APPLIED INTELLECT

Draft Final
Uniform Federal Policy Quality Assurance Project Plan
Data Gaps Investigation
Rolling Knolls Landfill

Great Swamp National Wildlife Refuge
Morris County, New Jersey

Contract: GS 10F-026BA | Task Order: 140F0520F0195

Prepared for:
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ACRONYMS

AI	Applied Intellect, LLC
amsl	above mean sea level
APC	Areas of Particular Concern
ARAR	Applicable or Relevant and Appropriate Requirements
ASTM	American Society for Testing and Materials
ATV	all-terrain vehicle
AVS	acid-volatile sulfides
BERA	Baseline Ecological Risk Assessment
bgs	below ground surface
BHHRA	Baseline Human Health Risk Assessment
BLM	Bureau of Land Management
CCP	Comprehensive Conservation Plan
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CO	Contracting Officer
COC	Chain of Custody
COPC	chemicals of potential concern
COPEC	contaminants of potential ecological concern
COR	Contracting Officer's Representative
CSM	conceptual site model
DO	Dissolved Oxygen
DOC	Dissolved Organic Carbon
DoD	Department of Defense
DPT	direct-push technology
DQI	Data Quality Indicators
DQO	Data Quality Objective
DU	Decision Units
EPC	Exposure Point Concentration
ESC	Ecological Screening Criteria
ESV	Ecological Screening Values
ETA	Eurofins/TestAmerica
FID	Flame Ionization Detector
FS	Feasibility Study
FSP	Field Sampling Plan
ft	feet
FWS	United States Fish and Wildlife Service
GPS	Global Positioning System
HAZWOPER	Hazardous Waste Operations and Emergency Response
HASP	Health and Safety Plan
ID	identification
IDW	Investigation Derived Waste
in^3	cubic inches
LCS	laboratory control sample
LCSD	laboratory control sample duplicate

LOD	limits of detection
LOQ	limit of quantitation
MNA	monitored natural attenuation
MS/MSD	matrix spike/matrix spike duplicate
MW	Monitoring Well
N	Northeastern Refuge Study Area
NJDEP	New Jersey Department of Environmental Protection
NPL	National Priority List
OCP	organochlorine pesticides
ORP	oxidation reduction potential
oz	Ounce
PA/SI	Preliminary Assessment/Site Inspection
PAH	polycyclic aromatic hydrocarbons
PCB	polychlorinated biphenyls
PFAS	polyfluorinated alkyl substances
PFOA	perfluorooctanoic acid
PFOS	perfluoro octane sulfonate
PG	Professional Geologist
PID	photoionization detector
PM	Project Manager
PMP	Project Management Professional
PQO	project quality objectives
PRG	preliminary remediation goals
PRP	potentially responsible party
QA/QC	quality assurance/quality control
QSM	Quality Systems Manual
Refuge	Great Swamp National Wildlife Refuge
RI	Remedial Investigation
RI/FS	Remedial Investigation/Feasibility Study
RPD	Relative Percent Difference
SAT	Site Assessment Team
SEM	simultaneously extracted metals
SI	Site Inspection
SOP	Standard Operating Procedure
SOW	Statement of Work
S	Southern Refuge Study Area
SU	Sampling Unit
SVOC	semi-volatile organic compounds
TAL	Target Analyte List
TBD	To Be Determined
TCBH	Township of Chatham Board of Health
TO	Task Order
TOC	total organic carbon
UFP-QAPP	Uniform Federal Policy Quality Assurance Project Plan

USEPA	United States Environmental Protection Agency
VOC	volatile organic compounds

EXECUTIVE SUMMARY

This Uniform Federal Policy Quality Assurance Project Plan (UFP-QAPP) is Part 1 of the Work Plan for the Data Gap Site Characterization (DGSC)at the Great Swamp National Wildlife Refuge (Refuge) associated with landfill contamination and debris from the Rolling Knolls Landfill Superfund Site located in Morris County, New Jersey (the Site). The purpose of the UFP-QAPP is to be the comprehensive site-specific planning and guidance document to govern the field sampling, field analysis, and environmental laboratory analysis for the work contracted under Task Order (TO) : 140F0520F0195 for the United States Fish and Wildlife Service (FWS). This document shall be utilized by the field sampling team and the laboratory analytical team to ensure this effort meets the specified project quality objectives (PQOs) for the TO. This project is contracted by the FWS, Division of Contracting and General Services under General Services Administration Contract GS10F026BA, TO 140F0520F0195. The Health and Safety Plan (HASP) is Part 2 of the Work Plan and will be submitted in a separate document.

The objectives of DGSC are to evaluate the threat to the Refuge from chemicals that have been released into the environment from Rolling Knolls Landfill activities, define the extent of contamination on the Refuge in accordance with the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) and regulations of the New Jersey Department of Environmental Protection (NJDEP).

The UFP-QAPP is the main body of the document. Appendices include:

- Appendix A – Scoping Meeting Minutes;
- Appendix B – Standard Operating Procedures (SOPs) for all field and data management tasks;
- Appendix C – Field Forms;
- Appendix D – Performance Criteria;
- Appendix E – SOPs for all Analytical Laboratory Test Methods

Attachment 1 is the Conceptual Site Model Cross-Sections from the previous RI report (Geosyntec, 2018b), Figure 6-1b.

1. QAPP WORKSHEET #1 - TITLE AND APPROVAL PAGE

Revision Log:

Revision #	Revision Date	Revision Description
0	7/31/20	Preliminary Draft
1	9/28/2020	Draft
2	10/13/2020	Draft Final

Signatories:

George Molnar Contracting Officer's Representative US Fish and Wildlife Service	[Signature]	[Date Signed]
Jeffrey S. Hart, PG, PMP Project Manager Applied Intellect, LLC	[Signature]	October 13, 2020 [Date Signed]
Paul Hunter Quality Assurance Manager Applied Intellect, LLC	[Signature]	October 13, 2020 [Date Signed]

By signing above, the signatories verify that they understand and concur with the information, procedures, and recommendations presented herein.

2. QAPP WORKSHEET #2 - IDENTIFYING INFORMATION

Project Name: Rolling Knolls Data Gaps Investigation, 2020

Area of Concern: Great Swamp National Wildlife Refuge

Contractor Name: Applied Intellect, LLC
Contract Number: Contract: GS 10F-026BA, Task Order: 140F0520F0195

1. This UFP-QAPP was prepared in accordance with the requirements of:

- Optimized UFP-QAPP Worksheets (United States Environmental Protection Agency [USEPA] 2012);
- UFP-QAPP Manual (USEPA 2005); and
- EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5, Quality Assurance Manual (USEPA 2002).

2. Identify regulatory program(s):

- CERCLA.

3. Identify regulatory Agency(ies):

- FWS
- NJDEP; and
- USEPA.

4. This UFP-QAPP is a:

- Project-specific document to provide detailed information on the execution of the field program for the DGSC at the Refuge and Rolling Knolls Landfill Superfund Site (the Site), to identify data quality objectives (DQOs) and ensure data quality will meet those objectives.

5. List dates of scoping sessions that were or will be held:

- August 13, 2020;
- August 18, 2020;
- September 10, 2020;

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

- Arcadis U.S., Inc. 2007. Remedial Investigation/Feasibility Study Work Plan. Rolling Knolls Landfill Superfund Site, Chatham, New Jersey. May.
- Arcadis U.S., Inc. 2008a. Drum Area Investigation Work Plan. Rolling Knolls Landfill Superfund Site, Chatham, New Jersey. April.
- Arcadis U.S., Inc. 2009a. Remedial Investigation/Feasibility Study Work Plan Addendum. Rolling Knolls Landfill Superfund Site, Chatham, New Jersey. August.
- Arcadis U.S., Inc. 2014. Data Gaps Sampling and Analysis Plan, Rolling Knolls Landfill Superfund Site, Chatham, New Jersey. November.
- Arcadis U.S. Inc. 2015. Addendum 1 to the Data Gaps Sampling and Analysis Plan, Rolling Knolls Landfill Superfund Site, Chatham, New Jersey. August.
- Geosyntec Consultants. 2016b. Supplemental Groundwater and Baseline Monitored Natural Attenuation Investigation Work Plan, Rolling Knolls Landfill Superfund Site. September.

7. List organization(s):

- FWS;
- Robert J. Miele as Trustee for the Trust created by the Last Will and Testament of Angelo J. Miele; and
- Green Village Fire Department.

8. UFP-QAPP Table of Contents:

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PA/SI – Preliminary Assessment / Site Inspection
QA – Quality Assurance
QC – Quality Control
SOP – Standard Operating Procedure
UFP-QAPP – Uniform Federal Policy Quality

3. QAPP WORKSHEET# 3 - DISTRIBUTION LIST

This worksheet identifies key project personnel for the lead organization and regulating authorities that will receive a complete copy of the UFP-QAPP, including future updates, change pages, and/or addenda

Table 3-1. Distribution List

Name of UFP- QAPP Recipients	Title/Role	Organization	Telephone Number (Optional)	E-mail Address or Mailing Address	Document Control Number (Optional)
George Molnar	Contracting Officer's Representative	FWS/Refuge		george_molnar@fws.gov	N/A
Graham Taylor	Program Manager	FWS/Refuge Supervisor		graham_taylor@fws.gov	N/A
Michael Horne	Refuge Manager	FWS/Refuge		michael_horne@fws.gov	N/A
Supinderjit Kaur	Project Manager	USEPA		kaur.supinderjit@epa.gov	N/A
Jill McKenzie	Project Manager	NJDEP		jill.mckenzie@dep.nj.gov	N/A
Paul Hunter	Quality Manager	AI		paul.hunter@ap-in.com	N/A
Jeff Hart	Project Manager	AI		Jeff.hart@ap-in.com	N/A
Preston Sowell	Installation Lead	AI		psowell@geoticsolutions.com	N/A
David Back	Field Team Leader	AI		davidback2@aol.com	N/A
Christopher Lammer	Project Chemist	AI		chris.lammer@ap-	N/A
Aidan Scott	Laboratory Project Manager	ETA		Aidan.Scott@Eurofinset.com	N/A
Stella Cuenco	Data Validator Project Manager	Laboratory Data Consultants		scuenco@lab-data.com"	N/A
TBD	Drilling Subcontractor	TBD			N/A

Notes:
AI – Applied Intellect, LLC
ETA – Eurofins/TestAmerica
FWS – US Fish and Wildlife Service
NJDEP – New Jersey Department of Environmental Protection
TBD – To Be Determined
USEPA – US Environmental Protection Agency

4. QAPP WORKSHEET #4 - PROJECT PERSONNEL SIGN-OFF SHEET

This worksheet documents that all key project personnel performing work have read the applicable sections of this UFP-QAPP and will perform the tasks as described.

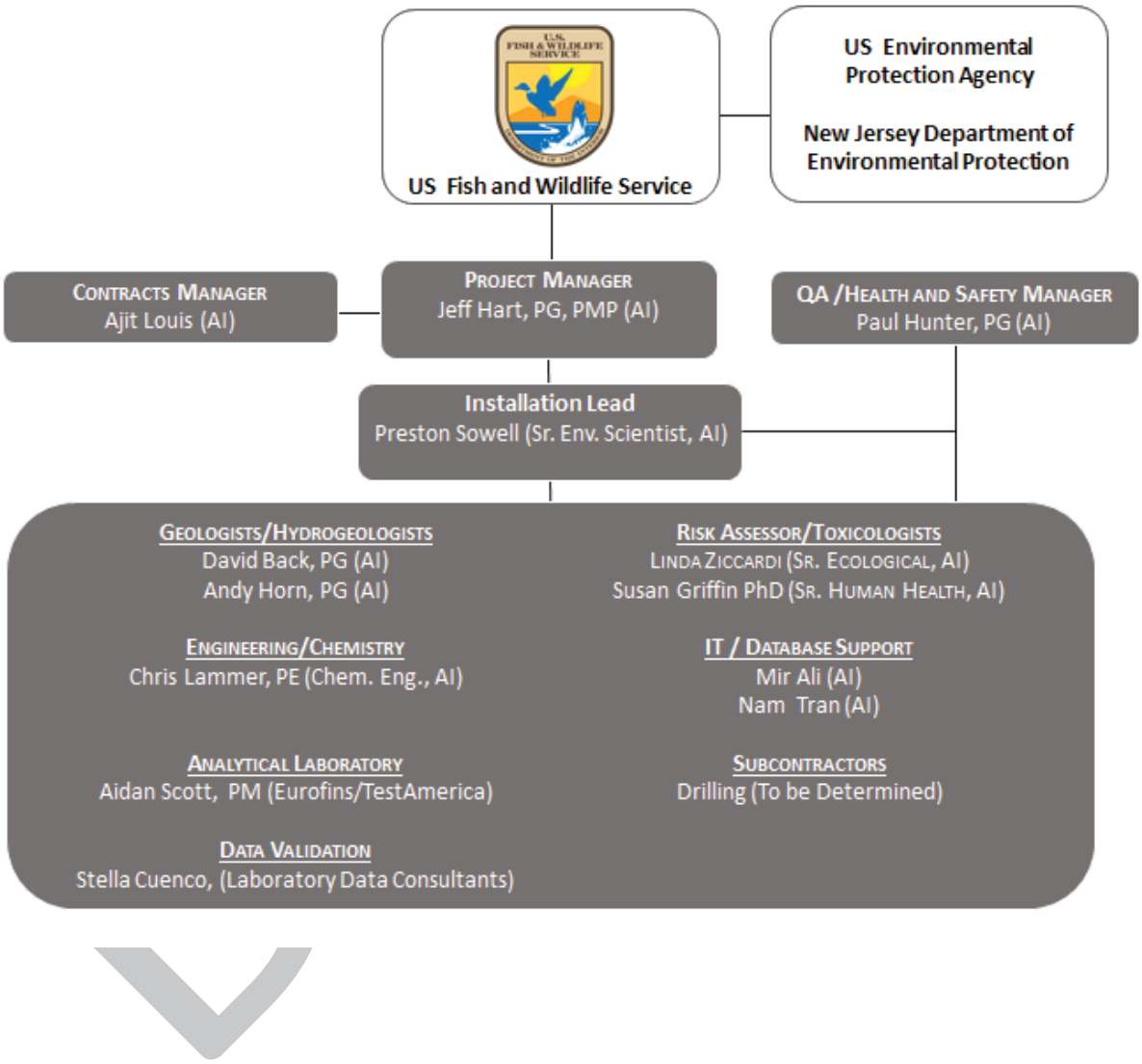
Table 4-1. Project Personnel Sign-Off Sheet

Name	Title	Organization	Telephone Number	Signature/Email Receipt	Date UFP-QAPP Reviewed
Paul Hunter	Quality Manager	AI	208.899.6784		
Jeff Hart	Project Manager	AI	720.884.7404		
Preston Sowell	Installation Lead	AI	303.775.6920		
David Back	Field Team Leader	AI	703.241.1718		
Chris Lammer	Project Chemist	AI	208.600.2295		
Aidan Scott	Laboratory Project Manager	ETA	646.745.0906		
Stella Cuenco	Data Validator	LDC	760.827.1100		
Supinderjit Kaur	Project Manager	EPA	TBD		
Jill McKenzie	Project Manager	NJDEP	TBD		

AI – Applied Intellect, LLC
ETA – Eurofins/TestAmerica
FWS – US Fish and Wildlife Service
NJDEP – New Jersey Department of Environmental Protection
TBD – To be Determined
USEPA – US Environmental Protection Agency

5. QAPP WORKSHEET #5 - PROJECT ORGANIZATION CHART

This worksheet details the organizational structure of the project and includes the lead organization, regulating authorities, contractors, subcontractors, and any other organization involved in the project. This structure shows reporting relationships between all entities involved in the project.



6. QAPP WORKSHEET #6 - COMMUNICATION PATHWAYS

This worksheet identifies the communication pathways between project personnel.

Table 6-1. Communication Pathways

Communication Drivers	Organization/Title	Name/Email	Phone Number	Procedure
FWS Points of Contact Manage and oversee the project	Joint Administrative Operations FWS/Contracting Officer	Christine Beauregard		E-mail/phone communication with AI PM, and FWS Technical Team.
	Refuge FWS/Contract Officer's Representative	George Molnar	Cell:(973) 294-1997	E-mail/phone communication FWS, AI PM, AI Installation Lead and State and EPA Regulators.
Regulatory Oversight	AI/Installation Lead	Preston Sowell	Cell:(303) 775-6920	E-mail/phone communication with Installation Points of Contact, FWS, and AI PM.
Manage all Contract Phases	AI/Project Manager	Jeff Hart	Cell:(720) 884-7404	All materials and information about the project will be forwarded from AI PM to Installation Points of Contact or their representative. AI PM will notify Installation Points of Contact or their representative of field-related problems by phone, email, or fax by the next business day.
Health and Safety Oversight	AI/Quality Assurance Quality Control, Health and Safety Manager	Paul Hunter	Cell: (208) 899-6784	Ensure HASP addresses safe execution of all field work and designate a site-safety and health officer. If site conditions become unsafe, stop work and report to the AI PM.
Field and Analytical Corrective Actions	AI/ Field Team Leader	David Back	Cell:(703) 489-4554:	AI Field Team Leader (or designee) will e-mail or fax daily field progress reports to AI PM.; AI Project Chemist will email or Fax daily analytical corrective action requirements to Laboratory PM and AI PM
	AI/Project Chemist	Chris Lammer	Cell:(202) 600-2295	
Reporting Lab Data Quality Issues	Eurofins TestAmerica Project Manager	Aidan Scott	(646) 745-0906	All QA/QC issues with project field samples will be reported by Laboratory PM to AI Project Chemist within two business days. If corrective measures are required, AI Project Chemist will notify the Quality Manager and AI PM.

Table 6-1. Communication Pathways				
Communication Drivers	**Organization/Title**	**Name/Email**	**Phone Number**	**Procedure**
Release of Analytical Data	AI/Project Chemist	Chris Lammer	Cell:(202) 600-2295	AI Project Chemist will email or fax analytical data as requested by AI PM or AI Refuge Lead to Installation COR.
UFP-QAPP Amendments	AI/Project Chemist	Chris Lammer/chris.lammer@ap-in.com	Cell:(202) 600-2295	AI Project Chemist will e-mail any changes to this UFP-QAPP to AI PM and AI Refuge Lead. Specific personnel are listed in Worksheet #3.

AI – Applied Intellect, LLC
FWS – US Fish and Wildlife Service
HASP – Health and Safety Plan
PM – Project Manager
Refuge – Great Swamp National Wildlife Refuge
QA/QC – Quality Assurance/Quality Control
UFP-QAPP – Uniform Federal Policy Quality Assurance Project Plan

7. QAPP WORKSHEET #7 - PERSONNEL RESPONSIBILITIES AND QUALIFICATION

This worksheet identifies the responsibilities of each project role. In addition, the education and experience qualifications are described for each assigned personnel.

