by exposure areas, or they can be based on irregular features of the site that define contaminant transport or receptor exposure.

DUs may be based on the understanding of the known or suspected distribution of contaminants. This can be used in and around source areas. Volumes of soil known or suspected to be contaminated are generally good candidates for designation as DUs because the decision over these volumes is best made separately from less-contaminated surrounding volumes. Human health or ecological exposure areas may also provide the basis for the designation of DUs. This approach has the advantage of being supported by the exposure assumptions that are used to develop most risk-based action levels.

DUs developed and based on the needs of remediation or excavation are appropriate. Examples include, using sidewalls and excavation floors as DUs to determine whether soil removal was sufficient.

Selection of DUs need to also consider geologic aspects of the Site defined in the CSM. If boundaries between different geologic formations are important for contaminant transport or exposure, they may provide a logical demarcation of the DU. However, a DU can extend across more than one geologic formation or soil type.

Two primary types of DUs according to ITRC include:

1. Those based on the known or suspected locations and dimensions of source areas, called "source area DUs"
2. Those based on the size assumptions of risk assessment, called "exposure area DUs"

A source area is defined here as a discernible volume of soil (or waste or other media) containing elevated or potentially elevated concentrations of contaminant in comparison to the surrounding soil. Source areas include the following:

- Areas with stained soil, known contaminations, obvious releases/filling
- Areas where contaminants were suspected to be stored, handled, or disposed
- Areas where sufficient sampling evidence indicates elevated concentrations relative to the surrounding soil over a significant volume of contaminated media

Exposure area DU's are a fundamental part of many environmental investigations and are an essential tool in risk assessments and risk-based decision making. An "exposure area" is defined as an area where human or ecological receptors could come into contact with contaminants in soil on a regular basis. ITRC defines the following example exposure area DU's: residential yards, schoolyards, playgrounds, gardens, areas of commercial/industrial properties, or areas designated as exposure areas through other means (e.g., state laws).

This definition highlights the difference between types of DUs. Source area DUs are differentiated from exposure area DUs in that the boundaries of source area DUs and the scale of sampling are based on the known or hypothesized extent of the contamination, while the boundaries of exposure area DUs are determined through the exposure assumptions of the risk scenario. It is important to differentiate between these two types of DUs so that concerns regarding the dilution of source area will be eliminated and to reiterate that the action levels derived from risk assessment scenarios are based on exposure assumptions that include a specified areal extent of contamination within which a mean concentration is of interest.

The February 2012 ITRC Guidance provides additional background on the specifics of defining DU's for a several of scenarios including: residential exposure, commercial/industrial exposure, ecological exposure, source areas, subsurface, stockpile, and excavation. The DU's defined at a site where ISM is employed will be tailored to site and the CSM defined.

4. FIELD PROCEDURE

4.1 NECESSARY EQUIPMENT

The following is a list of required equipment:

- GPS
- Field measuring tape
- Wooden stakes, marking paint, pin flags, string
- Tile sampler with ½-inch or 1-inch corer and ejector
- Stainless steel bowl, new polyethylene food storage bags, 5-gallon plastic containers, or other appropriate large container for placing the increments; the size of the container should be adequate to hold the total aliquots and allow homogenization.
- If IMS sampling is used for volatile organic compound analysis, EnCore® sampler and laboratory container partially filled with sufficient methanol for the planned number of aliquots (1:1).
- Subcontractor drilling/excavation equipment or sample collection device (e.g., hand auger, shovel, etc.) if sampling subsurface DUs
- Polyethylene sheeting
- Folding table (optional, for convenience)
- Sweeping brush
- Laboratory provided sample containers, cooler and ice
- Decontamination fluids
- Pipe brush or brush specific to sampling tool
- Deionized or distilled water
- One 5-gallon buckets with water and detergent, one 5-gallon bucket with water, and scrub brush
- Paper towels
- Appropriate personal protection equipment (PPE) as required by the Site-specific Health & Safety Plan
- Site plan
- Field logbook
- Chain of custody
- Ink pens
- Digital camera

4.2 TRAINING AND HEALTHY AND SAFETY

Credere employees must have received 40-hour Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) training pursuant to 29 CFR 1910.120 and be current on their 8-hour refresher. Employees must have 24 hours of training specifically in soil sampling procedures, described herein, and proven competence of the SOP in compliance with OSHA 29 CFR 1910.120(e)(4). This SOP assumes the samplers will have training and background understanding of typical environmental soil sampling procedures outline in Credere SOP CA-5: Environmental Soil Sampling.

Employees will follow the Credere Corporate Health and Safety (CHASP) and the site-specific HASP when collecting soil samples.