Table 7-1. Personnel Responsibilities and Qualifications

Name	Title/Role	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Christine Beauregard	FWS Contracting Officer	FWS	• Primary point of contact on all contractual matters	N/A
George Molnar	FWS Contracting Officer's Representative	FWS/Refuge	• Responsible for overall project execution and for coordination with base representatives, regulatory agencies, and FWS management	N/A
Michael Horne	FWS Refuge Manager	FWS/Refuge	• Provides Refuge management and support/insight during data collection and project execution	N/A
Supinderjit Kaur	Project Manager	USEPA	• Provides regulatory oversight	N/A
Jill McKenzie	Project Manager	NJDEP	• Provides regulatory oversight	N/A
Paul Hunter	Quality Manager	AI	• Responsible for defining field elements and developing the UFP-QAPP • Monitors field, laboratory, and validation activities to ensure compliance with UFP-QAPP requirements • Identifies non-conformances through QA/QC review activities/audits and recommends corrective action • Prepares reports for submittal	M.S. Geology, RG, over 30 years of experience
Paul Hunter	Health and Safety Manager	AI	• Responsible for development of Environmental Remediation Services Health and Safety Program. • Oversees preparation of HASP and Site Health and Safety Officer	B M.S. Geology, RG, over 30 years of experience

Table 7-1. Personnel Responsibilities and Qualifications				
Name	**Title/Role**	**Organizational Affiliation**	**Responsibilities**	**Education and Experience Qualifications**
Jeff Hart	Project Manager	AI	• Responsible for implementing all activities listed in TO • Prepares or supervises preparation of UFP-QAPP and approves document • Oversees field activities including sampling and visual monitoring • Prepares or supervises preparation of reports for submittal	B.S. Geophysics, Registered Professional Geologist (PG), Project Management Professional (PMP), 30 years of experience.
Preston Sowell	Installation Lead	AI	• Prepares project reports • Communicates with Project Manager and Installation Lead • Ensure HASP is followed	BS Environmental Science, 19 years of experience
David Back	Sr Hydrogeologist / Field Team Leader	AI	• Responsible for implementing all field activities • Communicates with Installation Lead and Project Manager • Monitors field activities to ensure compliance with UFP-QAPP requirements • Ensure HASP is followed	B.S. Geology/M.S. Hydrogeology, 35 years of environmental experience.
Chris Lammer	Project Chemist	AI	• Responsible for defining analytical requirements • Responsible for resolution of laboratory QC issues with Project Manager • Provides program-level QA/QC guidance to installation Points of Contact, AI Project Manager, and project team • Reviews validation reports before release to the project team	B.S. Chemical Engineering, Professional Engineer (PE), over 30 years of environmental engineering experience.
TBD	Driller	TBD	• Perform drilling activities • Licensed in New Jersey	N/A
Aidan Scott	Laboratory Project Manager	ETA	• Point of contact for AI from ETA • Responsible for adhering to laboratory SOW requirements	N/A

Table 7-1. Personnel Responsibilities and Qualifications				
Name	**Title/Role**	**Organizational Affiliation**	**Responsibilities**	**Education and Experience Qualifications**
Stella Cuenco	Validation Project Manager	Laboratory Data Consultant,	• Point of contact for AI for data validation • Responsible for adhering to validation SOW requirements	N/A

AI – Applied Intellect, LLC
ETA – Eurofins/TestAmerica
FWS – US Fish and Wildlife Service
HASP – Health and Safety Plan
N/A – Not Applicable
QA/QC – quality assurance/quality control
SOW – Statement of Work
TBD – To Be Determined
UFP-QAPP – Uniform Federal Policy Quality Assurance Project Plan

8. QAPP WORKSHEET #8 - SPECIAL PERSONNEL TRAINING REQUIREMENTS

This worksheet documents specialized training or course certification required on this project.

Table 8-1. Special Personnel Training Requirements

Project Function	Specialized Training – Title or Description of Course	Training Provider	Training Date	Personnel/Groups Receiving Training	Personnel Titles/ Organizational Affiliation	Location of Training Records/
Environmental Field Work	40-hour HAZWOPER Training	Qualified vendor	various	All AI and subcontractor personnel that will be onsite	AI Staff, subcontractors	Training records are maintained in the home office for each employee or onsite, as appropriate.
Environmental Field Work	8-Hour HAZWOPER Refresher Training	Qualified vendor	various	All AI and subcontractor personnel that will be onsite	AI staff, subcontractors	
Environmental Field Work	Ongoing training and monitoring to ensure field activities are performed in accordance with the SOPs	AI	various	All AI personnel that will be performing field work	AI Staff	
Environmental Field Work	CPR/Adult Standard First Aid	various	various	One AI person onsite at all times	AI staff	

AI – Applied Intellect, LLC
CPR – Cardiopulmonary Resuscitation
HAZWOPER – Hazardous Waste Operations and Emergency Response Standard
SOP – Standard Operating Procedure/Practice

9. QAPP WORKSHEET #9 - PROJECT SCOPING SESSION

Project Name/Number: Rolling Knolls Land Fill Data Gaps Investigation

Area of Concern: Great Swamp National Wildlife Refuge

Projected Date(s) of Sampling: October 20, 2020 through November 10, 2020

Site Location: Great Swamp National Wildlife Refuge, Morris County, New Jersey

Project Manager: Jeff Hart, Professional Geologist (PG), Project Management Professional (PMP), Applied Intellect, LLC

Scoping Meeting 1
Date: August 13, 2020

Table 9-1. Project Scoping Session Participants

Name of Meeting	Title/Role	Organization	Telephone Number	E-mail Address or Mailing Address
Graham Taylor	Refuge Supervisor-North Zone, North Atlantic-Appalachian Region	FWS	(413) 253-8356	graham_taylor@fws.gov
Michael Horne	Refuge Manager, Lenape National Wildlife Refuge Complex	FWS	Cell: (973) 417-9552	michael_horne@fws.gov 1547 County Route 565 Sussex, NJ 07461
George Molnar	COR Contaminants Biologist/Remedial Activities Coordinator North Atlantic-Appalachian Region IPM Coordinator Great Swamp National Wildlife Refuge, Lenape National Wildlife Refuge	FWS	Office: (973) 425-1222x4 Cell: (973) 294-1997	george_molnar@fws.gov 32 Pleasant Plains Road Basking Ridge, New Jersey 07920
Jeff Hart	Project Manager/ Senior Geologist	AI	Cell: (720) 884-7404	jeff.hart@ap-in.com 2801 Youngfield St, Ste 240 Golden Colorado 80204
Preston Sowell	Senior Environmental Scientist/ Installation Lead	AI	Cell: (303) 775-6920	psowell@geoticsolutions.com
David Back	Senior Hydrogeologist/ Field Team Leader	AI	Cell: (703) 489-4554	Davidback2@aol.com
Chris Lammer	Senior Environmental Engineer/Project Chemist	AI	Cell: (202) 600-2295	Chris.lammer@ap-in.com
Linda Ziccardi	Senior Ecological Risk Assessor	AI	Cell: (303) 619-5171	Linda.Ziccardi@ap-in.com

Scoping Session Purpose:

The FWS and AI Project team conducted the first of a series of DQO scoping sessions via teleconference to discuss regulatory oversight, Work Plan preparation (consisting of UFP-QAPP and HASP), and screening criteria and clean-up levels.

During the scoping meeting, the following key concepts were discussed:

- The FWS COR provided a general overview of the project objectives;
- Project Team reviewed the preliminary UFP-QAPP Worksheet 11 DQOs;
- Project Team presented initial considerations for analytical methods to be utilized for the Site Characterization.

Following the meeting, the team conducted a site walk to visit each site.

Key Action Items:

- David Back to conduct Site Walk on August 18, 2020 with George Molnar;
- AI will evaluate detection limits versus screening levels for SW 846 analytical methods to test for the same chemicals evaluated at the Landfill surface soils but the focus will be on characterizing subsurface soil and groundwater on the Refuge that is impacted by the Landfill;
- AI will continue to develop the UFP-QAPP worksheets based on the previous CERCLA investigation compounds of concern.
- Draft UFP-QAPP submittal date is September 1, 2020.

Complete scoping meeting minutes are provided in Appendix A.

10. QAPP WORKSHEET #10 - CONCEPTUAL SITE MODEL

This section presents the information used to develop a conceptual site model (CSM) for the study area, which is used to develop an understanding of what is known about the Site, to assist with the identification of data gaps, and to support development of DQOs for the study.

10.1 Site History

10.1.1 Site Description and History

The Rolling Knolls Landfill Superfund Site (the Site) is located in Chatham Township, New Jersey at the south end of Britten Road in the Green Village community (Figure 1). The Site consists of an approximately 140 acre landfill with an approximately 30 acre area of additional surface debris (i.e., waste was not observed below ground) spread along its western edge (Figure 2). The official National Priorities List (NPL) Site boundaries were delineated based on the extent of observable waste material across the Landfill's footprint; however, previous studies (e.g., Geosyntec 2018a) have documented Site-related contaminants outside of the Landfill footprint (i.e., outside of the currently designated Site boundaries). The Site is bordered on the north by a ballfield and shooting range owned by the Green Village Fire Department. The central and western portions of the Landfill are owned by a Trust created by the last will and testament of Angelo J. Miele, who was the former landfill operator. The current Trustee is Paul J. Miele (Geosyntec 2018b). The Refuge (Figure 1), owned by the United States Government and managed by the FWS, covers 7,768 acres. The Refuge boarders the Site to the east, south, and southwest (Figure 2) and approximately 35 acres of the Site (as the Site is currently defined) lies within the Refuge (Figure 2). The Refuge-portions of the Site and the Refuge area east and south of the Site are designated as Wilderness Area.

The Landfill received municipal and industrial wastes from Chatham Township and surrounding communities from the 1930s to approximately 1968 (Geosyntec 2018b). Previous investigations revealed that much of the waste consists of municipal solid wastes along with smaller areas of industrial waste. Foster Wheeler (2000) reported that Township of Chatham Board of Health (TCBH) records indicated that the municipal wastes included household refuse, residential septage wastes, construction and demolition debris, landscaping wastes, scrap metal, and tires. Homeowners and private trash haulers also reportedly brought household wastes to the Landfill. Further documentation by Foster Wheeler (2000) indicates that municipalities were disposing of sewage on top of the Landfill, which was restricted to the Township of Chatham by the mid-1960s.

Between 1955 and 1975 the TCBH required mosquito and rodent control measures, including surface water drainage and applications of minimal daily cover, which consisted of "swamp muck" obtained from the edges of the Landfill (Foster Wheeler 2000). TCBH records from 1962 indicate that landfill management also required the application of herbicides for weed control, the application of oil on the Landfill roads for dust suppression, and that dead animals were also disposed of in the Landfill. Geosyntec (2018b) reports that the Refuge-portion of the Landfill was

never properly covered after the Landfill was closed in 1968. Empirical reports (Geosyntec 2018a) indicate that much of the waste is still exposed at the surface and only a thin layer of soil covers other portions of the Landfill, which confirms that the entire landfill was never properly abandoned. Accessibility concerns raised after a 1974 wildfire at the Site prompted the Landfill owner (the Trust) to construct a series of raised fire roads. The fire roads were constructed between 1979 and 1982 and the Trust was issued a citation in 1979 for using unpermitted waste materials (reported as construction and demolition debris) in the road's construction.

10.1.2 Previous Investigations

This section describes the previous environmental investigations that have been conducted at the Site. For details regarding the investigation activities and results, refer to the relevant, cited reports.

A CERCLA Site Inspection (SI) was initiated in 1985 by USEPA Region II in response to reports of uncharacterized process wastes at the Site. Under that investigation, one surface soil and four sediment samples were collected on the Landfill and on Refuge property where landfill material reportedly did not exist (Foster Wheeler 2000). The samples were analyzed for volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), polychlorinated biphenyls (PCBs), organochlorine pesticides (OCPs), and metals. USEPA conducted a follow up investigation in 1986, during which they sought to define the Landfill depth, nature of soil contamination, and evaluate potential dioxin soil contamination. Eight borings were advanced and environmental samples were collected from multiple depths and analyzed for VOCs, SVOCs, PCBs, OCPs, metals, and dioxin (NUS Corporation 1986).

The FWS conducted a fish tissue and sediment survey in 1988 (FWS 1991). The study evaluated metals and polycyclic aromatic hydrocarbons (PAHs) present in fish tissue, and metals, OCPs, and PAHs present in sediment. The sampling locations were reportedly biased around potential source areas and roadways.

In 1989, the FWS and U.S. Geological Survey conducted a joint investigation of Loantaka Brook and the perimeter of the Landfill (the location of the perimeter investigation has not been verified). Surface water, sediment, and groundwater samples were collected and analyzed for metals, VOCs, and SVOCs. Sediment samples were collected and analyzed for metals.

USEPA conducted a current conditions investigation in 1999 (Foster Wheeler 2000). Seven soil borings were advanced and two monitoring wells were installed. Additionally, 21 surface water, 10 groundwater, 21 sediment, and 15 soil samples were collected and analyzed for VOCs, SVOCs, PCBs, OCPs, and metals. A follow up sampling event was conducted in 2000 to collect additional data.

The FWS conducted a 10-year follow-up investigation in 1999 relative to its 1988 investigation (FWS 2005). The objectives of this investigation were to:

- Quantify the concentrations of metals, OCPs, and PAHs in Refuge sediment, and metals and OCP concentrations in fish tissue;
- Compare those data to the data from 1988; and,
- Identify the potential change in sediment and fish tissue concentrations between the two sampling events.

All of the samples were collected within the Refuge boundaries because FWS was not granted access by surrounding property owners. Samples were also not collected within the Refuge portion of the Site.

During two field events in 2003, the USEPA Region II Site Assessment Team (SAT) conducted phased investigations at the Site:

- Phase I involved the collection of soil and sediment samples, which were screened for PCBs;
- Phase II involved the collection soil and sediment samples for laboratory confirmation of the Phase I screening results; and
- Additional sediment and soil samples were collected from locations where drums or other visual indications of possible source material were observed.

The additional samples were analyzed for Target Compound List and Target Analyte List (TAL) (excluding cyanide) constituents. Additional sample volumes were collected at the sediment sampling locations and analyzed for total organic carbon (TOC) and particle size (Weston Solutions Inc. 2003a, 2003b and 2003c). Subsequently, the SAT issued a Hazard Ranking System package in April 2003 and on September 29 of the same year, the Site was listed on the NPL - Site ID NJD980505192.

Subsequent to a potentially responsible party (PRP) search, a number of PRPs were identified as contributors to the hazardous substances found in the Landfill. The USEPA signed an Administrative Settlement Agreement and Order on Consent (Agreement; CERCLA-02-2005-2034) with Chevron Environmental Management Company, Alcatel Lucent USA Inc., and Novartis Pharmaceuticals Corporation (collectively, the Group) on September 30, 2005. Subsequent to the Agreement, the Group conducted investigations under USEPA oversight.

A Phase I RI was initiated by the Group in 2005 with a second phase conducted in 2014 and 2015. The objectives of Phase I RI included:

- Characterizing the Site's geology and hydrogeology;
- Characterizing landfill waste;
- Defining chemicals of potential concern (COPCs) and environmental media; and,
- Providing data to support risk assessments and remedy selection.

The results of the Phase I RI were reported in the Site Characterization Summary Report (Arcadis 2012) and the results were used to generate a Baseline Human Health Risk Assessment (BHHRA; CDM 2014) for the Site. A number of data gaps were identified after the Phase I RI was completed and a data gaps investigation was conducted between November 2014 and January 2015 to further define the nature and extent of contamination at the Site, and to provide additional data to support a Baseline Ecological Risk Assessment (BERA) and remedial alternative evaluations for the Feasibility Study (FS). The data gaps analysis investigation results were reported in the Data Gaps Technical Memorandum (Geosyntec 2016). The data gaps investigation also involved the collection of data used to evaluate the efficacy of monitored natural attenuation (MNA) at the Site, which was reported in the Supplemental Groundwater and Baseline Monitored Natural Attenuation Investigation Report (Geosyntec 2017).

Additional samples were collected in 2016, specifically to support the BERA (Integral 2016). The BERA report was completed in the same year.

To evaluate Site-specific, reuse-related considerations for the identification of reasonably anticipated future Site uses, the Group conducted a reuse assessment. The results were provided in the Reuse Assessment Report (TRC 2017a) and supplemented in a Reuse Assessment Addendum (TRC 2017b). The conclusions of the Reuse Assessment Addendum were that "the potential reuse of the Site is limited by:

1. The presence of extensive and state- and federally-regulated areas that limit development;
2. The environmentally-sensitive nature of the surrounding area;
3. State, county, and local planning documents that discourage development in environmentally-sensitive areas away from established centers and focus on protection of the [Refuge];
4. The lack of available infrastructure and associated Site accessibility issues; and
5. The presence of buried waste which complicates construction and makes it costlier."

A Final RI Report was submitted in January of 2018 (Geosyntec 2018a) and a Revised Final FS Report was submitted in July 2018 (Geosyntec 2018b). The sample locations from the RI and BERA are illustrated on Figure 3.

A summary of the Revised Draft FS alternatives with their key feature(s) relative to the impacts on the Refuge portion of the Site is provided below:

Alternative	**Key Features Relative to Impacts on the Refuge [a]**
Landfill 1[b]	No Action
Landfill 2	Site controls (i.e., Institutional Controls, Fencing and Signage);

Landfill 3	Capping of selected areas to reduce overall risk, remediation of "Areas of Particular Concern (APCs)", and remediation of non-vegetated areas of soil contamination above remediation goals. (Note: only one APC was identified in the Refuge, associated with soil sample SS-118 in the south part of the Landfill.)
Landfill 4	Excavation and off-site disposal of selected areas to reduce overall risk, remediation of APCs, and remediation of non-vegetated areas of soil contamination above remediation goals. (Note: only one APC was identified in the Refuge, associated with soil sample SS-118.)
Landfill 5	Capping of all landfill material (including the Refuge portion of the Site).
Groundwater 1	No Action with naturally occurring constituent of concern reductions.
Groundwater 2	Source control, institutional controls, constituent of concern reduction by ongoing natural processes, long term monitoring with potential need to make adjustments to the remedy in the future.
Groundwater 3	Source control, institutional controls, constituent of concern reduction by ongoing natural processes, long term monitoring with implementation of a contingent remedy.

[as] To address the area of the Refuge significantly impacted by the Site/landfill, and to comply with the Refuge Comprehensive Conservation Plan, the DOI ECM, and all other applicable or relevant and appropriate requirements.

[b] The Revised Draft FS refers to these alternatives as "Soil" alternatives; however, they address the source landfill waste as well as the soils contaminated by the Landfill waste and are more appropriately labeled as "Landfill" alternatives.

A critical review of the FS conducted by KMPower (2019) on behalf of FWS concluded that significant areas of the Refuge contain contaminated surface soils resulting from Landfill waste activities that pose an elevated risk to wildlife and recreational users. KMPower (2019) concluded that the preliminary remediation goals (PRGs) proposed in the Revised Draft FS (Geosyntec 2018b) were not adequate to protect wildlife or recreational visitors, including children, at the Refuge.

KMPower (2019) concluded that a comprehensive assessment of sediment contamination and its associated impacts was not conducted to support alternatives that would allow source landfill waste to remain on the Refuge and adjacent non-Refuge portions of the Site without containment, which presents a limiting data gap for FS decision making. Contaminants are reported at concentrations in excess of promulgated New Jersey groundwater quality limits and New Jersey groundwater quality limits are potentially applicable requirements (KMPower 2019). Only one alternative in the Revised Draft FS (Alternative 5) has the potential to prevent further migration of contaminants from the landfill waste into the groundwater. The remaining alternatives allow source landfill waste to remain onsite without containment. The FS did not

address current groundwater contamination, citing conclusions from the MNA study (Geosyntec 2017).