4.3 DECISION UNIT FIELD LAYOUT

Depending on the areal and vertical contaminant distribution profile, IMS sampling and processing is used to minimize these sources of error, resulting in an average concentration that is a much more precise and accurate estimate for the SU/DU when compared to conventional means. The sample grid dimensions, number of aliquots, and number of replicates will be predetermined during the systemic planning stages. The relative location of any replicate increments within each SU/DU cell will be established in a random manner to remove potential bias. The cell may be divided in turn into sub-grids and a sub-cell may be selected randomly to select the relative increment location for a replicate increment.

The aliquot locations can be located in the field through field measuring a pre-determined sample grid, which is best suited to large open level spaces, or through GPS located points. In either case, the SU/DU will be marked in the field using pin flags, spray paint, or rope/string and fixed with a GPS.

GPS Located Points

GPS located points will be located with a GPS with sub-foot accuracy. The points will be loaded into the GPS after overlaying the predetermined grid in GIS and determining the center point of each sample grid. Replicate aliquots will be collected from locations measured from a certain distance in a certain direction from the point (e.g., replicate DU1-1 aliquots will be collected from 6 inches north of the center point and replicate DU1-2 aliquots will be collected from 2 feet east of the center point).

Field Measured Grid

The field measured grid will be laid out on a pre-determined grid size or such that equally sized grid units fill the SU/DU. The grid will be marked with pin flags, spray paint and or string so that each grid unit is distinguishable. The grid lines ends and corners will be located with a GPS with sub-foot accuracy and labeled using a grid matrix naming schedule (e.g., letters for the x-axis and numbers for the y-axis).

Prior to implementing any IMS sampling work, the field team will put on the personal protective equipment outlined in the project HASP. The increments will be collected from the depth specified in the work plan or other planning documents using the project specified coring tool or other sampling method. Unless defined in the sampling plan, the vegetative mat will be included in the sampled interval. The horizontal limits of sampling will be dependent on past disposal practices and the chosen size of the SU/DU. The stainless-steel sampler will be pushed into the soil until the sampler is full of soil and is not to be penetrated further.

4.4 SOIL SAMPLE COLLECTION PROCEDURES

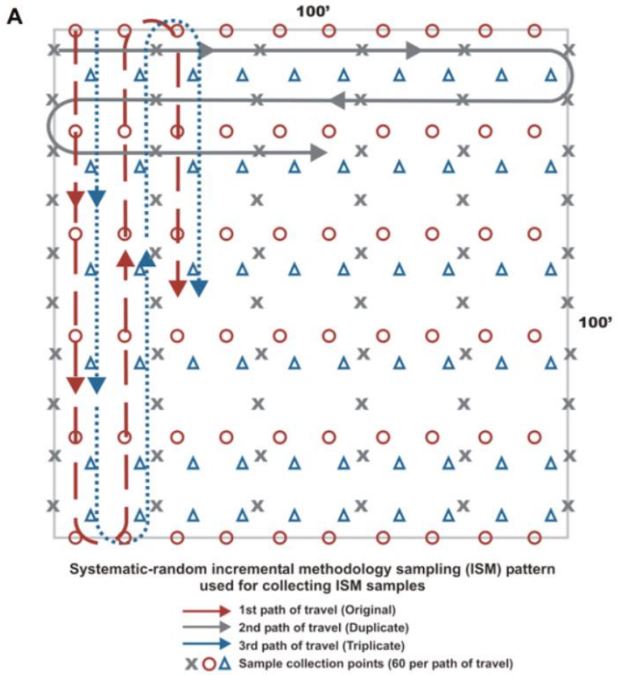
Equal increments of soil will be collected within each cell of the above located SU/DU. It is important that soil increments be approximately of the same weight.

Depending on the sample design, samples can be collected from surface to a desired depth (up to 2 feet bgs) or from a subsurface plane bound by a pre-determined criteria (e.g., depth, geological feature). The following sections describe the step-by-step procedure for sample collection for both these scenarios.

Surface Sampling Procedure

1. Upon arrival at the Site, begin a field logbook entry recording the Credere staff, scope of work for the day, and weather conditions in accordance with Credere SOP CA-1.
2. Calibrate field instruments according to Credere SOP CA-11 and product specifications. Keep record of calibration in the field logbook including instrument make and model, serial number, calibration gas and concentration, and span gas check.
3. Set up the sampling station with the optional folding table, truck tailgate, or on the ground surface by covering with clean polyethylene sheeting.
4. Prepare decontamination station. Note whether decontamination fluids must be containerized or can be discharged to the ground surface per the work plan.
5. Decontaminate tile sampler and homogenization container (or obtain a new bag) according to Credere SOP CA-2: Equipment Decontamination.
6. Begin collecting the first replicate by advancing the tile sampler to the desired depth. Eject the soil aliquot directly into a large re-sealable bag, 5-gallon bucket, or alternative large container.
    a. If sampling for VOCs, an individual VOA vial will be collected at each aliquot location to be composited by the laboratory. VOC aliquots will be collected directly from in situ soil and not from combined soil container.
7. Repeat step 6 for each grid location until the desired number of aliquots/grid units have been collected. It is not necessary to decontaminate the sampling tool between the aliquots within a SU/DU.
8. Homogenize the aliquots thoroughly, describe the combined soil type according to Credere SOP CA-4: Soil Description, and transfer the homogenized soil to a laboratory provided or approved container (typically a 1-liter wide mouth amber jar).
9. Label the sample container with the ID and DU as the sample ID: CA-DU1-1 (0-1) where CA-DU1 is the location, -1 is the replicate and 0-1 is the interval. If more than one DU has been defined for a Site, the SU designation should be added to the sample ID [e.g., CA-DU1/SU1-1 (0-1)].
10. Place samples immediately on ice. Record the sample time, ID, analyses, preservative, and sample volume in the field log book.