Several of the major conclusions, with respect to groundwater contamination from the Geosyntec (2017) MNA study, however, are not factual. The text states (p. 10, Section 3.2.1) that, "The data show that both dissolved and total metals concentrations in groundwater fluctuate, but have generally been stable over time since 2007. This indicates that natural processes upgradient, downgradient and within the Landfill are immobilizing and sequestering the metals in the soil matrix." Relatively stable concentrations of dissolved metals do not indicate that "….natural processes upgradient, downgradient and within the Landfill are immobilizing and sequestering the metals in the soil matrix." In fact, the dissolved metals concentrations indicate just the opposite, that the dissolved metals are readily available to migrate in the groundwater, as indicated by detections of dissolved metals in virtually every monitoring well. Dissolved metals will continue to migrate into the wetlands until, either their source(s) are depleted, or geochemical conditions change to those more favorable for sequestering the metals.

The Geosyntec (2017) MNA study text also states that (p. ix), "Metals are mostly not detected in filtered groundwater samples, indicating that metals concentrations are present in colloidal fractions, which are not readily transported with groundwater." As described above, dissolved metals have been detected at concentrations that exceed New Jersey's Groundwater Quality Standards in every monitoring well.

The decision to not include current groundwater contamination in the FS based on the assumptions that dissolved metals are mostly not detected in the groundwater, and that the metals are immobilized and sequestered in the soil matrix is not supported by the data.

10.2 Sources of Known or Suspected Hazardous Waste

Several known and potential sources of hazardous substances have been documented for the Site. As discussed in Section 10.1.1, the Landfill received municipal solid wastes and septic wastes from municipalities, private haulers, and homeowners for twenty years. Industrial wastes were also reportedly disposed of in the Landfill and the observations of drums containing such wastes appear to confirm those reports (Geosyntec 2018a). Pesticides, herbicides, and oil were used to control rodents, mosquitoes, and dust as part of the Landfill's operation. Though not confirmed by any of the previous investigations, other potential contaminant sources could be skeet shooting activities (i.e., PAHs from clay pigeon fragments and lead from shot) at the north end of the Site, lead from on-site hunting activities, petroleum hydrocarbons from the storage and maintenance of vehicles and landscaping equipment, and unauthorized dumping (Geosyntec 2018a). The unpermitted construction and demolition debris that was imported to build fire roads across the Site could also have introduced contaminants.

The 2018 RI Report also documented a number of potential upgradient sources of contaminants that could possibly contribute to surface water and sediment contamination within Loantaka Brook and Black Brook. The potential upgradient sources included greenhouses, sewage

treatment plants, runoff from roadways and golf courses, and a shooting range (see Section 4.1, Geosyntec 2018a).

10.3 Known or Suspected Contaminants

The specific COPCs for human health are identified in the 2014 BHHRA (CDM 2014). The COPCs for the Site for soils by class include VOCs, SVOCs, pesticides, PCBs, dioxins/furans, and metals. The COPCs for the Site for surface water by class include SVOCs, PCBs, and metals. The COPCs for the Site for sediment by class include VOCs, SVOCs, pesticides, PCBs, dioxins/furans, and metals. The COPCs for the Site for groundwater by class include VOCs, SVOCs, and metals.

The specific contaminants of potential ecological concern (COPECs) for ecological receptors are identified in the 2016 BERA. The COPECs for the Site by class include SVOCs, pesticides, PCBs, dioxins/furans, and metals.

10.4 Release and Transport Mechanisms

Hazardous substances including VOCs, SVOCs, PCBs, pesticides, dioxins/furans, and metals are found in surface and subsurface soils across the site. Many of the same contaminants have been detected in surface water, sediment, and groundwater across the Site and adjacent to the Site in the Refuge. Much of the Landfill waste is exposed at the surface, or only covered by a thin layer of soil (Geosyntec 2018a). The precipitation that falls on the Landfill that does not evapotranspire back into the atmosphere either infiltrates through the Landfill material and into the shallow groundwater below, or runs off into the onsite ponds or to the adjacent wetlands, including the Refuge. The meteoric water infiltrating through the waste material could mobilize contaminants from the waste and into the groundwater below. Runoff can mobilize waste material and contaminated soil into the adjacent waterways and wetlands, contaminating both sediment and surface water.

Groundwater is in contact with the waste material across the Site, which has contaminated the groundwater (Geosyntec 2018a). The groundwater redox conditions are highly reducing beneath the Site, which can further mobilize metal contaminants from the waste material that would otherwise not be soluble. A confining clay layer is present at depths of generally less than 20 feet (ft) below ground surface (bgs) across the Site, which is expected to limit the potential vertical migration of contaminated groundwater. As previously mentioned, groundwater is at or near the ground surface across the Site and in hydraulic communication with surrounding surface water bodies, including the Refuge's wetlands (Geosyntec 2018a). Therefore, contaminated groundwater could potentially contaminate surrounding surface water and the sediment at, and downstream from groundwater-surface water discharge locations. As the redox conditions change when groundwater leaves the Site, contaminants with solubilities that were redox-dependent (e.g., some dissolved metals) would be expected to precipitate out of solution, potentially leading to high contaminant concentrations at those redox inflection points, either within the subsurface soils or in sediments at the surface water discharge location.

With respect to the influence that redox reactions have on the speciation and mobility of heavy metals, the conceptualization is explained by Geosyntec (2017, p. 11) which states that, "*Groundwater at well MW-7, located in the middle of the Landfill, is highly reducing and methanogenic; it also has relatively high concentrations of total and dissolved organic carbon. The groundwater geochemistry at well MW-1 is similar to well MW-7, it is methanogenic with high concentrations of total and dissolved organic carbon. Due to the heterogeneous nature of landfills, these conditions may not be consistent across the entire site. The groundwater sample from downgradient well X-3* [Figure 3] *is oxidizing with a positive [oxidation reduction potential (ORP)], and there is no evidence of methane and total or dissolved organic carbon. There is no nitrate and the data suggest some manganese reduction, but overall, it is the most oxidizing of the four sample locations."* As shown in Figure 3, monitoring well MW-2 is located between MW-1 and X-3. At MW-2, dissolved arsenic, manganese, thallium and iron concentrations have been detected above New Jersey Ambient Groundwater Quality Standards indicative of reducing conditions (see RI Figure 4-2). Therefore, between MW-2 and X-3 there is a geochemical transition zone in which conditions become more oxidizing and metals precipitate out of solution. It is not known to what degree the metals precipitate within the aquifer matrix, pore-water, sediment, or surface water. If the metals precipitate out of the groundwater at greater depths, they should not result in adverse ecological impacts. However, if the metals remain in solution until entering shallower more oxidizing environments (e.g., pore-water, sediment, surface water), they could result in ecological impacts and potential human exposures. Furthermore, these dissolved metals could be bioconcentrated within the pore water and sediment due to 1) evapotranspiration removing the water but leaving the metals behind, and 2) bioaccumulation within the plants during the growth cycle and subsequent release and bioconcentration within the detritus following the plants death.

The geochemical transition zone between reducing and more oxidizing conditions likely forms a relatively narrow band, starting near the interface between the terrestrial system and the wetlands and ending several hundred feet within the wetlands.

For the purposes of this investigation, pore-water is defined as water within the zone being affected by evapotranspiration, and potentially discharging to surface water..

Dissolved metals have been detected in the 7 monitoring wells located along the geochemical transition zone on the Refuge property (i.e., MW-2, MW-4, MW-12, MW-14, MW 19, X-1 and X-2; Figure 3). Dissolved metals will continue to migrate into the wetlands until either their source(s) is depleted or geochemical conditions change.

10.5 Potential Receptors and Exposure Pathways

Relative to human health, receptors on the Refuge portion of the Site are expected to be limited to recreational users, which may occur through exposure to contaminated surface soils, sediments, surface water, and possibly shallow groundwater at groundwater-surface water discharge locations. The potential exposure routes for contaminated media at the Site include incidental ingestion, dermal contact, and inhalation.

Relative to ecological risk, receptors on the Refuge portion of the site are expected to be terrestrial birds and mammals, semi-aquatic birds and mammals, and aquatic vertebrates and invertebrates.

10.6 Land Use Considerations

As the Refuge portion of the Site and the adjacent wetlands are a designated Wilderness Area, land use would be limited to recreation.

Relative to portions of the Site outside of the Refuge, a portion of the Site is called the 'laydown area', which is used by two waste hauling companies (Chatham Disposal and South Orange Disposal) for storing and staging solid waste roll-off bins. The ballfield and shooting range located at the northern edge of the Site are occasionally used for recreational purposes.

Recent land uses that are no longer conducted:

- Two landscaping companies rented portions of the Landfill and surface debris areas for equipment storage and maintenance activities. These landscaping companies no longer rent this land.
- A small building located in the surface debris area, called the Hunt Club, was occasionally used for social functions. This activity is not currently taking place.

10.7 Environmental Setting

This section describes the environmental setting for the Site and surrounding properties.

10.7.1 Climate

The climate of the Chatham Township, New Jersey area is classified as humid continental, consisting of cold winters and warm summers. The mean annual temperature is approximately 51°F, with the coldest average temperature of 29°F occurring in January and the warmest average temperature of 73°F occurring in July. The coldest mean daily temperatures below 40°F occur between December and March (Rutgers 2016a).

The mean monthly precipitation ranges from 3.06 to 4.65 inches, with an annual total mean precipitation of approximately 47 inches. Rainfall is spread throughout the year, with the wettest months being July and August (Rutgers 2016b).

The prevailing wind is from the southwest in the summer and from the northwest during the remainder of the year. Average wind speeds range from 9 to 17 miles per hour (USA.com 2016).

10.7.2 Topography

The topography is relatively flat and poorly drained, consisting mostly of low-lying areas including the Refuge wetlands (Gill and Vecchioli, 1965). The Site's topography is illustrated on Figure 4. The Site and the surrounding area lie at an elevation of approximately 240 feet above mean sea level (amsl; Geosyntec 2018a). Survey data from the RI Report (Geosyntec 2018a) soil boring locations advanced throughout the Site and in the adjacent lower areas indicate that the ground elevations ranged from 227 to 250 feet amsl (Geosyntec 2018a). The fire roads built across the site are elevated approximately four ft above the surrounding landscape.

10.7.3 Surface water Drainage

The Site is relatively flat and poorly drained with some saturated areas and wetlands existing on the Site itself; however, the Site is relatively elevated above the surrounding wetlands due to landfilling. Precipitation that does not evapotranspire or infiltrate through landfill wastes to groundwater is expected to runoff. Several on-Site ponds are expected to receive inputs from surface runoff as well as groundwater. Sheet flow from the Site is expected to also run off into the surrounding wetlands.

Black Brook flows from north to south near the eastern boundary of the site. Though portions of Black Brook are channelized, the majority of the surface water flow is un-channelized, low energy flow through dense wetlands.

10.7.4 Soils

Though the thin soil layer over the Landfill is discontinuous, most of the Landfill soils are classified as Udorthents, Refuse Substratum. The soil is characterized as silty loam that is spread over organic material, and is classified as well drained and does not frequently flood (USDA 1976). A second soil type called Carlisle Muck is reportedly present in the southern part of the Landfill (Geosyntec 2018a). That soil type is characterized as very poorly drained and frequently flooded. It is typically found in floodplains and is composed of herbaceous and/or woody organic material (USDA 1976).

10.7.5 Geology

The Site lies within a former glacial lake called Lake Passaic, which was formed during the Wisconsin Glaciation (Geosyntec 2018a). Sediments deposited within the lake include till and glaciolacustrine sand and gravel, silt, and clay. Regionally, thick deposits of the Wisconsin Glaciation terminal moraine lie to the northeast of the Landfill and the third basalt sheet, locally known as Long Hill, lies directly to the south (Gill and Vecchioli, 1965). See Geosyntec (2018a), map showing local surficial geology on and near the Landfill. Two overburden units are mapped within the Landfill and include stream terrace deposits and swamp and marsh deposits. In general, the Landfill is underlain by post-glacial swamp deposits interbedded with silt and sand. Coarser material was deposited directly from the ice and finer sediments were deposited at slower rates from remnant lakes following the retreat of glacial ice. Peat was deposited from

shoreline vegetation of these remnant lakes and continues to be deposited throughout the heavily vegetated low-lying areas (Minard, 1967).

The most significant unconsolidated glacial sediments in the area of the Landfill are the glacial lake clay deposits. The glacial lake clay is characterized as medium to light gray and grayish-red to pale-red plastic clay containing intermixed silt. The clay forms a thick deposit that underlies the entire area and is reportedly more than 100 feet thick (Minard, 1967).

10.7.6 Hydrogeology

The hydrostratigraphic units at the Site generally consist of silt, peat and other organic materials, overlying stratified drift and sand channels (where present), beneath which lies a thick clay unit acting as an aquiclude (Geosyntec 2018a). The clay layer is located at depths of less than 20 ft bgs across the Site. A Site geologic cross section from Geosyntec (2018b) is included as Attachment 1. Groundwater is found at or within approximately five feet of the ground's surface across the Site.

10.7.7 Critical Habitats/Threatened or Endangered Species

The Site is surrounded by wetlands on its eastern, southern, and southwestern boarders (Figure 4). Wetlands also occur on the Landfill itself. Additionally, the Refuge-portion of the Site, along with the Refuge area adjacent to the Site are a designated Wilderness Area.

Threatened and Endangered species occurring at the Site include (Arcadis 2016):

- Bog Turtle (*Glyptemys muhlenbergii*), SE/FT
- Wood Turtle (*Glyptemys insculpta*), ST
- Blue-spotted Salamander (*Ambystoma laterale*), SE
- Barred Owl (*Strix varia*), ST
- Red-shouldered Hawk (*Buteo lineatus*), SE
- American Bittern (*Botaurus lentiginosus*), SE*
- Red-headed Woodpecker (*Melanerpes erythrocephalus*), ST
- Bobolink (*Dolichonyx oryzivorus*), SE*

Notes:
SE: State Endangered; ST: State Threatened; FT Federally Threatened
*Breeding population

10.7.8 Conceptual Site Model Figure

A graphical CSM illustrating the key Site features described in this section is included as Figure 5.

10.7.9 Data Gaps and Uncertainties

A review of the CSM reveals several data gaps relative to the Refuge-portion of the site and the potential migration of contaminants from the Site into the adjacent Refuge.

Subsurface soil and subsurface landfill waste has not been characterized for COPCs/COPECs throughout the Landfill, and specifically on the Refuge-portions of the Landfill. Twenty-one test pits were excavated on the Refuge to log landfill debris. Chemical testing was not conducted in the Landfill waste but focused on the Landfill-debris/natural soil interface. In addition, 14 of the 21 test pits were excavated at the edge of the Landfill to document horizontal extent of subsurface debris. Test pits within the Refuge study area are illustrated on Figure 3.

In addition, large areas of the Refuge portion of the Site were not sampled at all. The unsampled areas are also adjacent to some of the highest contaminant concentrations measured in soils (Geosyntec 2018a, 2018b). Examples of these unsampled areas are illustrated on Figures 6a and 6b. Note that the approximately 7 acre, unsampled area located in the northeast portion of the Site also lies adjacent to the area where the Revised Draft FS suggests that risks associated with high levels of contamination in that area indicate the need for a remedial action (Geosyntec 2018b). The northeast unsampled area also lies adjacent to wetlands in Refuge Wilderness Area.

A critical review of the RI/FS documents found that the sediment information collected in Black Brook during the RI was insufficient to determine the nature and extent of contamination, particularly with respect to potential upstream sources (KMPower 2019). Since Black Brook sediments have not been adequately characterized, the associated risk has not been fully assessed. Though 28 sediment samples were collected from within the Refuge, the majority of them were placed greater than 100 ft from the edge of the Landfill material, often 100s of feet from the nearest adjacent sample location (Figure 3). Considering that relief between the landfill and the surrounding wetlands is only a few feet, that the surface water flows throughout the wetlands are expected to be fairly low energy, and that the wetlands themselves are highly vegetated, any contaminated sediment transported off of the Site in overland flow events would not be expected to move far. Additionally, there are uncharacterized ponding areas near areas of high surface soil contamination on and off of the Site within the Refuge (Figure 6a). Notably, a pond that is clearly visible on many of the historical aerial photographs and satellite images lies at the northeast corner of the Site, near the contaminated area proposed for remedial action in the FS (Figure 6a; Geosyntec 2018b); however, the pond was not identified in the RI/FS documents and only one sediment sample was collected at its edge. Further, the surface flow patterns shown in RI Report figures (Figure 6a; Geosyntec 2018a) indicate that the surface runoff flows directly from those highly contaminated surface soils and into the pond area, but the sediment sample collected from the edge of the pond was not collected were the surface runoff would be deposited into the pond (Figure 6a).

The RI Report (Geosyntec 2018a) concluded that, “Black Brook likely receives hydrologic input from groundwater discharge,” and groundwater flows from the Site to Refuge property (Figures 6a and 6b), which indicates that the contaminated groundwater plume from the Site can be expected to discharge into surface waters on the Refuge at some point (if it is not currently). This represents a likely complete exposure pathway from contaminated groundwater to human and

ecological receptors within the Refuge. Furthermore, contaminated groundwater has the ability to contaminate surrounding surface water features, sediment pore water, and sediment at, and downstream from groundwater-surface water discharge locations. As the redox conditions change when groundwater leaves the Site, contaminants with solubilities that were redox-dependent (e.g., some dissolved metals) would be expected to precipitate out of solution, potentially leading to high contaminant concentrations at those redox inflection points, either within the subsurface soils or at the surface water discharge location [see discussion in Section 10.4).

A review of the previously collected Site data also indicates that 'emerging contaminants' often associated with landfill wastes and likely to occur at the Site were not included in previous analyte lists. According to USEPA (2014[c]), "An 'emerging contaminant' is a chemical or material that is characterized by a perceived, potential, or real threat to human health or the environment or by a lack of published health standards. A contaminant may also be "emerging" because a new source or a new pathway to humans has been discovered or a new detection method or treatment technology has been developed (DoD 2011)." These emerging contaminants — often detected in groundwater, surface water, sediment and soil — include Per- (and Poly-) Fluoro Alkyl substances (collectively, PFAS) such as Perfluorooctanoic Acid (PFOA) and Perfluorooctane Sulfonate (PFOS). The PFAS compounds were historically used in several consumer products and industrial applications. Another emerging contaminant frequently detected at landfill sites includes 1,4-Dioxane, which was used in solvents, paint strippers, dyes, greases, varnishes and waxes and is typically detected at sites where solvents are present. 1,4-Dioxane is also highly mobile in groundwater.

The data gaps and potential release mechanisms noted above also indicate that the remedial alternatives proposed in the Revised Draft Final FS (Geosyntec 2018b) would not be protective of human health and the environment on the Refuge portion of the Site or on the off-site portions of the adjacent Refuge.

11. QAPP WORKSHEET #11 - PROJECT DATA QUALITY OBJECTIVES/SYSTEMATIC PLANNING PROCESS STATEMENTS

This section presents the DQOs following the USEPA Guidance of Systematic Planning Using the DQO Process (USEPA 2006). The DQO process specifies anticipated project decisions, specific data types needed, the data quality required to support decisions, data collection requirements, and analytical techniques necessary to generate the specified data quality. The process also ensures that the resources required to generate the data are justified.