11. For each additional replicate repeat steps 5 through 10. Any replicate samples from the same SU/DU will be collected following a different path, as shown in the figure below (which was obtained from ITRC).



Subsurface Sampling Procedure

1. Upon arrival at the Site, begin a field logbook entry recording the Credere staff, scope of work for the day, and weather conditions in accordance with Credere SOP CA-1. Also record the drilling contractor, foreman, drill rig make and model, and tooling details.
2. Calibrate field instruments according to Credere SOP CA-11 and product specifications. Keep record of calibration in the field logbook including instrument make and model, serial number, calibration gas and concentration, and span gas check.
3. Set up the sampling station with the optional folding table, truck tailgate, or on the ground surface by covering with clean polyethylene sheeting.
4. Prepare decontamination station. Note whether decontamination fluids must be containerized or can be discharged to the ground surface per the work plan.
5. Decontaminate spoons/sampling tools and homogenization container (or obtain a new bag) according to Credere SOP CA-2: Equipment Decontamination.
6. Begin advancing soil borings to the desired subsurface increment according to Credere SOP CA-5: Environmental Soil Sampling.
7. Collect the desired increment from the sampled core and place in the large re-sealable bag, 5-gallon bucket, or alternative large container. If multiple depth increments are to be collected, prepare a dedicated container and label with the depth to ensure they are not mixed up during subsequent borings.

   a. If sampling for VOCs, an individual VOA vial will be collected at each aliquot location to be composited by the laboratory. VOC aliquots will be collected directly from in situ soil and not from combined soil container.

8. Repeat step 7 for each additional boring/aliquot until the desired number of aliquots/grid units have been collected. It is not necessary to decontaminate the sampling tool between the increments within a DU or SU.
9. Homogenize the aliquots thoroughly and transfer the homogenized soil to a laboratory provided or approved container (typically a 1-liter wide mouth amber jar). Soil samples should not include large any rocks or pebbles unless they are part of the overall soil matrix.
10. Label the sample container with the ID and DU as the sample ID: CA-DU1-1 (2-3) where CA-DU1 is the location, -1 is the replicate and 2-3 is the interval. If more than one DU has been defined for a Site, the SU designation should be added to the sample ID [e.g., CA-DU1/SU1-1 (2-3)].
11. Place samples immediately on ice. Record the sample time, ID, analyses, preservative, and sample volume in the field log book.
12. For each additional replicate repeat steps 5 through 11. Any replicate samples from the same SU/DU will be collected following a different path, as shown in the figure above (which was obtained from ITRC).
13. Holes left by sampling will be filled using surrounding soil or sand may be used to bring the subsurface sampling areas back to original grade.

4.5 POST-SAMPLING PROCEDURE

The following procedure should be completed after collection of soil samples:

1. Complete the chain-of-custody using the notes recorded in the field logbook in accordance with Credere SOP CA-16.
2. Decontaminate non-dedicated equipment for use at the next sample location or for transport in accordance with Credere SOP CA-2. If using an incremental sampling tool, shovel, or hand auger to collect soil samples, using 5-gallon bucket of detergent and rinse water to remove bulk soil and perform subsequent decontamination with appropriately selected decontamination fluids, rinse, and dry method per Credere SOP CA-2.
3. Store the sample containers on ice being sure to avoid pooled water in the cooler. Regularly replace ice and drain meltwater.

5. QUALITY ASSURANCE/QUALITY CONTROL

Quality assurance (QA) and quality control (QC) will be obtained through collection of additional samples. The types of samples to be collected are dependent on the data quality objectives and should be defined in the project's proposal/scope of work, Site-Specific Quality Assurance Project Plan (SSQAPP), regulatory/client guidance, or similar. Types of QA/QC samples that may be collected include the following:

- Field duplicates (as needed or required)
- Matrix spike/matrix spike duplicates (MS/MSD; as needed or required)
- Trip blanks (should be included with volatile analyses)
- Equipment blanks (e.g., after decontaminating an auger or sampler)
- Temperature blanks (laboratory specific; should be included in sample coolers for certain labs)

Field quality control will also be evaluated through comparison of replicate results.