The DQO process consists of the following seven steps, described in detail below:

1. State the Problem.
2. Identify the Goal of the Investigation.
3. Identify the Information Inputs.
4. Define the Boundaries of the Investigation.
5. Develop the Analytic Approach.
6. Specify Performance or Acceptance Criteria.
7. Develop the Plan for Obtaining Data.

The following sections detail each step in the DQO process for this investigation.

11.1 State the Problem

The existing data are insufficient to determine extent of contamination and to evaluate the potential effectiveness of the proposed remedial alternatives. This investigation will be conducted to supplement existing data sets. The combined data will be used to fill data gaps relative to the nature and extent of environmental contamination within sediment, sediment pore water, surface water, groundwater, soil and landfill wastes on Refuge property, and if warranted, to develop a remedial alternative consistent with the Refuge's Comprehensive Conservation Plan (CCP) and Refuge-specific applicable or relevant and appropriate requirements (ARARs).

11.2 Goals of the Study

The environmental samples and data collected under this study are intended to address several existing data gaps surrounding the nature and extent of contamination on the Refuge and for the development of a remedial alternative that is consistent with the Refuge's CCP and Refuge-specific ARARs.

Therefore, the main goals of this project are to answer the following study questions:

1. What are the distributions and concentrations of sediment contaminants on, and adjacent to the Refuge portion of the Site in uncharacterized areas?

2. What are the distributions and concentrations of surface water contaminants on, and adjacent to the Refuge portion of the Site in uncharacterized areas?
3. What are the distributions and concentrations of contaminants within pore water and sediment at potential groundwater discharge to surface water areas adjacent to the Refuge portion of the Site in uncharacterized areas?
4. What are the distributions and concentrations of pore water (shallow groundwater) contaminants on, and adjacent to the Refuge portion of the Site in uncharacterized areas?
5. What are the distributions and concentrations of soil contaminants on the Refuge portion of the Site in uncharacterized areas?
    a. Large areas of surficial soils near previously identified hotspots have not been chemically characterized (See Figures 6a and 6b); therefore, additional surface soil samples are required to:
        i. Evaluate exposure point concentrations (EPCs) of contaminants in select areas; and,
        ii. Determine whether surface soils from on-Site portions of the Refuge could act as source areas.
    b. A primary data gap surrounding the Refuge portion of the Site is the lack of environmental samples collected at depth within the Landfill. These data are needed to identify potential hotspots within the Landfill waste, to understand the leaching potential of the Landfill waste (now and after it may be disturbed), and to generate a potential remedial alternative consistent with the Refuge's CCP and Refuge-specific ARARs.
6. What are the chemical properties of the soil between the Landfill wastes and the underlying clay layer on the Refuge portion of the Site (i.e., have contaminants migrated from the overlying landfill into the native soils beneath)?

The possible alternative outcomes of study questions 1-5(a) include:

- The data confirm releases of Site-related contaminants into the environment, and specifically, the Refuge.
- The data confirm releases of Site-related contaminants into the Refuge with EPCs that indicate the need for further response action(s).

The decision statement for study questions 1-5 is: Use the supplemental data collected under this study, in conjunction with previously collected Site data (that meets the study's DQOs) to determine the need for, or extent of remediation required to reduce risks to human health and ecological receptors on Refuge property and to prevent future Site-related contaminant releases to off-Site portions of the Refuge.

The possible alternative outcomes of study questions 5(b) and 6 include:

- The data are sufficient to support the development and implementation of a remedial alternative that removes all landfill wastes from the Refuge portion of the Site.

- The data are insufficient to support the development and implementation of a remedial alternative that removes all landfill wastes from the Refuge portion of the Site.

The decision statement for study questions 5(b) and 6 is: Use the supplemental data collected under this study, in conjunction with previously collected Site data (that meets the study's DQOs) to design a potential remedial alternative consistent with the Refuge's CCP and Refuge-specific ARARs.

11.3 Information Inputs

This section specifies the types of data that are required to address the data gaps relative to the CSM associated with the Refuge portion of the Site and adjacent Refuge properties.

11.3.1 Previous Data Usability

As detailed in Section 10, previous investigations have been conducted at the site under USEPA oversight, which produced data that may be useful for this study.

The initial investigations that were used to define the Site were conducted between 1985 and 2003 (Geosyntec 2018a; Geosyntec 2018b). Subsequently, the Group conducted an RI between 2005 and 2011. Samples intended specifically for ecological risk assessment were collected for a BERA in 2016 (Integral 2017). The data from the RI and BERA reports were available for review and were compared to the DQOs developed for this study.

The data collected as part of the RI and BERA were approved for their intended uses by USEPA. A subset of those data are from the Refuge portion of the Site and are deemed of sufficient quality to inform the placement of locations for this study and in the evaluation of this study's investigation-derived data. Groundwater will also be evaluated on a site-wide basis using existing datasets. The usable data from previous studies includes:

- 59 soil samples (56 include surface soils);
- 28 sediment samples;
- 7 surface water samples;
- 2 pore water samples;
- Groundwater samples from 27 monitoring wells and 9 temporary well points, site-wide (includes multiple rounds of sampling between 2007 and 2015);
- Ground water elevations from 27 monitoring wells, site-wide;
- Soil boring logs from 9 locations; and,
- Test pit logs from 29 locations.

The distinction between soil and sediment samples in the RI is confusing because some samples identified as "soil" were submerged under water at the time of collection (e.g. SS-162), and some samples identified in the RI as "sediment" were not submerged at the time of collection (e.g., SD-

61). It is unclear exactly how these samples were used to characterize the Site within the RI. For the purposes of this study and to evaluate the potential areal extent of contamination on the study area, the surface soil samples within the study area that were re-classified as a sediment matrix (e.g., SS-162) will be evaluated with the other soil matrix samples; this use is also consistent with the approach taken in the RI for other areas of the Site (Geosyntec Consulting 2018b, Section 4.9.2).

The locations for these previously collected data are illustrated in Figure 3.

11.3.2 Data to be Collected in the Current Investigation

Table 11-1 below lists the data requirements for the study and how each type of data will be used to help answer the investigation questions. Quality control samples are discussed in Section 14.7. The Field Sampling Plan (FSP) including the sampling design and rationale is detailed in Section 17 Worksheet #17.

Table 11-1: Data/Samples Required

Data Type	Data Purpose
Sediment contaminant concentrations collected from open surface water bodies and/or surface water drainage swales within the Refuge portion of the Site and adjacent Refuge property.	Evaluation of potential migration pathways, and the spatial extent and relative concentrations of site-related contaminants.
Sediment contaminant concentrations from upgradient of the Refuge portion of the Site.	Evaluation of potential upgradient sediment contaminant contributions.
Survey to identify potential groundwater discharge points within the Refuge portion of the Site and adjacent Refuge property.	Evaluation of potential groundwater discharge points to surface water and to optimize pore-water/sediment/surface water sampling locations.
Sediment pore water and collocated sediment contaminant concentrations from areas adjacent to the Site where contaminated groundwater is expected to discharge.	Evaluation of contaminated groundwater potentially discharging into Refuge wetlands.
Surface water contaminant concentrations from within the Refuge portion of the Site and adjacent Refuge property.	Evaluation of potential migration pathways, and the spatial extent and relative concentrations of site-related contaminants.
Surface water contaminant concentrations from upgradient of the Refuge portion of the Site.	Evaluation of potential upgradient surface water contaminant contributions.
Surface water flow measurements from the Refuge portion of the Site and adjacent Refuge property.	Evaluation of the relative contributions of groundwater to surface water flow.
Pore water (shallow groundwater) contaminant concentrations from within the Refuge portion of the Site and adjacent Refuge property.	Evaluation of potential migration pathways, and the spatial extent and relative concentrations of site-related contaminants in pore water and where contaminated pore water might be discharging into surface water.

Data Type	Data Purpose
Pore-water quality parameters from within the Refuge portion of the Site and adjacent Refuge property.	Evaluation of overall pore-water quality and provides an indication of the presence of, and types of contaminants potentially present in pore water. This information will also enable pre-remedial implementation baseline conditions to be established.
Speciation of metals (specifically Fe and Mn) in pore water from within the Refuge portion of the Site and adjacent Refuge property.	Provides an indication of the ORP of pore water flowing through the Landfill wastes and of the implications of changing that environment through implementation of a remedial alternative.
Surface soil contaminant concentrations from within the Refuge portion of the Site.	Evaluation of potential hotspots/source areas, migration pathways, and the spatial extent and relative concentrations of Site-related contaminants.
Subsurface soil and waste contaminant concentrations and fraction of organic carbon from within the Refuge portion of the Site (i.e., the Landfill on the Refuge portion of the Site).	Determine physical, chemical, and geotechnical properties of the soil and wastes to evaluate fate and transport of contaminants and the leaching potential of the Landfill wastes, and identify potential hotspots/source areas, and provide information necessary for remedial design and implementation.
Subsurface soil contaminant concentrations below the Landfill in the Refuge portion of the Site to the upper bound of the underlying clay layer.	Determine whether the soils beneath the Landfill wastes have been contaminated and whether they would be classified as a hazardous waste.
GPS coordinates of sample locations	Documentation of sample locations and evaluation of the spatial distribution of contamination across the site.
Visual descriptions of soils at soil boring locations	Provides a potential visual indication of waste material and mineral speciation (i.e., color associated with oxidation/reduction) and for saturated intervals indicating groundwater contact with material. Also provides information for the estimation of potential volumes of contaminated material.
Topography/geomorphology	Evaluation of potential migration pathways from on-site source areas. (e.g., drainage patterns, drainage areas, pooling areas, etc.)
Aerial and satellite imagery	Allows remote identification of possible surface water pooling areas and flow regimes, vegetation changes, and optimization of sample placement planning and access to sample locations.

11.4 Study Boundaries

The objective of this step is to define the temporal and spatial boundaries of the investigation and to identify its sampling units (SUs) and decision units (DUs). Figure 7 provides a map of the sampling area with the horizontal boundaries of the study identified.

11.4.1 Temporal Boundaries

A FS has already been drafted for the Site by the PRP Group and FWS intends for the data generated under this study to supplement the RI and the Draft FS. Therefore, this study must be conducted under an accelerated timeframe. The Draft report for this study is scheduled for delivery in Spring 2021 and field work would be difficult during the winter months after the

ground freezes. Sufficient time must be available to conduct data validation and evaluations after the field work is complete. Therefore, the available timeframe to conduct the field work will be fall of 2020.

11.4.2 Spatial Boundaries

This section describes the spatial boundaries of the area that will be sampled.

The areal boundaries of the study area are illustrated on Figure 7 and were defined to investigate the Refuge portions of the Site where landfill waste is found in the subsurface and where data gaps exist. This includes both on the Refuge portion of the Site and on the adjacent Refuge property as the CSM indicates that Site-related contaminants may have migrated into the wetlands laying adjacent to the Landfill boundaries. The study's areal extent also incorporates the need to more clearly evaluate the potential for upgradient sources of contaminants to sediment and surface water. The total study area encompasses approximately 179 acres lying entirely within the Refuge boundary. Approximately 35 acres of the study area are covered with landfill material extending to depths of up to 16 ft bgs.

To remain consistent with previous studies (specifically the Site's BERA [Integral 2016]) the vertical boundary of soils classified as surface soils within the study area is defined as soils from the ground's surface to 0.5 ft bgs.

The goals of the study require the chemical and physical characterization of the Landfill material to its full depth, which has been documented to as deep as 16 ft bgs on the Refuge portion of the site (Geosyntec 2018). Furthermore, a subset of samples from the clay layer underlying the Landfill are needed for physical, chemical, and geotechnical analyses to support potential remedial alternative development. Cross-sections illustrating the geology across the Landfill portions of the study area are included as Attachment 1.

11.4.3 Sampling Units

This section details the SUs for this study. Worksheet 19 details the sample volumes required by the analytical laboratory for each sample type. The sampling methodology for each media/sample type are described in Worksheet #18.

Surface soils: Discrete (grab) surface soil samples will be collected across the study area to a maximum depth of 6 in. The surface soil samples will be collected using a 3.25 in. diameter soil hand auger, which will yield a 49.77 cubic inches (in^3), or 28-ounce (oz) sample by volume (plus bulking). All surface soil samples will be collocated with subsurface soil and waste sampling locations.

Subsurface soil/waste samples: Sub-surface soil samples will be collected at select locations collocated with surface soil samples across the study area. Sub-surface soil samples will be

collected from up to 3 intervals using a direct-push technology (DPT) rig with a 2.25 in diameter MacroCore acetate sleeve:

- Interval 1: A composite subsurface soil sample will be collected from 0 to the total depth of the Landfill from each boring;
- Interval 2: (Optional) If evidence of elevated contamination is present at a specific interval within each boring, that interval will be discretely sampled, with a maximum one discrete sample per boring;
- Interval 3: (Optional) A maximum of 10 discrete subsurface soil samples will be collected from the native soil below the Landfill wastes and above the upper boundary of the underlying clay layer (Section 10) to determine if contaminants leaching from the Landfill material have contaminated the underlying soils.

Each sample interval will be homogenized with the exception of VOCs, and a subsample will be sent to the analytical laboratory for analyses.

Sediment Samples: Discrete sediment samples will be collected at select locations collocated with surface water samples across the study area.

Sediment Pore Water Samples: Discrete pore water samples will be collected at select locations collocated with sediment samples and surface water samples.

Surface Water Samples: Discrete surface water samples will be collected at select locations collocated with sediment samples.

11.4.4 Decision Units

DUs are the smallest user-defined areas for which a decision will be made based on sampling. As the study's goal is to determine the nature and extent of contamination across the site and not to make decisions regarding specific areas within the study boundary, the DU for this study is the entire study area (Figure 2).

11.5 Analytic Approach

The purpose of this section is to define the analytic or evaluation approach that will be used to answer the principal study questions.

11.5.1 Decision Rules

The data generated under this study are meant to supplement the Site characterization data collected to date in order to address data gaps identified by FWS (Section 10.7.9), and to provide the data necessary for remedial design relative to a proposed remedy that would remove all

landfill wastes from the Refuge portion of the Site. Therefore, the supplemental data will be combined (where comparable) with previously collected characterization data and evaluated as a whole for each media type. Initially, combined datasets will be evaluated relative to the CSM and nature and extent conclusions presented in the RI/FS documents. If the new data contradict the conclusions presented in the RI/FS, the new information will be presented to the stakeholders so that the RI/FS conclusions can be revised accordingly.

Secondly, new EPCs will be calculated from the combined datasets and compared to the risk benchmarks already developed for the Site and with existing, applicable screening levels from the DEP Ecological Screening Criteria (ESC; see Section 15 of this document for risk benchmarks).

A subset of the data to be collected is meant to inform the new remedial alternative development, but there are no applicable decision rules for these data. They will be evaluated as usable and complete — or not complete — for the purposes of remedial design.

11.5.2 Sample Analytical Parameters and Methods

The analytical parameters for each data type identified in Section 11.3 are presented in:

- Section 17 - Project Sample Design and Rationale
- Section 18 - Sample Locations, and Methods/ SOP Requirements
- Section 20 - Field Quality Control Sample Summary; and
- Section 23 - Analytical SOP References.

11.6 Performance and Acceptance Criteria

The purpose of this step is to establish the criteria needed to maximize the ability of the investigation to obtain the data needed to answer the principal investigation question accurately and with confidence.

There are two types of decision errors: sampling design errors and measurement errors. Sampling design errors are a function of the selection of sample locations or analytical methods used to characterize the site. Measurement errors are a function of the procedures used to collect and analyze the samples. The possible decision errors are presented below:

- Concluding that a contaminant is present in an environmental media when it is not actually present. This error results in investigating or cleaning up a non-impacted site.
- Concluding that a contaminant is not present when it is actually present. This error results in incomplete characterization or incorrectly concluding that a response actions is unnecessary at an impacted site.

The following will reduce the uncertainty associated with these errors:

- The sampling design will be based on historical and current site reconnaissance, previous investigations, and a well-developed CSM for the Site.
- Procedures for all field and reporting activities will follow USEPA-approved SOPs (Appendix B).
- At a minimum, the analytical laboratories will be certified under the State of New Jersey Environmental Laboratory Certification Program.
- All definitive data will be compared to the measurement performance criteria specified in Worksheet #12 to determine acceptability of analytical laboratory results.
- QA/QC procedures will be applied throughout the study process as detailed in the following Worksheets.

11.7 Design for Obtaining Data

Worksheet #17 presents the detailed sampling design and rationale for each location. Worksheets #19, 20, 24-28, and 30 specify analysis design requirements.

11.8 Assessments and Audits

Worksheet #31 presents a summary table as well as a detailed description of the assessment/audit tasks. Worksheet #33 lists the QA Management reports to be completed during the RI.

11.9 Data Review and Verification

Worksheets #34 and 35 specifies data verification process for Step I, and Step IIa and IIb, respectively. Worksheet #36 presents a cumulative analytical data validation summary.

11.10 Data Management

Worksheet #14 describes the data management tasks associated with this study.

12. QAPP WORKSHEET #12 - MEASUREMENT PERFORMANCE CRITERIA

Measurement Performance Criteria are defined in this worksheet to provide a data set that will be technically defensible for project decisions. The criteria are related to the Data Quality Indicators (DQI) of precision, accuracy/bias, representativeness, comparability, completeness, and sensitivity. The criteria for each matrix and analytical group are consolidated from the Department of Defense (DoD) Quality Systems Manual (QSM) v5.0 and the analytical methods, where applicable.

The following parameters will be used to measure outliers associated with project results.

12.1 Precision

For each field duplicate and laboratory duplicate pair (including laboratory control sample [LCS]/ laboratory control sample duplicate [LCSD] and matrix spike [MS]/ matrix spike duplicate [MSD]), the relative percent difference (RPD) will be calculated for each analyte whose original and duplicate values are both greater than or equal to the limit of quantitation (LOQ). The RPDs will be checked against the measurement performance criteria presented on Worksheet #12. The RPDs exceeding criteria will be identified in the Data Gaps Report. Additionally, the RPD of each analyte will be averaged across all duplicate pairs whose original and duplicate values are both greater than or equal to the LOQ, and the combined overall average RPD for each analysis will be calculated for the laboratory duplicates. Any conclusions about the precision of the analyses will be drawn and any limitations on the use of the data will be described in the Data Gaps Report.

Precision is most often expressed in terms of RPD:

$$RPD = 100 \times \left(\frac{C_R - C_D}{[C_R + C_D]/_2} \right)$$

Where:

RPD = Relative Percent Difference;
CR = Measured concentration of the Result; and
CD = Measured concentration of the Duplicate Result.

12.2 Accuracy/Bias Contamination

Results for all laboratory method blanks and equipment blanks will be reviewed by the data validator. In addition, LCS/LCSDs, MS/MSDs, surrogates, post-digestion spikes, and serial dilutions will be reviewed. The results for each analyte will be checked against the measurement performance criteria presented on Worksheet #12. Results for analytes that exceed criteria will

be identified in the data validation report. A discussion will follow summarizing the results of the laboratory accuracy/bias. Any conclusions about the accuracy/bias of the analyses based on contamination or outlying recoveries will be drawn and any limitations on the use of the data will be described in the Data Gaps Report.