Additionally, field documentation will be reviewed to assure it is compliant with Credere SOP CA-1: Field Activity Documentation and methods used for sampling are compliant with the protocol herein.

6. FIELD DOCUMENTATION

The following information should be recorded in the field logbook in compliance with Credere SOP CA-1:

- Date of field activity
- Credere personnel
- Scope of work
- Weather (particularly precipitation)
- Health and safety precautions
- Contractor/foreman/equipment make and model (if applicable)
- Changes in scope or deviations from the work plan
- Correspondence with Project Managers and/or Clients
- Sketches of sample locations relative to Site features and measurements (i.e., "swing ties") from permanent landmarks where location accuracy is required. GPS will be used to define the limits of each SU/DU in the field and sketch will detail the relative location of aliquots in the field.
- Sample details including IDs, number of aliquots per replicate, time of collection, requested analyses, volume of sample collected, and preservatives
- Decontamination procedures
- General timeline of field activities

7. REFERENCES

Credere Associates, LLC, *Standard Operating Procedure CA-1: Field Activity Documentation*, Revision 0, dated August 4, 2015.

Credere Associates, LLC, *Standard Operating Procedure CA-2: Equipment Decontamination*, Revision 0, dated March 17, 2016.

Credere Associates, LLC, *Standard Operating Procedure CA-4: Soil Description*, Draft, dated TBD.

Credere Associates, LLC, *Standard Operating Procedure CA-7: Headspace Field Screening*, Draft, dated TBD.

Credere Associates, LLC, *Standard Operating Procedure CA-16: Chain of Custody Preparation*, Draft, dated TBD.

Interstate Technology Regulatory Council (ITRC), *Technical & Regulatory Guidance, Incremental Sampling Methodology*, Prepared by ITRC Incremental Sampling Methodology Team, February 2012.

U.S. DoD, *Environmental Field Sampling Handbook, Revision 1.0*, dated April 2013.

U.S. Environmental Protection Agency Environmental Response Team, *Soil Sampling*, SOP#: 2012, dated February 18, 2000.

U.S. Environmental Protection Agency, *Standard Operating Procedure for Soil, Sediment and Solid Waste Sampling*, SOP EIASOP_SOILSAMPLING2, Revision 2, dated February 13, 2004.

U.S. Environmental Protection Agency, Region 4, Science and Ecosystem Support Division, Operating Procedure, *Soil Sampling*, SESDPROC-300-R3, Revision 3, dated August 21, 2014.

APPENDIX F

HUMAN HEALTH AND SCREENING LEVEL ECOLOGICAL RISK ASSESSMENT METHODOLOGY

APPENDIX F
Human Health and Ecological Risk Assessment Methodology
Gould Island (D01RI33800)
Narragansett Bay, Jamestown, Rhode Island

INTRODUCTION

This appendix presents an outline of the methodology to be followed in preparation of a Human Health Risk Assessment (HHRA) and a Screening Level Ecological Risk Assessment (SLERA) for the Gould Island site (Site) in Jamestown, Rhode Island.

HUMAN HEALTH RISK ASSESSMENT

Objectives and Applicable Guidance

The purpose of an HHRA is to quantify potential human health risks and hazards associated with exposure to constituents of concern (COCs) in Site media under current and potential future site conditions and uses. These quantified risks are compared to federal and state acceptable risk/hazard levels to identify the need for limitations on Site uses (if any), the need for further Site characterization, or to document that Site conditions are acceptable for current and potential future site uses. The following generally-recognized risk assessment and regulatory guidance will be applied, as applicable, to the human HHRA:

- US Environmental Protection Agency (US EPA) (1988). Superfund Exposure Assessment Manual. OSWER Directive 9285.5-1.
- US EPA (1989). Risk Assessment Guidance for Superfund (RAGS): Volume 1, Part A - Human Health Evaluation Manual. EPA/540/1-89/002).
- US EPA (1991). Risk Assessment Guidance for Superfund. Volume 1: Human Health Evaluation Manual, Supplemental Guidance: Standard Default Exposure Factors (OSWER Directive 9285.6-03).
- US EPA (2001). Risk Assessment Guidance for Superfund: Volume I – Human Health Evaluation Manual (Part D, Standardized Planning, Reporting, and Review of Superfund Risk Assessments).
- US EPA (2002). Calculating Upper Confidence Limits for Exposure Point Concentrations at Hazardous Waste Sites. OSWER 9285.6-10.
- US EPA (2003). Human Health Toxicity Values in Superfund Risk Assessments. OSWER 9285.7-53.
- US EPA (2004). Risk Assessment Guidance for Superfund, Volume I: Human Health Evaluation Manual (Part E, Supplemental Guidance for Dermal Risk Assessment).
- US EPA (2011). Exposure Factors Handbook.
- US EPA (variable). Integrated Risk Information System.
- Rhode Island Department of Environmental Management (RIDEM) (2011). Rules and Regulations for the Assessment and Remediation of Hazardous Material Releases.
- American Society for Testing and Materials (ASTM) (2015). Standard Guide for Risk-Based Corrective Action.

Additional federal, state, or scientific guidance may be applied.

APPENDIX F
Human Health and Ecological Risk Assessment Methodology
Gould Island (D01RI33800)
Narragansett Bay, Jamestown, Rhode Island

Methodology

An HHRA is typically described as having four basic steps

- Step 1 – Hazard Identification
- Step 2 - Dose-Response Assessment
- Step 3 – Exposure Assessment
- Step 4 – Risk Characterization

Each of these steps is discussed in greater detail in the following subsections.

Hazard Identification

The objectives of the hazard identification step are to identify the chemicals present at the Site as a result of Site releases and to select those constituents to be carried through the HHRA. Activities of the hazard identification step include the following:

- Review and compilation of available information on the physical and hydrogeological setting of the Site.
- Review and compilation of historical and/or on-going occurrences of contaminant releases.
- Review and compilation of available chemical analytical data in Site media and comparison of detected results and analytical reporting limits to applicable risk-based screening levels to verify utility of the data. Data to be applied to the HHRA will be evaluated and validated in accordance with the project's Quality Assurance Project Plan (QAPP) under other activities prior to use.
- Selection of constituents of concern (COCs). This activity involves comparing available data with various criteria to identify the constituents to be carried through the HHRA. These criteria include: frequency of detection, comparison with site-specific soil background concentrations, presence in trip or method blank samples, role as an essential nutrient, and magnitude of detected concentrations relative to risk-based screening criteria. Selected COCs will be those constituents present at concentrations above background levels, detected in more than one sample, not present as a result of trip or method blank contamination, and exceeding one or more conservative risk-based screening criteria. These risk-based screening criteria will include US EPA Regional Screening Levels for soil and groundwater at conservative risk/hazard levels [1 in 1,000,000 ($1x10^{-6}$) risk level or non-carcinogenic hazard index of 0.1], Rhode Island Department of Environmental Management (RIDEM) Method 1 residential soil standards, drinking water standards for groundwater (RIDEM GA groundwater standards, US EPA maximum contaminant levels), and other appropriate media-specific screening levels. All detected constituents will be evaluated in the COC selection process and the rationale for exclusion of any detected constituent as a COC will be identified. A summary of the fate and transport characteristics of each COC will also be provided.

Dose-Response Assessment

The objectives of a dose response assessment are to identify the types of potential adverse human health effects potentially posed by exposure to COCs (e.g., cancer, systemic toxicity, acute effects) and to select the appropriate toxicity values for use in the HHRA. All constituents adopted as COCs will have toxicity values compiled from generally recognized sources in accordance with the source hierarchy described in US EPA (2003); primarily US EPA's Integrated Risk Information System (IRIS) or US EPA's Provisional Peer-Reviewed Toxicity Values (PPRTV). For volatile and extractable petroleum hydrocarbon (VPH/EPH) fractions analyzed by Massachusetts Department of Environmental Protection

(MassDEP) methodologies, applied toxicity values will be those adopted by MassDEP for these fractions. Supporting factors, such as oral or dermal relative absorption factors, will be identified and compiled. Brief toxicity profiles will also be prepared.

Exposure Assessment

The objectives of the exposure assessment are to identify current and potential future human uses of the Site; to identify potentially exposed human receptor groups for these uses; to identify pathways through which human receptors may be exposed; to identify physical locations at which exposures may occur (exposure points); and to derive exposure point concentrations (EPCs) for each COC at each exposure location. The exposure assessment will also identify appropriate exposure factors describing the magnitude, frequency, and duration of the exposure of each human receptor group. This information will be used to refine the conceptual Site model (CSM) provided in Appendix B of the QAPP.

The current human uses of the Site are recreational and/or trespassing (in areas not open for recreational use). However, to assess potential future uses, the HHRA will assume unrestricted future site uses including residential use (child and adult), industrial/commercial use, and development of the site with potential exposure of construction or utility workers. Assessment of these receptors will identify whether any future use should be prohibited or if Site conditions are acceptable for unrestricted future uses. Potential exposure pathways by which selected human receptors may be exposed are shown below.