Bias values are commonly expressed as percent recovery, which is calculated as follows:

$$\%R = \frac{C_S - C_R}{C_{Sknown}} \times 100$$

Where:
%R = Percent Recovery;
CR = Measured concentration of the Result;
CS = Measured concentration of the Spiked Result; and
CSknown = Known concentration of Spike sample.

12.3 Representativeness

Representativeness is the measure of how accurately and precisely data represents a characteristic of a population. Field and laboratory techniques must follow appropriate premixing/homogenization procedures to ensure that all sub-samples taken from a given sample or sampling point are representative of the sample as a whole. Samples requiring volatile analysis shall not undergo any premixing or homogenization. Representativeness will be assessed by a review of the precision obtained from the field and laboratory duplicate samples. Representativeness is also assessed through proper sample handling techniques and the use of equipment, material, trip, and method blanks. Existing project data may be employed to assess the representativeness of a population by defining the continuity of data from point to point.

12.4 Comparability

Sample data shall be comparable for similar samples and sample conditions. This goal is achieved using standard techniques to collect representative samples, consistent application of analytical method protocols and reporting analytical results with appropriate units.

12.5 Completeness

A completeness check will be done on all of the data generated by the laboratory. Completeness criteria are presented on Worksheet #12. For each analyte, completeness will be calculated as the number of data points for each analyte that meets the measurement performance criteria for precision, accuracy/bias, and sensitivity, divided by the total number of data points for each analyte. Analytical results qualified as rejected during data validation do not meet the measure performance criteria. A discussion summarizing the calculation of data completeness will be

included in the Data Gaps Report. Any conclusions about the completeness of the data for each analyte will be drawn and any limitations on the use of the data will be described.

12.6 Sensitivity

Results for all LCS/LCSDs will be reviewed by the data validator for each analysis. The results for each analyte will be checked against the measurement performance criteria presented in Tables 12-1 to 12-6 and cross-checked against the limits of detection (LODs) presented on Worksheet #15. Results for analytes that exceed criteria will be identified in the data validation report. A discussion will follow summarizing the results of the laboratory sensitivity. Any conclusions about the sensitivity of the analyses will be drawn and any limitations on the use of the data will be described in the Data Gaps Report.

In addition to target analytes, field samples may be analyzed for additional parameters to evaluate the presence of fate and transport and/or geochemical conditions of the subsurface at the site. These data are collected as screening-level data and as such, these data are not validated unless further evaluation is warranted (e.g., review of anomalous data). The following parameters are used for site evaluation purposes only:

- pH, turbidity, dissolved oxygen (DO), specific conductivity;
- HACH test kits will be used to detect concentrations of arsenic, manganese, lead, and iron;
- Alkalinity;
- Hardness;
- Total organic carbon (TOC); and
- Dissolved organic carbon (DOC).

Measurement Performance Criteria for each matrix and analytical group are compiled in the tables shown below. For the site evaluation parameters, full measurement performance criteria are included; however, depending on the site-specific goals, full validation may not be performed on those parameters.

Measurement Performance Criteria for each matrix and analytical group are compiled in Appendix D and in Table 12 .1 through 12.7 below.

Table 12-1. Measurement Performance Criteria Table for VOCs

Matrix	Water and Soil
Analytical Group	VOCs
Analytical Method/ SOP Reference[1]	SW-846 8260D

DQI	QC Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	Frequency of QC Check	QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S+A)
Bias/Contamination and Representativeness	Method Blank	No analytes detected > 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	Daily and/or 1 per batch of 20 samples	A
Accuracy/Bias	LCS	See Appendix E	Daily and/or 1 per batch of 20 samples	A
Accuracy/Bias	MS/MSD	See Appendix E	1 per batch of 20 samples if client designated	S+A
Accuracy/Bias	IS	Retention times ± 30 seconds from retention time of midpoint standard in the ICAL; and EICP area within -50 to + 100% of ICAL midpoint standard	Every sample	A
Accuracy/Bias	Surrogates	See Appendix E	Every sample	A
Precision	LCS/LCSD, MS/MSD, and/or field duplicates	See Appendix E	When analyzed, all parent and duplicate samples	S+A
Bias/Contamination and Representativeness	Trip Blank	No analytes detected > 1/2 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.	1 per cooler used to ship VOC samples	S
Bias/Contamination and Representativeness	Equipment Blank	No analytes detected > 1/2 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.	1 per site/sampling event for samples collected using non-dedicated sampling equipment	S
Sensitivity	LOD Determination and Verification	Laboratory establishes the LOD by spiking a matrix at 2-4x the DL.	Initial establishment and quarterly verification	A
Sensitivity	LOQ Determination and Verification	Laboratory sets the LOQ within the calibration range of the instrument. The LOQ must be greater than the LOD.	Initial establishment and quarterly verification	A
Completeness	Data completeness check	≥ 95%	After validation is complete	S+A
Accuracy	ICAL	See Worksheet #24	See Worksheet #24	A
Accuracy	ICV	See Worksheet #24	See Worksheet #24	A
Accuracy	CCV	See Worksheet #24	See Worksheet #24	A
Accuracy	BFB Tune	See Worksheet #24	See Worksheet #24	A

Table 12-2. Measurement Performance Criteria Table for SVOCs

Matrix	Water and Soil
Analytical Group	SVOCs
Analytical Method/ SOP Reference[1]	SW-846 8270E

DQI	QC Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	Frequency of QC Check	QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S+A)
Bias/Contamination and Representativeness	Method Blank	No analytes detected > 1/2 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.	Daily and/or 1 per batch of 20 samples	A
Accuracy/Bias	LCS	See Appendix E	Daily and/or 1 per batch of 20 samples	A
Accuracy/Bias	MS/MSD	See Appendix E	1 per batch of 20 samples if client designated	S+A
Accuracy/Bias	IS	Retention times ± 30 seconds from retention time of midpoint standard in the ICAL; and EICP area within -50 to + 100% of ICAL midpoint standard	Every sample	A
Accuracy/Bias	Surrogates	See Appendix E	Every sample	A
Precision	LCS/LCSD, MS/MSD, and/or field duplicates	See Appendix E	When analyzed, all parent and duplicate samples	S+A
Bias/Contamination and Representativeness	Equipment Blank	No analytes detected > 1/2 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.	1 per day for soil samples; none for samples collected using dedicated or disposable sampling equipment	S
Sensitivity	LOD Determination and Verification	Laboratory establishes the LOD by spiking a matrix at 2-4x the DL.	Initial establishment and quarterly verification	A
Sensitivity	LOQ Determination and Verification	Laboratory sets the LOQ within the calibration range of the instrument. The LOQ must be greater than the DL.	Initial establishment and quarterly verification	A
Completeness	Data completeness check	≥ 95%	After validation is complete	S+A
Accuracy	ICAL	See Worksheet #24	See Worksheet #24	A
Accuracy	ICV	See Worksheet #24	See Worksheet #24	A
Accuracy	CCV	See Worksheet #24	See Worksheet #24	A
Accuracy	DFTPP Tune	See Worksheet #24	See Worksheet #24	A

Table 12-3. Measurement Performance Criteria Table for Metals

Matrix	Water and Soil
Analytical Group	Metals
Analytical Method/ SOP Reference[1]	SW-846 6020B

DQI	QC Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	Frequency of QC Check	QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S+A)
Bias/Contamination and Representativeness	Method Blank	No analytes detected > 1/2 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.	Daily and/or 1 per batch of 20 samples	A
Accuracy/Bias	LCS	See Appendix E	Daily and/or 1 per batch of 20 samples	A
Accuracy/Bias	MS/MSD	See Appendix E	1 per batch of 20 samples if client designated	S+A
Accuracy/Bias	Serial Dilution	5x dilution must agree within 20% of the original result	1 per batch of 20 samples	A
Bias	Post Digestion Spike Addition	Recovery limits 75-125%	When dilution test fails or analyte concentration is <50x RL	S+A
Precision	LCS/LCSD, MS/MSD, and/or field duplicates	RPD ≤ 20%	When analyzed, all parent and duplicate samples	S+A
Bias/Contamination and Representativeness	Equipment Blank	No analytes detected > 1/2 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.	1 per site/sampling event for samples collected using non-dedicated sampling equipment	S
Sensitivity	MDL Determination and Verification	Laboratory establishes the MDL by spiking a matrix at 2-4x the DL.	Initial establishment and quarterly verification	A
Sensitivity	RL Determination and Verification	Laboratory sets the RL within the calibration range of the instrument. The RL must be greater than the MDL.	Initial establishment and quarterly verification	A
Completeness	Data completeness check	≥ 95%	After validation is complete	S+A
Accuracy	ICAL	If more than one standard is used, r ≥ 0.995	Daily	A
Accuracy	ICV	90-110%	After ICAL, prior to beginning a sample run	A
Accuracy	CCV	90-110%	After every 10 sample injections and at the end of the run	A
Accuracy	ICSA	Absolute value of the true concentration < RL.	At beginning of analytical run	A
Accuracy	ICSAB	± 20% of true concentration	At beginning of analytical run	A
Accuracy	Low Level Calibration Check	± 20% of true concentration	Daily, after one-point ICAL	A

Table 12-4. Measurement Performance Criteria Table for Mercury

Matrix	Water and Soil
Analytical Group	Mercury
Analytical Method/ SOP Reference[1]	SW-846 7470A / 7471B

DQI	QC Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	Frequency of QC Check	QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S+A)
Bias/Contamination and Representativeness	Method Blank	< RL; < 5% of the regulatory limit; or <5% of the measured concentration in the sample	Daily and/or 1 per batch of 20 samples	A
Accuracy/Bias	LCS	See Appendix E	Daily and/or 1 per batch of 20 samples	A
Accuracy/Bias	MS/MSD	See Appendix E	1 per batch of 20 samples if client designated	S+A
Accuracy/Bias	Serial Dilution	5x dilution must agree within 10% of the original result	1 per batch of 20 samples	A
Bias	Recovery test	Recovery limits 85-115%	When dilution test fails or analyte concentration is <25x LOD	S+A
Precision	LCS/LCSD, MS/MSD, and/or field duplicates	See Appendix E	When analyzed, all parent and duplicate samples	S+A
Bias/Contamination and Representativeness	Equipment Blank	No analytes detected > 1/2 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.	1 per site/sampling event for samples collected using non-dedicated sampling equipment	S
Sensitivity	LOD Determination and Verification	Laboratory establishes the LOD by spiking a matrix at 1-4x (multi-analyte standard) or 2-3x (single-analyte standard) the DL.	Initial establishment and quarterly verification	A
Sensitivity	LOQ Determination and Verification	Laboratory sets the LOQ within the calibration range of the instrument. The LOQ must be greater than the LOD.	Initial establishment and quarterly verification	A
Completeness	Data completeness check	≥ 95%	After validation is complete	S+A
Accuracy	ICAL	Minimum 3 standards and a calibration blank, r ≥ 0.995	Daily	A
Accuracy	ICV	90-110%	After ICAL, prior to beginning a sample run	A
Accuracy	CCV	90-110%	After every 10 sample injections and at the end of the run	A

Table 12-5. Measurement Performance Criteria Table for Pesticides

Matrix	Water and Soil
Analytical Group	Pesticides
Analytical Method/ SOP Reference[1]	SW-846 8081B

DQI	QC Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	Frequency of QC Check	QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S+A)
Bias/Contamination and Representativeness	Method Blank	No analytes detected > RL	Daily and/or 1 per batch of 20 samples	A
Accuracy/Bias	LCS	See Appendix E	Daily and/or 1 per batch of 20 samples	A
Accuracy/Bias	MS/MSD	See Appendix E	1 per batch of 20 samples if client designated	S+A
Accuracy/Bias	Confirmation of positive results	RPD <= 40% between primary and secondary column	Every positive result	A
Accuracy/Bias	Surrogates	See Appendix E	Every sample	A
Precision	LCS/LCSD, MS/MSD, and/or field duplicates	See Appendix E	When analyzed, all parent and duplicate samples	S+A
Bias/Contamination and Representativeness	Equipment Blank	No analytes detected > 1/2 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.	1 per site/sampling event for samples collected using non-dedicated sampling equipment	S
Sensitivity	LOD Determination and Verification	Laboratory establishes the LOD by spiking a matrix at 2-4x the DL.	Initial establishment and quarterly verification	A
Sensitivity	LOQ Determination and Verification	Laboratory sets the LOQ within the calibration range of the instrument. The LOQ must be greater than the LOD.	Initial establishment and quarterly verification	A
Completeness	Data completeness check	≥ 95%	After validation is complete	S+A
Accuracy	ICAL	See Worksheet #24	See Worksheet #24	A
Accuracy	ICV	+- 20%	After every ICAL	A
Accuracy	CCV	+- 20%	Daily, Every 12 hours	A

Table 12-6. Measurement Performance Criteria Table for PCBs

Matrix	Water and Soil
Analytical Group	PCBs
Analytical Method/ SOP Reference[1]	SW-846 8082A

DQI	QC Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	Frequency of QC Check	QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S+A)
Bias/Contamination and Representativeness	Method Blank	No analytes detected > RL	Daily and/or 1 per batch of 20 samples	A
Accuracy/Bias	LCS	See Appendix E	Daily and/or 1 per batch of 20 samples	A
Accuracy/Bias	MS/MSD	See Appendix E	1 per batch of 20 samples if client designated	S+A
Accuracy/Bias	Confirmation of positive results	RPD <= 40% between primary and secondary column	Every positive result	A
Accuracy/Bias	Surrogates	See Appendix E	Every sample	A
Precision	LCS/LCSD, MS/MSD, and/or field duplicates	See Appendix E	When analyzed, all parent and duplicate samples	S+A
Bias/Contamination and Representativeness	Equipment Blank	No analytes detected > 1/2 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.	1 per site/sampling event for samples collected using non-dedicated sampling equipment	S
Sensitivity	LOD Determination and Verification	Laboratory establishes the LOD by spiking a matrix at 2-4x the DL.	Initial establishment and quarterly verification	A
Sensitivity	LOQ Determination and Verification	Laboratory sets the LOQ within the calibration range of the instrument. The LOQ must be greater than the LOD.	Initial establishment and quarterly verification	A
Completeness	Data completeness check	≥ 95%	After validation is complete	S+A
Accuracy	ICAL	See Worksheet #24	See Worksheet #24	A
Accuracy	ICV	+- 20%	After every ICAL	A
Accuracy	CCV	+- 20%	Daily, Every 12 hours	A

Table 12.7. Measurement Performance Criteria Table for Dioxins/Furans

Matrix	Water and Soil
Analytical Group	Dioxins/Furans
Analytical Method/ SOP Reference[1]	SW-846 8290A

DQI	QC Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	Frequency of QC Check	QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S+A)
Bias/Contamination and Representativeness	Method Blank	No analytes detected > 1/2 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	Daily and/or 1 per batch of 20 samples	A
Accuracy/Bias	LCS	See Appendix E	Daily and/or 1 per batch of 20 samples	A
Accuracy/Bias	MS/MSD	See Appendix E	1 per batch of 20 samples if client designated	S+A
Accuracy/Bias	Confirmation of positive results	RPD <= 40% between primary and secondary column	Every positive result	A
Accuracy/Bias	Surrogates	See Appendix E	Every sample	A
Precision	LCS/LCSD, MS/MSD, and/or field duplicates	See Appendix E	When analyzed, all parent and duplicate samples	S+A
Bias/Contamination and Representativeness	Equipment Blank	No analytes detected > 1/2 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.	1 per site/sampling event for samples collected using non-dedicated sampling equipment	S
Sensitivity	LOD Determination and Verification	Laboratory establishes the LOD by spiking a matrix at 2-4x the DL.	Initial establishment and quarterly verification	A
Sensitivity	LOQ Determination and Verification	Laboratory sets the LOQ within the calibration range of the instrument. The LOQ must be greater than the LOD.	Initial establishment and quarterly verification	A
Completeness	Data completeness check	≥ 95%	After validation is complete	S+A
Accuracy	ICAL	See Worksheet #24	See Worksheet #24	A
Accuracy	ICV	+- 20%	After every ICAL	A
Accuracy	CCV	+- 20%	Daily, Every 12 hours	A

Table 12.8. Measurement Performance Criteria Table for PFAs

Matrix	Water
Analytical Group	PFAs
Analytical Method/ SOP Reference[1]	SW-846 533

DQI	QC Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	Frequency of QC Check	QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S+A)
Bias/Contamination and Representativeness	Method Blank	No analytes detected > 1/2 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	Daily and/or 1 per batch of 20 samples	A
Accuracy/Bias	LCS	See Appendix E	Daily and/or 1 per batch of 20 samples	A
Accuracy/Bias	MS/MSD	See Appendix E	1 per batch of 20 samples if client designated	S+A
Accuracy/Bias	Confirmation of positive results	TBD	TBD	A
Accuracy/Bias	Surrogates	See Appendix E	TBD	A
Precision	LCS/LCSD, MS/MSD, and/or field duplicates	See Appendix E	TBD	S+A
Bias/Contamination and Representativeness	Equipment Blank	TBD	TBD	S
Sensitivity	LOD Determination and Verification	TBD	TBD	A
Sensitivity	LOQ Determination and Verification	TBD	TBD	A
Completeness	Data completeness check	≥ 95%	TBD	S+A
Accuracy	ICAL	See Worksheet #24	TBD	A
Accuracy	ICV	TBD	TBD	A
Accuracy	CCV	TBD	TBD	A

13. QAPP WORKSHEET #13 – SECONDARY DATA CRITERIA AND LIMITATIONS

This worksheet identifies sources of existing and historical data pertinent to project decisions. For each data source, the following considerations were evaluated: whether the data was validated or reviewed, whether the analytical methodologies or technical protocols are comparable to current data handling procedures, and if limitations on use of the data can be identified.

Table 13-1. Secondary Data Criteria and Limitations

Secondary Data	Data Source (Originating Organization, Report	Data Generator(s) (Originating Org., Data Types, Data Generation/Collection	How Data Will Be Used	Limitations on Data Use
Analytical data collected during 2018 RI	Geosyntec, 2018a. RI Report, Rolling Knolls Landfill Superfund Site, Morris County, New Jersey, January.	All soil, sediment, surface water, pore water and groundwater collected during the RI.	Data will be used to locate current site sampling locations and to determine COPCs	Data was collected using USEPA CLP methods and the data was verified for EPA decision-making.
Analytical data collected during the 2016 BERA	Integral, 2016. BERA Report, Rolling Knolls Landfill Superfund Site, Morris County, New Jersey, December.	All soil, sediment, surface water, and groundwater and biota tissue samples collected during the BERA.	Data will be used to locate current site sampling locations and to determine COPECs	Data was collected using USEPA CLP methods and the data was verified for EPA decision-making.

BERA – Baseline Ecological Risk Assessment
CLP – USEPA Contract Laboratory Program
COPC – Chemicals of Potential Concern
COPEC – Chemicals of Potential Ecological Concern
RI – Remedial Investigation
USEPA – United States Environmental Protection Agency

14. QAPP WORKSHEET #14 -SUMMARY OF PROJECT TASKS

14.1 PROJECT TASKS

The implementation of the Data Gaps Investigation will consist of several project tasks including, but not limited to installation access and digging permits, utility clearance, investigation-derived waste (IDW) staging, management and disposal, surveying, photoionization detector (PID) monitoring, soil sampling, groundwater sampling, data management, and document and record keeping. SOPs (Appendix B) have been developed to cover all aspects of field operations, environmental sampling, field measurements, and record keeping. SOPs are included in Appendix B.