Exposure Pathway	Human Receptor Groups to be Assessed				
	Recreator	**Trespasser**	**Future Resident**	**Future Industrial/ Commercial Worker**	**Future Construction/ Utility Worker**
Soil Ingestion	✓	✓	✓	✓	✓
Soil Dermal Contact	✓	✓	✓	✓	✓
Outdoor Inhalation of Entrained Soil Particles	✓	✓	✓	✓	✓
Outdoor Inhalation of Volatile COCs in Soil or Groundwater [1]	✓	✓	✓	✓	✓
Indoor Inhalation of Volatile COCs in Soil or Groundwater [1]			✓	✓	
Ingestion of Groundwater			✓	✓	
Dermal Contact with Groundwater			✓	✓	✓
Sediment Ingestion	✓	✓	✓		
Sediment Dermal Contact	✓	✓	✓		
Surface Water Ingestion	✓	✓	✓		
Surface Water Dermal Contact	✓	✓	✓		

[1] Contingent upon the presence of volatile compounds in Site media.

The completeness of these exposure pathways depends on the presence of COCs in associated Site media, so assessed exposure pathways are subject to change. If additional media are available (e.g., fish tissue), the appropriate exposure pathways will be assessed.

The nature and extent of chemical contamination of the site will be examined to identify probable locations of exposure (exposure points) to Site COCs. Areas of the Site not showing any degree of contamination will be identified and excluded from assessed exposure points. To some degree, the extent

of areas will be defined by the placement of sample locations, so assessed exposure points may be used to represent uncharacterized areas. This will depend, in part, on the nature of the release and expectation of chemical presence in uncharacterized areas.

Within these exposure points, exposure point concentrations (EPCs) will be calculated from the available data, in general accordance with US EPA (2002). Data may be segregated by depth intervals to derive some EPCs, such as surface soil (e.g., 0-2 feet); however, RIDEM's soil compliance point as the entire vadose zone may require combining soil samples from all depths above the groundwater surface when deriving EPCs. The approach ultimately taken will be based on the distribution of contaminant concentrations in soil and clearly identified in the HHRA. For groundwater, if evidence of more than one discrete aquifer exists, EPCs for each aquifer will be derived. Otherwise, groundwater EPCs will represent the most shallow aquifer. EPCs for sediment and surface water may vary depending on the amount of data available, as subsequently discussed.

It is anticipated that sufficient soil data will be available such that soil EPCs will generally be the 95th percentile upper confidence limit of the sample mean COC concentration (95% UCL). Reported estimated concentrations below the analytical reporting limit (i.e., J-qualified data) will be applied as detections if validated as such. For samples accompanied by a field duplicate, the higher detected concentration of the sample/duplicate pair will be used to represent the sample; if both results of a sample/duplicate pair are reported as non-detections, the lower of the two reporting limits will be used to represent the sample. In soil samples reported as non-detections at an elevated reporting limit above all detected COC concentrations in soil as a result of sample dilution, matrix interferences, or the presence of other constituents in the sample, the elevated reporting limit may be excluded from the calculation of an EPC to avoid biasing the EPC. This will be performed on a case-by-case basis and clearly identified in the HHRA. 95% UCL concentrations in soil will be calculated by US EPA's ProUCL. ProUCL handles non-detections differently depending on the distribution of the data and statistical test employed. For summary (descriptive) statistics, mean COC concentrations may be calculated assuming that a non-detected COC is present at that location at a concentration equal to one-half the sample's analytical reporting limit. In the case of constituents that have a unique risk assessment methodology, such as lead [assessed by US EPA's Integrated Exposure, Uptake, and Biokinetic (IEUBK) model], the soil EPC will be consistent with the recommended risk assessment methodology.

Depending on the amount of current (i.e., within the past 5 years) groundwater data available, groundwater EPCs may be the maximum detected concentration of each COC among site monitoring wells over this time period or the maximum arithmetic mean concentration in and among site monitoring wells over this current time period. EPCs for groundwater will not be averaged between monitoring wells. Since the number of groundwater samples from individual monitoring wells is not expected to be sufficient to use ProUCL (e.g., >10 samples), groundwater EPCs (if well arithmetic means are calculated) will be spreadsheet-calculated by applying one-half the sample's analytical reporting limit to represent non-detected results.

EPCs for sediment and surface water will depend on the amount of data available. EPCs may be the 95% UCL COC concentration or the maximum detected concentration of each COC at each exposure point (appropriate for surface water).

EPCs for air exposure pathways will be modeled from soil and groundwater EPCs by applying generally recognized exposure models, such as those described by US EPA (1998), ASTM (2015), or other relevant guidance. All calculations of modeled EPCs will be presented in the HHRA.

Exposure factors that describe the magnitude, frequency, and duration of exposure will be obtained from US EPA sources (e.g., US EPA 2001) or from best professional judgment, depending on the availability of recommended values.

Risk Characterization

The objective of the risk characterization is to derive quantitative cancer risk estimates and non-carcinogenic health hazards by combining the results of the hazard identification, dose-response assessment and exposure assessment. The quantitative results are then compared with maximum acceptable cumulative risk and hazard levels to determine the acceptability of Site conditions.

Generally-accepted risk characterization equations, such as those described in US EPA (1989), and appropriate EPCs and exposure factors will be combined to quantify the degree of exposure (e.g., as an average daily exposure). The quantified exposure will then be combined with the appropriate toxicity value(s) to generate COC- and pathway-specific non-carcinogenic hazard quotients (HQs) and/or excess lifetime cancer risks, as appropriate for the COC. Within each exposure pathway, all non-carcinogenic HQs will be summed to derive a pathway-specific Hazard Index (HI). Similarly, within a pathway, all cancer risks will be summed to derive a pathway-specific cancer risk. For all co-occurring exposure pathways for a given receptor group, pathway-specific HIs and cancer risks will each be summed to derive a total (or cumulative) HI or excess lifetime cancer risk.

Total HIs and excess lifetime cancer risks for each receptor group will be compared with maximum acceptable hazard and risk levels adopted by RIDEM (2011) and consistent with the acceptable risk range supported by US EPA: a total non-carcinogenic HI of 1 and an excess lifetime cancer risk of 1 in 100,000 (denoted as $1x10^{-5}$). If, for a given human receptor group, the total (cumulative) HI is at or below 1 and the total (cumulative) excess lifetime cancer risk is at or below $1x10^{-5}$, the receptor exposure will be concluded to be acceptable; i.e., no unacceptable health hazards or risks are posed by the exposure. If the total hazards and/or risks are above maximum acceptable levels, the COC(s) and exposure pathway(s) contributing the greatest amount to the total HI/risk will be identified.

For COCs that apply a unique methodology to quantify health risks, such as lead that is assessed by the IEUBK model, these constituent-specific approaches will be applied. As appropriate, the results from these unique methodologies will be either quantitatively or qualitatively combined with the numerical results of the primary risk assessment to consider combined hazards/risks.
The risk characterization will also include a qualitative uncertainty analysis that will identify factors in the HHRA that contribute notable uncertainty to the quantitative hazard/risk estimates. General categories that will be considered in the uncertainty analysis include the Site data itself (e.g., amount, age, location, data quality issues), the selection of potentially exposed receptor groups and associated exposure factors, applied EPCs, and applied toxicity values. If the uncertainty evaluation identifies data gaps or other limitations that could be resolved through additional Site characterization, such recommendations for additional studies will be brought forward.

SCREENING LEVEL ECOLOGICAL RISK ASSESSMENT

Objectives and Applicable Guidance

The purpose of a screening level ecological risk assessment (SLERA) is to identify the potential for environmental risks associated with exposure to COCs in Site media under current and potential future site conditions and uses. The evaluation of these risks will take the form of a comparison to ecological-based screening criteria. The conclusion of a SLERA is either that there is a potential for unacceptable levels of ecological risks to exist and that a more in-depth baseline ecological risk assessment is

warranted, or that there is no evidence that the Site poses a risk to ecological receptors and no further assessment is needed. The following guidance will be applied, as applicable, to the SLERA:

- US EPA (1997) Ecological Risk Assessment Guidance for Superfund: Process for Designing and Conducting Ecological Risk Assessments. OSWER 9285.7-25.
- US EPA (1998). Guidelines for Ecological Risk Assessment. EPA/630/R-95/002F.
- US EPA (2001). The Role of Screening Level Risk Assessments and Refining Contaminants of Concern in Ecological Risk Assessments. OSWER Publication 9345.0-14.
- US EPA (2016). Generic Ecological Assessment Endpoints (GEAEs) for Ecological Risk Assessment: Second Additional with Generic Ecosystem Services Endpoints Added. EPA/100/F15/005.
- US EPA (chemical-specific; varied dates). Interim Ecological Soil Screening Documents.

Methodology

A SLERA is typically described as having three basic steps:

- Step 1 – Screening Level Problem Formulation and Ecological Effects Evaluation
- Step 2 – Screening Level Exposure Estimate and Risk Calculation
- Step 3 – Scientific/management decision point

Each of these steps is discussed in greater detail in following subsections.