14.2 MOBILIZATION TASKS

Mobilization tasks will occur prior to initiating sampling tasks.

14.2.1 Installation Access and Digging Permits

Installation access permits will be completed prior to the field team accessing the Refuge. This includes preparing a "Contractor Request Letter for Access to the Refuge" provided to the Refuge Environmental Manager or designee and following any installation-specific instructions. Digging permits will be completed prior to any digging or intrusive work in accordance with the procedures outlined in the Utility Location SOP (Appendix B).

14.2.2 Utility Clearance

As required by the digging permit, prior to any intrusive digging, the location of all utilities will be verified within 10 ft of the excavation/drilling location. Each excavation location will be screened for utilities using geophysical techniques, such as ground penetrating radar. If any indication is present that utilities may be present, either the boring location will be moved to an area that is definitively cleared, or the area will be cleared by hand auguring or water excavation techniques to below 3 ft bgs.

14.3 Investigation-Derived Waste

Prior to intrusive activities, a staging area for solid and liquid waste will be determined, which will allow storage of up to 25, 55-gal drums of soil and liquid waste until each drum can be categorized for disposal purposes. Drums will include soil cuttings, and water from decontamination activities. All drums will be labeled to identify locations and contents, as well as the contract and TO as described below and in Appendix B. All drums and contents will be removed within the appropriate timeframe once the contents have been categorized for disposal. SOPs for handling and sampling IDW are provided in Appendix B.

14.3.1 Investigation-Derived Waste Sampling

Soil cuttings, and rinse water generated during soil and groundwater sampling will be sampled and analyzed in accordance with local landfill requirements.

If wastes are determined to require off-site disposal, once the manifest(s) and other appropriate documentation is signed by a Refuge representative, AI will coordinate the pickup and provide the transporter with the relevant portions of the manifest on behalf of the Refuge. AI will provide the Refuge Environmental Manager or designee with the original generator copy of the waste manifest subsequent to release of the shipment. The treatment/disposal facility-signed generator copy should be returned directly to AI PM by the treatment/disposal facility within 30 days of shipment. The AI PM will forward the required copy to the Refuge and, on behalf of the Refuge, to the appropriate agency contacts.

14.4 SAMPLING TASKS

The locations of surface soil samples, soil borings, pore-water samples, surface water samples, and sediment samples are described in Worksheet #17. The samples to be collected are outlined in Worksheet #18 and the sampling requirements for each type of analysis (e.g., bottle ware, preservation, and holding time) are listed in Worksheet #19. Each sampling task is described in detail in the SOPs in Appendix B.

14.5 ANALYSIS TASKS

All soil and groundwater samples will be analyzed by Eurofins/TestAmerica (ETA). Chemical analyses will be performed in accordance with this UFP-QAPP, and the analytical methods. The laboratory will meet the detection limits specified in Worksheet #15.

14.6 QUALITY CONTROL SAMPLES

The following field QC samples will be collected for all methods and matrices: field duplicates and MS/MSDs. Field duplicates will be collected from areas known or suspected to be contaminated. Triple sample volume will be collected for MS/MSDs from relatively clean sampling locations (e.g., upgradient) to capture effects of the matrix sampled.

When appropriate, trip blank and equipment blanks will be collected. Trip blanks, prepared by the laboratory using water demonstrated to be free of COPCs, will be placed in the cooler used to ship volatile samples (e.g., VOCs). One equipment blank will be collected per day at each site during soil and pore-water sampling.

Worksheet #18 specifies the number and type of field QC sample as well as the frequency of collection.

14.7 SECONDARY DATA

Secondary data summarized in Worksheet #13 will be reviewed and evaluated for project use. Where appropriate, secondary data will be used to plan sample locations. SOPs for secondary data activities are provided in Appendix B.

Sample Location Survey Data

Sampling locations will be surveyed using a global positioning system (GPS) with an accuracy and precision of ± 6 ft.

14.8 DATA MANAGEMENT TASKS

Field forms will be electronically generated and reviewed by the Field Team Leader prior to sample shipment. The sample handling and custody requirements, including field logs, sample collection paperwork, sample labels, and custody seals as described in Worksheets #26 and #27 will be followed.

The Project Chemist will track the samples during analysis and through data validation. All final laboratory data will be submitted in a format with Contract Laboratory Program-like deliverables. Data validation will be performed by the data validator in accordance with the procedures described in Worksheets #35 and #36. The data validator will review all definitive analytical data and will note any validation findings in data validation reports. Data validation reports will be submitted as an appendix of the RI Report. Validation qualifiers will be entered into a project database compatible spreadsheet. A 100 percent QC check will be performed by the Project Data Manager (or designee) to ensure accuracy of all hand-entered data (e.g., validation flags added by the data validation subcontractor). Sample location, field measurements and laboratory analytical data will be uploaded into project database after validation.

14.9 ASSESSMENTS AND AUDITS

Worksheet #31 presents a summary table as well as a detailed description of the assessment/audits tasks. Worksheet #33 lists the QA Management reports to be completed during the RI.

14.10 DATA REVIEW AND VERIFICATION

Worksheets #34 and #35 specifies data verification process for Step I, and Step IIa and IIb, respectively. Worksheet #36 presents a cumulative analytical data validation summary.

14.11 DOCUMENTATION AND RECORDS

Information regarding field tasks will be recorded on site field logs in accordance with SOPs AI-W-RK-01, AI-W-RK-02, and AI-W-RK-03. Sample collection information will be recorded on individual sample field forms. Any changes that are made to the field logs or the field forms will be initialed and dated. Documents will be maintained in project files and will be submitted as an

appendix to the RI Report. COC and air bills will also be completed for each sampling event. SOPs for general recordkeeping and logbook are provided in Appendix B. Field Forms are provided in Appendix C.

15. QAPP WORKSHEET #15 - REFERENCE LIMITS AND EVALUATION

This worksheet provides the target analytes, the Project Action Limits, the laboratory's Reporting Limit and Detection Limit values, where applicable. Where available, investigation results will be compared to the Ecological Screening Values (ESVs) developed in the current National Park Service guidance document, Protocol for the Selection and Use of ESVs for Non-Radiological Analytes, 2018, the NJDEP ESC https://www.nj.gov/dep/srp/guidance/ecoscreening/, and EPA Equilibrium Partitioning Sediment Benchmarks for the Protection of Benthic Organisms. Because the calculation of ESVs and ESCs do not account for current technology capabilities, a few target analytes will not meet the Project Action Limits by commercial environmental laboratories. These standards are shown in the table as highlighted yellow.

Table 15-1
Reference Limits and Evaluation Table for Soil and Sediments

Category	Analyte	CASRN	Birds and Mammals SLERA COPEC Selection ESV[a] (mg/kg)	Soil Refined SLERA ESVs[b]		Plants and Invertebrates SLERA COPEC Selection ESV (mg/kg) [c]	Soil Refined SLERA ESVs [b]		Soil NJDEP ESC	Sediment SLERA ESV[c] (mg/kg)	Sediment Refined SLERA ESVs[b]		Eurofins/TestAmerica SW 846 Soil Limits		
				Mammals (mg/kg)	Birds (mg/kg)		Terrestrial Plant (mg/kg)	Soil Invertebrate (mg/kg)	Ecological (mg/kg)		Threshold Effects Based ESV (mg/kg)	Probable Effects Based ESV (mg/kg)	Reporting Limit	Detection Limit	Unit
Metals (EPA Method 6020B/7471A)	Aluminum	7429-90-5	[d]	[d]	[d]	50	[d]	[d]	50	25,000	25,000	58,000	20	2.6	mg/Kg
	Antimony	7440-36-0	0.248	0.27	No ESV	5.0	5.0	78	0.27	No ESV	No ESV	No ESV	1	0.146	mg/Kg
	Arsenic	7440-38-2	0.25	46	43	6.8	18	60	9.9	9.7	9.8	33	1	0.1	mg/Kg
	Barium	7440-39-3	17.2	2,000	720	110	500	330	283	150	150	300	2	0.145	mg/Kg
	Beryllium	7440-41-7	2.42	21	No ESV	2.5	10	40	10	No ESV	No ESV	No ESV	0.4	0.057	mg/Kg
	Cadmium	7440-43-9	0.27	0.36	0.77	4.0	32	140	0.36	0.58	0.99	5.0	1	0.113	mg/Kg
	Calcium	7440-70-2	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	100	17	mg/Kg
	Chromium III	16065-83-1	0.83	34	26	No ESV	No ESV	No ESV	26	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Chromium VI	1333-82-0	12.01	130	140	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Chromium (total)	7440-47-3	23	63	23	0.34	1.0	0.40	0.4	36	43	111	2	0.174	mg/Kg
	Cobalt	7440-48-4	76	230	120	13	13	No ESV	0.14	No ESV	No ESV	No ESV	2	0.148	mg/Kg
	Copper	7440-50-8	14	49	28	50	70	80	5.4	28	32	149	2	0.22	mg/Kg
	Iron	7439-89-6	[g]	[g]	[g]	No ESV	[b]	[b]	No ESC	20,000	188,400	247,600	60	20.2	mg/Kg
	Lead	7439-92-1	0.94	56	11	50	120	1,700	0.0537	35	36	128	0.6	0.2	mg/Kg
	Magnesium	7439-95-4	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	100	10.2	mg/Kg
	Manganese	7439-96-5	322	4,000	4,300	220	220	450	220	460	631	1,185	4	0.403	mg/Kg
	Mercury, total	7439-97-6	0.013	1.7	0.013	0.050	0.30	0.10	0.3	0.18	0.18	1.1	0.017	0.004	mg/Kg
	Mercury, methyl	22967-92-6	0.00035	0.0031	0.00035	2.5	No ESV	2.5	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Molybdenum	7439-98-7	0.52	0.52	15	2.0	2.0	No ESV	2	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Nickel	7440-02-0	10	130	210	30	38	280	13.68	20	23	49	2	0.194	mg/Kg
	Potassium	7440-09-7	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	100	11.2	mg/Kg
	Selenium	7782-49-2	0.331	0.63	1.2	0.52	0.52	4.1	0.0276	0.72	0.72	2.9	1.25	0.118	mg/Kg
	Silver	7440-22-4	2.6	14	4.2	2.0	560	No ESV	2	0.50	0.50	5.0	1	0.089	mg/Kg
	Sodium	7440-23-5	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	100	15.6	mg/Kg
	Thallium	7440-28-0	0.027	0.42	4.5	0.050	1.0	No ESV	1	No ESV	No ESV	No ESV	0.4	0.041	mg/Kg
	Vanadium	7440-62-2	0.714	280	7.8	2.0	2.0	No ESV	2	No ESV	No ESV	No ESV	2	0.206	mg/Kg
	Zinc	7440-66-6	12	79	46	6.6	160	120	6.62	98	121	459	8	2.29	mg/Kg

Table 15-1
Reference Limits and Evaluation Table for Soil and Sediments

Category	Analyte	CASRN	Soil Refined SLERA ESVs[b]			Soil Refined SLERA ESVs [b]		Soil NJDEP ESC	Sediment Refined SLERA ESVs[b]			Eurofins/TestAmerica SW 846 Soil Limits		
			Birds and Mammals SLERA COPEC Selection ESV[a] (mg/kg)	Mammals (mg/kg)	Birds (mg/kg)	Plants and Invertebrates SLERA COPEC Selection ESV (mg/kg) [c]	Terrestrial Plant (mg/kg)	Soil Invertebrate (mg/kg)	Ecological (mg/kg)	Sediment SLERA ESV[c] (mg/kg)	Threshold Effects Based ESV (mg/kg)	Probable Effects Based ESV (mg/kg)	Reporting Limit	Detection Limit	Unit
Pesticides (EPA Method 8081B)	4,4´-DDD (Dichlorodiphenyldichloroethane)	72-54-8	0.002	0.021	0.093	No ESV	No ESV	No ESV	0.758	0.0049	0.0049	0.028	0.0067	0.00114	mg/Kg
	4,4´-DDE (Dichlorodiphenyldichloroethylene)	72-55-9	0.002	0.021	0.093	No ESV	No ESV	No ESV	0.596	0.0031	0.0032	0.031	0.0067	0.00079	mg/Kg
	4,4´-DDT (Dichlorodiphenyltrichloroethane)	50-29-3	0.002	0.021	0.093	4.1	4.1	No ESV	0.0035	0.0041	0.0042	0.063	0.0067	0.00123	mg/Kg
	Aldrin	309-00-2	0.037	0.037	No ESV	0.0033	No ESV [i]	No ESV	0.00332	7.4	7.4	No ESV	0.0067	0.00101	mg/Kg
	alpha-BHC	319-84-6	0.07	59	0.46	No ESV	No ESV	No ESV	0.0994	0.027	0.027	No ESV	0.002	0.00068	mg/Kg
	beta-BHC	319-85-7	0.27	0.27	14	0.0040	No ESV [i]	No ESV	0.00398	0.0050	0.0050	0.050	0.002	0.00075	mg/Kg
	Chlordane	57-74-9	1.8	9.1	1.8	0.22	No ESV [i]	No ESV	0.224	0.0032	0.0032	0.018	#N/A	#N/A	#N/A
	Chlorobenzilate	510-15-6	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	alpha-Chlordane	5103-71-9	0.27	0.27	0.27	2.2	2.2	No ESV	No ESC	0.0032	0.0032	0.017	#N/A	#N/A	#N/A
	gamma-BHC (Lindane)	58-89-9	0.0095	0.0095	0.21	0.0050	0.10 [i]	No ESV	0.005	0.0023	0.0024	0.0050	0.002	0.00062	mg/Kg
	delta-BHC	319-86-8	0.07	0.07	0.46	No ESV	No ESV	No ESV	No ESC	0.14	0.14	No ESV	0.002	0.00041	mg/Kg
	Diallate	2303-16-4	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	0.001	0.00046	mg/Kg
	Dieldrin	60-57-1	0.0045	0.0049	0.022	10	10	No ESV	0.00238	0.0019	0.0019	0.062	0.002	0.00087	mg/Kg
	Endosulfan I	959-98-8	0.56	0.64	15	No ESV	No ESV	No ESV	No ESC	6.4E-06	6.4E-06	No ESV	0.0067	0.00102	mg/Kg
	Endosulfan II	33213-65-9	0.56	0.64	15	No ESV	No ESV	No ESV	No ESC	6.4E-06	6.4E-06	No ESV	0.0067	0.00172	mg/Kg
	Endosulfan sulfate	1031-07-8	0.56	0.64	15	No ESV	No ESV	No ESV	0.0358	6.4E-06	6.4E-06	No ESV	0.0067	0.00084	mg/Kg
	Endrin	72-20-8	0.0014	0.023	0.0014	0.0034	0.0034	No ESV	0.0101	0.0022	0.0022	0.21	0.0067	0.00096	mg/Kg
	Endrin aldehyde	7421-93-4	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	0.0105	0.0044	0.0044	No ESV	0.0067	0.00158	mg/Kg
	Endrin ketone	53494-70-5	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	0.0067	0.0013	mg/Kg
	Heptachlor	76-44-8	0.059	0.059	0.3	0.40	0.40	No ESV	0.00598	0.0024	0.0024	0.016	0.0067	0.00079	mg/Kg
	Heptachlor epoxide	1024-57-3	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	0.152	0.0025	0.0025	0.016	0.0067	0.001	mg/Kg
	Isodrin	465-73-6	No ESV	No ESV	No ESV	0.0033	No ESV [i]	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Methoxychlor	72-43-5	5.1	5.1	18	No ESV	No ESV	No ESV	0.0199	0.019	0.030	0.30	0.0067	0.00153	mg/Kg
	Toxaphene	8001-35-2	4.1	5.9	4.1	No ESV	No ESV	No ESV	0.119	0.00010	0.00010	0.0010	0.067	0.0242	mg/Kg
	trans-Chlordane	5103-74-2	2.2	2.3	2.2	2.2	2.2	No ESV	No ESC	0.0032	0.0032	0.017	#N/A	#N/A	#N/A

Table 15-1
Reference Limits and Evaluation Table for Soil and Sediments

Category	Analyte	CASRN	Soil Refined SLERA ESVs[b]			Soil Refined SLERA ESVs [b]		Soil NJDEP ESC	Sediment Refined SLERA ESVs[b]			Eurofins/TestAmerica SW 846 Soil Limits		
			Birds and Mammals SLERA COPEC Selection ESV[a] (mg/kg)	Mammals (mg/kg)	Birds (mg/kg)	Plants and Invertebrates SLERA COPEC Selection ESV (mg/kg) [c]	Terrestrial Plant (mg/kg)	Soil Invertebrate (mg/kg)	Ecological (mg/kg)	Sediment SLERA ESV[c] (mg/kg)	Threshold Effects Based ESV (mg/kg)	Probable Effects Based ESV (mg/kg)	Reporting Limit	Detection Limit	Unit
Pesticides (EPA Method 8081B)	4,4´-DDD (Dichlorodiphenyldichloroethane)	72-54-8	0.002	0.021	0.093	No ESV	No ESV	No ESV	0.758	0.0049	0.0049	0.028	0.0067	0.00114	mg/Kg
	4,4´-DDE (Dichlorodiphenyldichloroethylene)	72-55-9	0.002	0.021	0.093	No ESV	No ESV	No ESV	0.596	0.0031	0.0032	0.031	0.0067	0.00079	mg/Kg
	4,4´-DDT (Dichlorodiphenyltrichloroethane)	50-29-3	0.002	0.021	0.093	4.1	4.1	No ESV	0.0035	0.0041	0.0042	0.063	0.0067	0.00123	mg/Kg
	Aldrin	309-00-2	0.037	0.037	No ESV	0.0033	No ESV [i]	No ESV	0.00332	7.4	7.4	No ESV	0.0067	0.00101	mg/Kg
	alpha-BHC	319-84-6	0.07	59	0.46	No ESV	No ESV	No ESV	0.0994	0.027	0.027	No ESV	0.002	0.00068	mg/Kg
	beta-BHC	319-85-7	0.27	0.27	14	0.0040	No ESV [i]	No ESV	0.00398	0.0050	0.0050	0.050	0.002	0.00075	mg/Kg
	Chlordane	57-74-9	1.8	9.1	1.8	0.22	No ESV [i]	No ESV	0.224	0.0032	0.0032	0.018	#N/A	#N/A	#N/A
	Chlorobenzilate	510-15-6	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	alpha-Chlordane	5103-71-9	0.27	0.27	0.27	2.2	2.2	No ESV	No ESC	0.0032	0.0032	0.017	#N/A	#N/A	#N/A
	gamma-BHC (Lindane)	58-89-9	0.0095	0.0095	0.21	0.0050	0.10 [i]	No ESV	0.005	0.0023	0.0024	0.0050	0.002	0.00062	mg/Kg
	delta-BHC	319-86-8	0.07	0.07	0.46	No ESV	No ESV	No ESV	No ESC	0.14	0.14	No ESV	0.002	0.00041	mg/Kg
	Diallate	2303-16-4	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	0.001	0.00046	mg/Kg
	Dieldrin	60-57-1	0.0045	0.0049	0.022	10	10	No ESV	0.00238	0.0019	0.0019	0.062	0.002	0.00087	mg/Kg
	Endosulfan I	959-98-8	0.56	0.64	15	No ESV	No ESV	No ESV	No ESC	6.4E-06	6.4E-06	No ESV	0.0067	0.00102	mg/Kg
	Endosulfan II	33213-65-9	0.56	0.64	15	No ESV	No ESV	No ESV	No ESC	6.4E-06	6.4E-06	No ESV	0.0067	0.00172	mg/Kg
	Endosulfan sulfate	1031-07-8	0.56	0.64	15	No ESV	No ESV	No ESV	0.0358	6.4E-06	6.4E-06	No ESV	0.0067	0.00084	mg/Kg
	Endrin	72-20-8	0.0014	0.023	0.0014	0.0034	0.0034	No ESV	0.0101	0.0022	0.0022	0.21	0.0067	0.00096	mg/Kg
	Endrin aldehyde	7421-93-4	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	0.0105	0.0044	0.0044	No ESV	0.0067	0.00158	mg/Kg
	Endrin ketone	53494-70-5	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	0.0067	0.0013	mg/Kg
	Heptachlor	76-44-8	0.059	0.059	0.3	0.40	0.40	No ESV	0.00598	0.0024	0.0024	0.016	0.0067	0.00079	mg/Kg
	Heptachlor epoxide	1024-57-3	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	0.152	0.0025	0.0025	0.016	0.0067	0.001	mg/Kg
	Isodrin	465-73-6	No ESV	No ESV	No ESV	0.0033	No ESV [i]	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Methoxychlor	72-43-5	5.1	5.1	18	No ESV	No ESV	No ESV	0.0199	0.019	0.030	0.30	0.0067	0.00153	mg/Kg
	Toxaphene	8001-35-2	4.1	5.9	4.1	No ESV	No ESV	No ESV	0.119	0.00010	0.00010	0.0010	0.067	0.0242	mg/Kg
	trans-Chlordane	5103-74-2	2.2	2.3	2.2	2.2	2.2	No ESV	No ESC	0.0032	0.0032	0.017	#N/A	#N/A	#N/A