Screening Level Problem Formulation and Ecological Effects Evaluation

This phase of a SLERA includes the following components:

- Identification of the environmental setting of the Site and identification of ecological COCs. Much of this component will have been completed as part of the HHRA. Additional factors that are uniquely relevant to the SLERA will be discussed.
- Identification of contaminant fate and transport pathways for the COCs. Much of this component will have been completed as part of the HHRA. Additional factors that are uniquely relevant to the SLERA will be discussed.
- Description of contaminant mechanisms of ecotoxicity and categories of receptors likely affected. Mechanisms of toxicity for the SLERA will generally include organism-level effects on survival, growth, and/or reproduction. If the SLERA concludes that continuation to a baseline environmental risk assessment is warranted, additional mechanisms of toxicity (e.g., population-, community-, or ecosystem-level endpoints) may be addressed. Based on the setting of the Site within Narragansett Bay and current use of the Site as a bird sanctuary, the following ecological receptors are identified:
    - Terrestrial bird species
    - Piscivorous (fish-eating) bird species
    - Terrestrial mammalian species
    - Piscivorous mammalian species
    - Pelagic (water column) aquatic organisms
    - Benthic (sediment-dwelling) aquatic organisms

- Identification of complete exposure pathways and selection of generic assessment endpoints. The completeness of exposure pathways will be identified by the presence of Site COCs in media and locations that ecological receptors are reasonably expected to contact. For upland (terrestrial) locations, exposure may occur through direct contact with contaminated media, contaminant ingestion through food consumption (e.g., food chain effects), or (less likely) through inhalation. For aquatic locations (i.e., in sediment adjacent to contaminated upland areas), exposure may similarly occur through direct contact with contaminated media or contaminant ingestion through food consumption (e.g., food chain effects).
- Selection of screening ecotoxicity values. Screening level ecotoxicity values will be identified and compiled for use in the SLERA. Most applied Screening level ecotoxicity values take the form of media concentrations and may include the following:
    - For soil:
        - US EPA EcoSSLs.
        - Site-specific background soil concentrations.
    - For surface water (note that Narragansett Bay is an estuary, so both freshwater and salt water screening values may be used):
        - US EPA National recommended ambient water quality criteria for aquatic life (*https://www.epa.gov/wqc/national-recommended-water-quality-criteria-aquatic-life-criteria-table*).
        - RIDEM surface water criteria [RIDEM Water Quality Regulations (2009)].
        - US EPA Regional III Biological Technical Assistance Group (BTAG) Freshwater Screening Benchmarks.
        - US EPA Region III BTAG Marine Screening Benchmarks.
    - For sediment:
        - Consensus-based threshold effect concentrations (TECs) and probable effect concentrations (PECs) (MacDonald et al. 2000).
        - US EPA Region III BTAG Freshwater Sediment Screening Benchmarks.
        - US EPA Region III BTAG Marine Sediment Screening Benchmarks.
        - Calculated sediment benchmarks using the equilibrium partitioning approach and site-specific information on total organic carbon content of sediment.

Because the basis for these screening benchmarks may and do differ, multiple benchmarks will initially be presented and utility (or lack thereof) of the benchmark discussed. Additional federal, state, or scientific guidance may be applied. Note that there are no generally-recognized ecological benchmarks for air or groundwater, so these media will not specifically be considered for ecological effects.

Screening Level Exposure Estimate and Risk Calculation

This phase of a SLERA includes the following components:

- Determination of screening level exposure estimates [i.e., media exposure point concentrations (EPCs)]. The EPC to which an ecological receptor may be exposed may be the same as the EPC applied in the HHRA, but may differ because of unique features of the ecological exposure. All EPCs applied to the SLERA will be identified and discussed. Treatment of duplicate samples,

non-detections, and elevated reporting limits will generally be handled in the same manner as in the HHRA.

- Calculation of the risk estimate. Ecological risks for a specific receptor group, medium, and exposure pathway will be estimated numerically using the Hazard Quotient (HQ) ratio approach for each COC and relevant exposure pathway. The HQ is calculated as:

  HQ = COC EPC/applicable screening benchmark

  where the COC EPC is the relevant medium EPC for the given exposure and the applicable screening benchmark is the most relevant benchmark for the medium, chemical, and receptor being assessed. The COC- and pathway-specific results are interpreted in the following manner: an HQ > 1.0 indicates harmful effects cannot be ruled out; HQ = 1.0 indicates that the COC alone is not likely to cause an unacceptable ecological risk; and HQ <1.0 indicates that harmful effects are not likely. For each receptor group and co-occurring exposure pathways, HQs are summed to derive an overall Hazard Index, interpreted in a manner similar to that for the HHRA.

- Evaluation of uncertainties. As with the HHRA, inputs into the quantitative ecological risk results will be examined to identify applied factors that contribute uncertainty to the risk estimate. General categories that will be considered in the uncertainty analysis include the Site data itself (e.g., amount, age, location, data quality issues), the selection of potentially exposed receptor groups and associated exposure factors, applied EPCs, and applied toxicity values. If the uncertainty evaluation identifies data gaps or other limitations that could be resolved through additional Site characterization, such recommendations for additional studies will be brought forward.

DOCUMENTATION

The HHRA and SLERA will be documented in stand-alone reports containing all data, calculations, and assumptions applied to the assessments and appended to the Remedial Investigation (RI) report. A summary of the conclusions of the HHRA and SLERA will be incorporated into the RI report.