Table 15-1
Reference Limits and Evaluation Table for Soil and Sediments

Category	Analyte	CASRN	Birds and Mammals SLERA COPEC Selection ESV[a] (mg/kg)	Soil Refined SLERA ESVs[b]		Plants and Invertebrates SLERA COPEC Selection ESV (mg/kg) [c]	Soil Refined SLERA ESVs [b]		Soil NJDEP ESC	Sediment SLERA ESV[c] (mg/kg)	Sediment Refined SLERA ESVs[b]		Eurofins/TestAmerica SW 846 Soil Limits		
				Mammals (mg/kg)	Birds (mg/kg)		Terrestrial Plant (mg/kg)	Soil Invertebrate (mg/kg)	Ecological (mg/kg)		Threshold Effects Based ESV (mg/kg)	Probable Effects Based ESV (mg/kg)	Reporting Limit	Detection Limit	Unit
Polychlorinated Biphenyls (PCBs) (EPA Method 8082B)	Aroclor 1016	12674-11-2	1.1	1.1	No ESV	No ESV	No ESV	No ESV	No ESC	0.059	0.059	0.59	0.067	0.0089	mg/Kg
	Aroclor 1221	11104-28-2	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	0.067	0.0089	mg/Kg
	Aroclor 1232	11141-16-5	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	0.067	0.0089	mg/Kg
	Aroclor 1242	53469-21-9	0.041	0.39	0.041	No ESV	No ESV	No ESV	No ESC	0.059	0.059	0.59	0.067	0.0089	mg/Kg
	Aroclor 1248	12672-29-6	0.0073	0.0073	0.041	No ESV	No ESV	No ESV	No ESC	0.059	0.059	0.59	0.067	0.0089	mg/Kg
	Aroclor 1254	11097-69-1	0.041	0.45	0.041	160	160	No ESV	No ESC	0.059	0.059	0.34	0.067	0.0092	mg/Kg
	Aroclor 1260	11096-82-5	0.88	10	0.88	No ESV	No ESV	No ESV	No ESC	0.059	0.059	0.59	0.067	0.0092	mg/Kg
	PCBs/Total PCBs	1336-36-3	No ESV	No ESV	No ESV	40	40	No ESV	0.000332	0.032	0.060	0.68	0.067	0.0092	mg/Kg

Table 15-1
Reference Limits and Evaluation Table for Soil and Sediments

Category	Analyte	CASRN	Birds and Mammals SLERA COPEC Selection ESV[a] (mg/kg)	Soil Refined SLERA ESVs[b]		Plants and Invertebrates SLERA COPEC Selection ESV (mg/kg) [c]	Soil Refined SLERA ESVs [b]		Soil NJDEP ESC	Sediment SLERA ESV[c] (mg/kg)	Sediment Refined SLERA ESVs[b]		Eurofins/TestAmerica SW 846 Soil Limits		
				Mammals (mg/kg)	Birds (mg/kg)		Terrestrial Plant (mg/kg)	Soil Invertebrate (mg/kg)	Ecological (mg/kg)		Threshold Effects Based ESV (mg/kg)	Probable Effects Based ESV (mg/kg)	Reporting Limit	Detection Limit	Unit
Dioxin/Furans (EPA Method 8290A)	Individual dioxin/furan congeners	--	[q]	[q]	[q]	[q]	[q]	[q]	No ESC	[q]	[q]	[q]	#N/A	#N/A	#N/A
	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)	1746-01-6	0.00000029	0.00000029	0.0000116	5.0	No ESV	5.0	0.000000199	8.5E-07	8.5E-07	8.5E-06	0.000000001	1.5E-10	mg/Kg

Table 15-1
Reference Limits and Evaluation Table for Soil and Sediments

Category	Analyte	CASRN	Soil Refined SLERA ESVs[b]			Soil Refined SLERA ESVs [b]		Soil NJDEP ESC	Sediment Refined SLERA ESVs[b]			Eurofins/TestAmerica SW 846 Soil Limits		
			Birds and Mammals SLERA COPEC Selection ESV[a] (mg/kg)	Mammals (mg/kg)	Birds (mg/kg)	Plants and Invertebrates SLERA COPEC Selection ESV (mg/kg) [c]	Terrestrial Plant (mg/kg)	Soil Invertebrate (mg/kg)	Ecological (mg/kg)	Sediment SLERA ESV[c] (mg/kg)	Threshold Effects Based ESV (mg/kg)	Probable Effects Based ESV (mg/kg)	Reporting Limit	Detection Limit	Unit
Volatile Organic Compounds (VOCs) (EPA Method 8260B)	1,1,1,2-Tetrachloroethane	630-20-6	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	0.001	0.00019	mg/Kg
	1,1,1-Trichloroethane	71-55-6	260	260	No ESV	No ESV	No ESV	No ESV	29.8	0.012	0.070	0.70	0.001	0.000233	mg/Kg
	1,1,2,2-Tetrachloroethane	79-34-5	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	0.127	0.57	0.57	No ESV	0.001	0.000214	mg/Kg
	1,1,2-Trichloroethane	79-00-5	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	28.6	0.60	0.60	No ESV	0.001	0.000178	mg/Kg
	1,1-Dichloroethane	75-34-3	210	210	No ESV	No ESV	No ESV	No ESV	No ESC	0.0082	0.020	0.20	0.001	0.000206	mg/Kg
	1,1-Dichloroethene	75-35-4	11	11	No ESV	No ESV	No ESV	No ESV	8.28	0.015	0.10	1.0	0.001	0.000225	mg/Kg
	1,1-Dichloropropene	563-58-6	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	1,2,3-Trichlorobenzene	87-61-6	No ESV	No ESV	No ESV	20	No ESV	20	20	0.14	0.14	No ESV	0.001	0.000181	mg/Kg
	1,2,3-Trichloropropane	96-18-4	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	1,2,3,4-Diepoxybutene	1464-53-5	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	1,2,4-Trichlorobenzene	120-82-1	0.27	0.27	No ESV	1.2	No ESV	20	20	0.011	0.011	0.11	0.001	0.000358	mg/Kg
	1,2,4-Trimethylbenzene	95-63-6	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	1,2-Dibromo-3-chloropropane	96-12-8	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	0.001	0.00046	mg/Kg
	1,2-Dibromoethane	106-93-4	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	0.001	0.00018	mg/Kg
	1,2-Dichlorobenzene	95-50-1	0.92	0.92	No ESV	20	No ESV	20	No ESC	0.0043	0.0043	No ESV	0.001	0.000144	mg/Kg
	1,2-Dichloroethane	107-06-2	0.85	27	0.85	No ESV	No ESV	No ESV	21.2	0.017	0.017	No ESV	0.001	0.000296	mg/Kg
	1,2-Dichloroethene	540-59-0	24	24	No ESV	No ESV	No ESV	No ESV	No ESC	0.20	0.20	2.0	#N/A	#N/A	#N/A
	1,2-Dichloropropane	78-87-5	No ESV	No ESV	No ESV	700	No ESV	700	32.7	No ESV	No ESV	No ESV	0.001	0.000423	mg/Kg
	1,3,5-Trimethylbenzene	108-67-8	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	1,3-Dichloro-2-propanol	96-23-1	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	1,3-Dichlorobenzene	541-73-1	0.74	0.74	No ESV	20	No ESV	20	37.7	0.44	0.44	No ESV	0.001	0.000159	mg/Kg
	1,3-Dichloropropane	142-28-9	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	1,4-Dichlorobenzene	106-46-7	0.89	0.89	No ESV	1.2	No ESV	20	0.546	0.030	0.030	0.30	0.001	0.000225	mg/Kg
	1,4-Dioxane	123-91-1	1.83	1.83	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	0.02	0.00918	mg/Kg
	1-Chlorobutane	109-69-3	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	1-Chlorohexane	544-10-5	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	1-Propanol	71-23-8	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	2,2-Dichloropropane	594-20-7	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	2-Butanone (Methyl ethyl ketone) (MEK)	78-93-3	350	350	No ESV	No ESV	No ESV	No ESV	No ESC	0.92	0.92	No ESV	0.005	0.00271	mg/Kg
	2-Chloroethanol	107-07-3	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	2-Chloroethyl vinyl ether	110-75-8	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	2-Chlorotoluene	95-49-8	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	2-Hexanone	591-78-6	0.36	5.4	0.36	No ESV	No ESV	No ESV	No ESC	0.013	0.013	No ESV	0.005	0.00171	mg/Kg
	2-Hydroxypropionitrile	78-97-7	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	2-Nitropropane	79-46-9	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	2-Pentanone	107-87-9	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	2-Picoline	109-06-8	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	2-Propanol	67-63-0	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	3-Chloropropionitrile	542-76-7	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	4-Chlorotoluene	106-43-4	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	4-Methyl-2-pentanone (MIBK)	108-10-1	9.7	9.7	No ESV	No ESV	No ESV	No ESV	No ESC	0.032	0.032	No ESV	0.005	0.00156	mg/Kg
	Acetone	67-64-1	1.2	1.2	7.5	No ESV	No ESV	No ESV	No ESC	0.065	0.065	0.65	0.006	0.00572	mg/Kg
	Acetonitrile	75-05-8	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Acrolein (Propenal)	107-02-8	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	5.27	0.00015	0.00015	No ESV	#N/A	#N/A	#N/A

Table 15-1
Reference Limits and Evaluation Table for Soil and Sediments

Category	Analyte	CASRN	Birds and Mammals SLERA COPEC Selection ESV[a] (mg/kg)	Soil Refined SLERA ESVs[b]		Plants and Invertebrates SLERA COPEC Selection ESV (mg/kg) [c]	Soil Refined SLERA ESVs [b]		Soil NJDEP ESC	Sediment SLERA ESV[c] (mg/kg)	Sediment Refined SLERA ESVs[b]		Eurofins/TestAmerica SW 846 Soil Limits		
				Mammals (mg/kg)	Birds (mg/kg)		Terrestrial Plant (mg/kg)	Soil Invertebrate (mg/kg)	Ecological (mg/kg)		Threshold Effects Based ESV (mg/kg)	Probable Effects Based ESV (mg/kg)	Reporting Limit	Detection Limit	Unit
Volatile Organic Compounds (VOCs) (EPA Method 8260B)	Acrylonitrile	107-13-1	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	0.0239	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Allyl Alcohol	107-18-6	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Allyl chloride	107-05-1	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Benzene	71-43-2	24	24	No ESV	No ESV	No ESV	No ESV	0.255	0.010	0.010	0.10	0.001	0.000258	mg/Kg
	Benzyl chloride	100-44-7	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	beta-Propiolactone	57-57-8	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Bis(2-chloroethyl)sulfide	505-60-2	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Bromoacetone	598-31-2	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Bromobenzene	108-86-1	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Bromochloromethane	74-97-5	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	0.001	0.000281	mg/Kg
	Bromodichloromethane	75-27-4	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	0.54	No ESV	No ESV	No ESV	0.001	0.000257	mg/Kg
	Bromoform	75-25-2	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	15.9	0.28	0.28	No ESV	0.001	0.000425	mg/Kg
	Bromomethane	74-83-9	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	0.235	No ESV	No ESV	No ESV	0.001	0.000474	mg/Kg
	Carbon disulfide	75-15-0	0.81	0.81	No ESV	No ESV	No ESV	No ESV	No ESC	0.00042	0.00042	No ESV	0.001	0.000266	mg/Kg
	Carbon tetrachloride	56-23-5	58.6	58.6	No ESV	No ESV	No ESV	No ESV	2.98	0.017	0.017	No ESV	0.001	0.000387	mg/Kg
	Chloral hydrate	302-17-0	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Chloroacetonitrile	107-14-2	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Chlorobenzene	108-90-7	43	43	No ESV	2.4	No ESV	40	13.1	0.0028	0.030	0.30	0.001	0.000177	mg/Kg
	Chloroethane	75-00-3	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	0.001	0.000522	mg/Kg
	Chloroform	67-66-3	8	8	No ESV	No ESV	No ESV	No ESV	1.19	0.00072	0.00072	No ESV	0.001	0.000319	mg/Kg
	Chloromethane	74-87-3	No ESV		No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	0.001	0.000435	mg/Kg
	Chloroprene	126-99-8	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Crotonaldehyde	4170-30-3	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	cis-1,2-Dichloroethene	156-59-2	89.6	89.6	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	0.001	0.000152	mg/Kg
	cis-1,3-Dichloropropene	10061-01-5	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	2.5E-05	2.5E-05	No ESV	0.001	0.000273	mg/Kg
	cis-1,4-Dichloro-2-butene	1476-11-5	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Dibromochloromethane	124-48-1	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	2.05	No ESV	No ESV	No ESV	0.001	0.000194	mg/Kg
	Dibromofluoromethane	1868-53-7	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#VALUE!	#VALUE!	mg/Kg
	Dibromomethane	74-95-3	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Dichlorodifluoromethane	75-71-8	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	0.001	0.000338	mg/Kg
	Diethyl ether	60-29-7	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Epichlorohydrin	106-89-8	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Ethanol	64-17-5	117	117	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Ethyl acetate	14178-6	330	330	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Ethylbenzene	100-41-4	No ESV		No ESV	No ESV	No ESV	No ESV	5.16	0.026	0.026	No ESV	0.001	0.000199	mg/Kg
	Ethylene oxide	75-21-8	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Ethyl methacrylate	97-63-2	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Hexachloroethane	67-72-1	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	0.596	0.21	0.21	No ESV	0.033	0.0114	mg/Kg
	Iodomethane	74-88-4	0.038	No ESV	0.038	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Isobutyl alcohol	78-83-1	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Isopropylbenzene	98-82-8	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	0.001	0.000126	mg/Kg
	Malononitrile	109-77-3	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Methacrylonitrile	126-98-7	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Methanol	67-56-1	183.2	183.2	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Methyl acetate	79-20-9	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	0.005	0.0043	mg/Kg
	Methyl methacrylate	80-62-6	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Methyl tert-butyl ether (MTBE)	1634-04-4	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	0	No ESV	No ESV	No ESV	0.001	0.000125	mg/Kg
	Methylene chloride	75-09-2	2.6	2.6	No ESV	1,600	1,600	No ESV	4.05	0.011	0.018	0.18	0.001	0.000464	mg/Kg
	n-Butanol	71-36-3	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	n-Butylbenzene	104-51-8	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	N-Nitroso-di-n-butylamine	924-16-3	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A

Table 15-1
Reference Limits and Evaluation Table for Soil and Sediments

Category	Analyte	CASRN	Soil Refined SLERA ESVs[b]			Soil Refined SLERA ESVs [b]		Soil NJDEP ESC	Sediment Refined SLERA ESVs[b]			Eurofins/TestAmerica SW 846 Soil Limits		
			Birds and Mammals SLERA COPEC Selection ESV[a] (mg/kg)	Mammals (mg/kg)	Birds (mg/kg)	Plants and Invertebrates SLERA COPEC Selection ESV (mg/kg) [c]	Terrestrial Plant (mg/kg)	Soil Invertebrate (mg/kg)	Ecological (mg/kg)	Sediment SLERA ESV[d] (mg/kg)	Threshold Effects Based ESV (mg/kg)	Probable Effects Based ESV (mg/kg)	Reporting Limit	Detection Limit	Unit
Volatile Organic Compounds (VOCs) (EPA Method 8260B)	n-Propylamine	107-10-8	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	n-Propylbenzene	103-65-1	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	p-Isopropyltoluene	99-87-6	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Paraldehyde	123-63-7	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Pentachloroethane	76-01-7	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Pentafluorobenzene	363-72-4	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Propargyl alcohol	107-19-7	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Propionitrile (ethyl cyanide)	107-12-0	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	o-Toluidine	95-53-4	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	sec-Butylbenzene	135-98-8	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Styrene	100-42-5	No ESV	No ESV	No ESV	1.2	300	1.2	4.69	0.56	0.56	No ESV	0.001	0.000278	mg/Kg
	tert-Butylbenzene	98-06-6	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Tetrachloroethene	127-18-4	0.18	0.18	No ESV	10	10	No ESV	9.92	0.0020	0.0020	0.020	0.001	0.000143	mg/Kg
	Toluene	108-88-3	23	23	No ESV	200	200	No ESV	200	0.0036	0.010	0.10	0.001	0.000234	mg/Kg
	trans-1,2-Dichloroethene	156-60-5	89.6	89.6	No ESV	No ESV	No ESV	No ESV	0.784	0.31	0.31	No ESV	0.001	0.000246	mg/Kg
	trans-1,3-Dichloropropene	10061-02-6	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	2.5E-05	2.5E-05	No ESV	0.001	0.000266	mg/Kg
	trans-1,4-Dichloro-2-butene	110-57-6	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Trichloroethene	79-01-6	1.387	42	No ESV	No ESV	No ESV	No ESV	12.4	0.035	0.078	0.78	0.001	0.000144	mg/Kg
	Trichlorofluoromethane	75-69-4	52	52	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	0.001	0.000406	mg/Kg
	Vinyl acetate	108-05-4	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	0.00084	0.00084	No ESV	#N/A	#N/A	#N/A
	Vinyl chloride	75-01-4	0.12	0.12	No ESV	No ESV	No ESV	No ESV	0.646	No ESV	No ESV	No ESV	0.001	0.000546	mg/Kg
	Xylene, m-	108-38-3	4.162	4.162	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Xylenes, m+p-	108-38-3+p	4.162	4.162	No ESV	No ESV	No ESV	No ESV	No ESC	0.0070	0.0070	No ESV	#N/A	#N/A	#N/A
	Xylene, o-	95-47-6	4.162	4.162	No ESV	No ESV	No ESV	No ESV	No ESC	0.047	0.047	No ESV	0.001	0.000194	mg/Kg
	Xylene, p-	106-42-3	4.162	4.162	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Xylenes, Total	1330-20-7	1.4	1.4	41	10	100	No ESV	10	0.13	0.13	1.3	#N/A	#N/A	#N/A

Table 15-1
Reference Limits and Evaluation Table for Soil and Sediments

Category	Analyte	CASRN	Birds and Mammals SLERA COPEC Selection ESV[a] (mg/kg)	Soil Refined SLERA ESVs[b]		Plants and Invertebrates SLERA COPEC Selection ESV (mg/kg) [c]	Soil Refined SLERA ESVs [b]		Soil NJDEP ESC	Sediment Refined SLERA ESVs[b]			Eurofins/TestAmerica SW 846 Soil Limits		
				Mammals (mg/kg)	Birds (mg/kg)		Terrestrial Plant (mg/kg)	Soil Invertebrate (mg/kg)	Ecological (mg/kg)	Sediment SLERA ESV[c] (mg/kg)	Threshold Effects Based ESV (mg/kg)	Probable Effects Based ESV (mg/kg)	Reporting Limit	Detection Limit	Unit
Polycyclic Aromatic Hydrocarbons (PAHs) (EPA Method 8270D)	*LOW MOLECULAR WEIGHT*		*LOW MOLECULAR WEIGHT*			*LOW MOLECULAR WEIGHT*			*LOW MOLECULAR WEIGHT*				#N/A	#N/A	#N/A
	2-Methylnaphthalene	91-57-6	16	16	No ESV	No ESV	No ESV	No ESV	3.24	0.076	0.076	0.76	0.33	0.00925	mg/Kg
	Acenaphthene	83-32-9	130	130	No ESV	0.25	20	No ESV	20	0.076	0.076	0.76	0.33	0.0241	mg/Kg
	Acenaphthylene	208-96-8	120	120	No ESV	No ESV	No ESV	No ESV	682	0.076	0.076	0.76	0.33	0.00342	mg/Kg
	Anthracene	120-12-7	210	210	No ESV	6.8	6.8	No ESV	1480	0.010	0.057	0.85	0.33	0.0101	mg/Kg
	Fluoranthene	206-44-0	22	22	No ESV	10	No ESV	10	122	0.031	0.42	2.2	0.33	0.0116	mg/Kg
	Fluorene	86-73-7	250	250	No ESV	3.7	No ESV	30	122	0.010	0.077	0.54	0.33	0.00449	mg/Kg
	Naphthalene	91-20-3	3.4	9.6	3.4	1.0	1.0	No ESV	0.0994	0.014	0.18	0.56	0.33	0.00572	mg/Kg
	Phenanthrene	85-01-8	11	11	No ESV	5.5	No ESV	5.5	45.7	0.019	0.20	1.2	0.33	0.00581	mg/Kg
	Total LMW PAHs	--	100	100	No ESV	29	No ESV	29	No ESC	0.076	0.076	1.2	#N/A	#N/A	#N/A
	HIGH MOLECULAR WEIGHT		*HIGH MOLECULAR WEIGHT*			*HIGH MOLECULAR WEIGHT*			*HIGH MOLECULAR WEIGHT*						
	Benzo(a)anthracene	56-55-3	0.73	3.4	0.73	18	18	No ESV	5.21	0.015	0.11	1.1	0.033	0.0115	mg/Kg
	Benzo(g,h,i)perylene	191-24-2	25	25	No ESV	No ESV	No ESV	No ESV	119	0.016	0.016	0.25	0.33	0.00976	mg/Kg
	Benzo(a)pyrene	50-32-8	1.98	62	No ESV	No ESV	No ESV	No ESV	1.52	0.032	0.15	1.5	0.033	0.00881	mg/Kg
	Benzo(b)fluoranthene	205-99-2	44	44	No ESV	18	18	No ESV	59.8	0.19	0.19	1.9	0.033	0.00856	mg/Kg
	Benzo(k)fluoranthene	207-08-9	71	71	No ESV	No ESV	No ESV	No ESV	148	0.24	0.24	2.4	0.033	0.00649	mg/Kg
	Chrysene	218-01-9	3.1	3.1	No ESV	No ESV	No ESV	No ESV	4.73	0.026	0.17	1.3	0.33	0.00559	mg/Kg
	Indeno(1,2,3-cd)pyrene	193-39-5	71	71	No ESV	No ESV	No ESV	No ESV	109	0.017	0.017	0.24	0.033	0.0129	mg/Kg
	Pyrene	129-00-0	23	23	33	10	No ESV	10	78.5	0.044	0.20	1.5	0.33	0.00823	mg/Kg
	Total HMW PAHs	--	1.1	1.1	No ESV	18	No ESV	18	No ESC	0.19	0.19	2.3			
Semi-Volatile Organic Compounds (SVOCs) (EPA Method 8270D)	2,2'-oxybis (1-chloropropane)	108-60-1	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	19.9	No ESV	No ESV	No ESV	0.33	0.00599	mg/Kg
	2,4,5-Trichlorophenol	95-95-4	No ESV	No ESV	No ESV	4.0	4.0	9.0	No ESC	0.29	0.29	No ESV	0.33	0.0337	mg/Kg
	2,4,6-Trichlorophenol	88-06-2	No ESV	No ESV	No ESV	10	No ESV	10	4	No ESV	No ESV	No ESV	0.133	0.0425	mg/Kg
	2,4-Dichlorophenol	120-83-2	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	87.5	No ESV	No ESV	No ESV	0.133	0.0212	mg/Kg
	2,4-Dimethylphenol	105-67-9	No ESV	No ESV	No ESV	0.010	No ESV	No ESV	0.01	No ESV	No ESV	No ESV	0.33	0.0145	mg/Kg
	2,4-Dinitrophenol	51-28-5	No ESV	No ESV	No ESV	20	20	No ESV	0.0609	No ESV	No ESV	No ESV	0.266	0.163	mg/Kg
	2,4-Dinitrotoluene	121-14-2	14	14	No ESV	6.0	6.0	18	1.28	0.29	0.29	2.9	0.067	0.0356	mg/Kg
	2,6-Dinitrotoluene	606-20-2	4	4	52	30	No ESV	30	No ESC	No ESV	No ESV	No ESV	0.067	0.0239	mg/Kg
	2-Chloronaphthalene	91-58-7	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	0.33	0.0153	mg/Kg
	2-Chlorophenol	95-57-8	0.39	0.54	0.39	No ESV	No ESV	No ESV	No ESC	0.027	0.055	0.55	0.33	0.0118	mg/Kg
	2-Methylphenol	95-48-7	580	580	No ESV	0.67	0.67	No ESV	No ESC	0.012	0.012	No ESV	0.33	0.0124	mg/Kg
	2-Nitroaniline	88-74-4	5.3	5.3	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	0.33	0.0124	mg/Kg
	2-Nitrophenol	88-75-5	No ESV	No ESV	No ESV	7.0	No ESV	7.0	No ESC	No ESV	No ESV	No ESV	0.33	0.0331	mg/Kg
	3,3´-Dichlorobenzidine	91-94-1	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	0.133	0.05	mg/Kg
	3-Nitroaniline	99-09-2	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	0.33	0.0373	mg/Kg
	4,6-Dinitro-2-methylphenol	534-52-1	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	0	No ESV	No ESV	No ESV	0.266	0.0537	mg/Kg
	4-Bromophenyl phenyl ether	101-55-3	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	0.26	0.26	No ESV	0.33	0.0131	mg/Kg
	4-Chloro-3-methylphenol	59-50-7	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	0.33	0.0186	mg/Kg
	4-Chloroaniline	106-47-8	No ESV	No ESV	No ESV	1.0	1.0	1.8	No ESC	No ESV	No ESV	No ESV	0.33	0.0231	mg/Kg
	4-Chlorophenyl phenyl ether	7005-72-3	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	0.33	0.0117	mg/Kg
	4-Methylphenol	106-44-5	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	0.33	0.0207	mg/Kg
	4-Nitroaniline	100-01-6	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	0.33	0.038	mg/Kg
	4-Nitrophenol	100-02-7	No ESV	No ESV	No ESV	7.0	No ESV	7.0	5.12	No ESV	No ESV	No ESV	0.67	0.0539	mg/Kg
	bis(-2-chloroethoxy)Methane	111-91-1	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	0.33	0.0258	mg/Kg
	bis(-2-chloroethyl)Ether	111-44-4	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	23.7	No ESV	No ESV	No ESV	0.033	0.0115	mg/Kg
	bis(2-ethylhexyl)Phthalate	117-81-7	0.02	0.6	0.02	No ESV	No ESV	No ESV	0.925	453	453	No ESV	0.33	0.0175	mg/Kg
	Butylbenzylphthalate	85-68-7	90	90	No ESV	No ESV	No ESV	No ESV	0.239	0.10	0.10	1.0	0.33	0.0155	mg/Kg
	Carbazole	86-74-8	79	79	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	0.33	0.0126	mg/Kg

Table 15-1
Reference Limits and Evaluation Table for Soil and Sediments

Category	Analyte	CASRN	Birds and Mammals SLERA COPEC Selection ESV[a] (mg/kg)	Soil Refined SLERA ESVs[b]		Plants and Invertebrates SLERA COPEC Selection ESV (mg/kg) [c]	Soil Refined SLERA ESVs [b]		Soil NJDEP ESC	Sediment SLERA ESV[c] (mg/kg)	Sediment Refined SLERA ESVs[b]		Eurofins/TestAmerica SW 846 Soil Limits		
				Mammals (mg/kg)	Birds (mg/kg)		Terrestrial Plant (mg/kg)	Soil Invertebrate (mg/kg)	Ecological (mg/kg)		Threshold Effects Based ESV (mg/kg)	Probable Effects Based ESV (mg/kg)	Reporting Limit	Detection Limit	Unit
Semi-Volatile Organic Compounds (SVOCs) (EPA Method 8270D)	Dibenzo(a,h)anthracene	53-70-3	14	14	No ESV	No ESV	No ESV	No ESV	18.4	0.033	0.033	0.33	0.033	0.0143	mg/Kg
	Dibenzofuran	132-64-9	No ESV	No ESV	No ESV	6.1	6.1	No ESV	No ESC	0.30	0.51	5.1	0.33	0.00465	mg/Kg
	Di-n-butyl phthalate	84-74-2	0.011	180	0.011	160	200	No ESV	0.15	0.011	0.011	0.11	0.33	0.0584	mg/Kg
	Di-n-octyl phthalate	117-84-0	0.91	0.91	No ESV	No ESV	No ESV	No ESV	No ESC	17	17	No ESV	0.33	0.0175	mg/Kg
	Diethyl phthalate	84-66-2	3,600	3,600	No ESV	100	100	No ESV	24.8	0.60	0.60	No ESV	0.33	0.00479	mg/Kg
	Dimethyl phthalate	131-11-3	38	38	No ESV	10	No ESV	200	No ESC	No ESV	No ESV	No ESV	0.33	0.0752	mg/Kg
	Hexachlorobenzene	118-74-1	0.079	0.2	0.079	10	10	10	0.199	No ESV	No ESV	No ESV	0.033	0.0157	mg/Kg
	Hexachlorobutadiene	87-68-3	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	0.0398	0.70	0.70	No ESV	0.067	0.00704	mg/Kg
	Hexachlorocyclopentadiene	77-47-4	No ESV	No ESV	No ESV	10	10	No ESV	0.755	No ESV	No ESV	No ESV	0.33	0.029	mg/Kg
	Hexachloroethane	67-72-1	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	0.596	0.21	0.21	No ESV	0.033	0.0114	mg/Kg
	Isophorone	78-59-1	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	139	No ESV	No ESV	No ESV	0.133	0.0956	mg/Kg
	n-Nitroso-di-n-propylamine	621-64-7	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	0	No ESV	No ESV	No ESV	0.033	0.024	mg/Kg
	N-Nitrosodiphenylamine	86-30-6	No ESV	No ESV	No ESV	20	No ESV	20	0.545	0.52	0.52	No ESV	0.33	0.00633	mg/Kg
	Nitrobenzene	98-95-3	4.8	4.8	No ESV	2.2	No ESV	40	No ESC	No ESV	No ESV	No ESV	0.033	0.00794	mg/Kg
	Pentachlorophenol	87-86-5	0.36	2.8	2.1	3.0	5.0	31	0.119	0.010	0.010	0.10	0.266	0.0678	mg/Kg
	Phenol	108-95-2	37	37	No ESV	0.79	70	30	30	0.0012	0.0012	No ESV	0.33	0.0122	mg/Kg
	Pyridine	110-86-1	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A

Table 15-1
Reference Limits and Evaluation Table for Soil and Sediments

Category	Analyte	CASRN	Birds and Mammals SLERA COPEC Selection ESV[a] (mg/kg)	Soil Refined SLERA ESVs[b]		Plants and Invertebrates SLERA COPEC Selection ESV (mg/kg) [c]	Soil Refined SLERA ESVs [b]		Soil NJDEP ESC	Sediment SLERA ESV[c] (mg/kg)	Sediment Refined SLERA ESVs[b]		Eurofins/TestAmerica SW 846 Soil Limits		
				Mammals (mg/kg)	Birds (mg/kg)		Terrestrial Plant (mg/kg)	Soil Invertebrate (mg/kg)	Ecological (mg/kg)		Threshold Effects Based ESV (mg/kg)	Probable Effects Based ESV (mg/kg)	Reporting Limit	Detection Limit	Unit
Anions	Cyanide (amenable) (Methods 9010B/9013/9014)	57-12-5-A	No ESV	No ESV	No ESV	No ESV	No ESV	No ESV	No ESC	No ESV	No ESV	No ESV	#N/A	#N/A	#N/A
	Cyanide (total) (Methods 9010B/9013/9014)	57-12-5	0.098	330	0.098	No ESV	No ESV	No ESV	1.33	0.10	0.10	1.0	0.00024	0.000123	mg/Kg

Notes:
[a]Lowest ESV across all NPS-approved sources
[b]Selection hierarchy:
1 -- EcoSSL
2 -- LANL ECORISK Database
3 -- Sample et al. (1996) food-based value
[c]The lowest available ESV from approved sources should be selected as the SLERA ESL to select COPECs and be protective of all receptors
[e]Aluminum is identified as a COPEC only when soil pH is below 5.5
[h] Individual congeners should be evaluated in terms of toxicity equivalence relative to TCDD.

Eco-SSL = Ecological Soil Screening Level
ORNL = Oak Rdge National Laboratory
mg/kg = milligrams per kilogram
dw = dry weight
ESV = ecological screening value
ESL = ecological screening level
SLERA = screening level ecological risk assessment
CASRN = Chemical Abstracts Service Registry Number

0.011 Highlighted standard is lower than the laboratory method detection limit
#NA Not Analyzed
No ESV - indicates that no ESV is available from the designated source

Table 15-2
Reference Limits and Evaluation Table for Surface Water, Pore Water and Groundwater

Category	Analyte	CASRN	SLERA COPEC Selection ESV (µg/L) [1]	Refined SLERA ESVs[2]		NJDEP ESC	Eurofins SW846 Water		
				Acute ESV (µg/L)	Chronic ESV (µg/L)	Freshwater (ug/L)	Reporting Limit (µg/L)	Detection Limit (µg/L)	Units
Metals EPA Method 6020B/7471A	Aluminum	7429-90-5	5.0	750	87	0	40	8.01	ug/L
	Antimony	7440-36-0	30	180	30	80	2	0.757	ug/L
	Arsenic	7440-38-2	3.1	340	150	150	2	0.887	ug/L
	Barium	7440-39-3	3.9	110	4.0	220	4	0.913	ug/L
	Beryllium	7440-41-7	0.66	35	0.66	4	0.8	0.098	ug/L
	Cadmium	7440-43-9	0.070	[a]	[a]	0.09	2	0.156	ug/L
	Calcium	7440-70-2	116,000	No ESV	116,000	No ESC	200	22.7	ug/L
	Chromium III	16065-83-1	8.9	[a]	[a]	11	#N/A	#N/A	#N/A
	Chromium VI	1333-82-0	1.0	16	11	No ESC	#N/A	#N/A	#N/A
	Chromium, total	7440-47-3	No ESV	No ESV	No ESV	42	4	0.688	ug/L
	Cobalt	7440-48-4	3.0	1,500	23	24	4	0.263	ug/L
	Copper	7440-50-8	0.23	[b]	[b]	4	4	2.45	ug/L
	Iron	7439-89-6	158	No ESV	1,000	No ESC	120	8.52	ug/L
	Lead	7439-92-1	0.92	[a]	[a]	5.4	1.2	0.11	ug/L
	Magnesium	7439-95-4	82,000	No ESV	82,000	No ESC	200	15.4	ug/L
	Manganese	7439-96-5	112	2,300	120	0.00	8	1.11	ug/L
	Mercury, total	7439-97-6	0.026	1.4	0.77	0.77	0.2	0.091	ug/L
	Mercury, methyl	22967-92-6	0.0028	0.099	0.0028	No ESC	#N/A	#N/A	#N/A
	Molybdenum	7439-98-7	73	16,000	370	0.00E+00	#N/A	#N/A	#N/A
	Nickel	7440-02-0	5.0	[a]	[a]	20	4	0.447	ug/L
	Potassium	7440-09-7	53,000	No ESV	53,000	No ESC	200	112	ug/L
	Selenium	7782-49-2	1.0	No ESV	5.0	5.00	2.5	0.456	ug/L
	Silver	7440-22-4	0.067	[a]	[a]	0.12	2	0.194	ug/L
	Sodium	7440-23-5	680,000	No ESV	680,000	No ESC	200	58.2	ug/L
	Thallium	7440-28-0	0.030	110	12	10	0.8	0.168	ug/L
	Vanadium	7440-62-2	19	280	20	12	4	0.369	ug/L
	Zinc	7440-66-6	30	[a]	[a]	52	16	5.14	ug/L

Table 15-2
Reference Limits and Evaluation Table for Surface Water, Pore Water and Groundwater

Category	Analyte	CASRN	SLERA COPEC Selection ESV (µg/L) [1]	Refined SLERA ESVs[2]		NJDEP ESC	Eurofins SW846 Water		
				Acute ESV (µg/L)	Chronic ESV (µg/L)	Freshwater (ug/L)	Reporting Limit (µg/L)	Detection Limit (µg/L)	Units
Pesticides (EPA Method 8081B)	4,4´-DDD (Dichlorodiphenyldichloroethane)	72-54-8	0.011	0.19	0.011	0.00E+00	0.02	0.006	ug/L
	4,4´-DDE (Dichlorodiphenyldichloroethylene)	72-55-9	100	No ESV	100	4.51E-09	0.02	0.002	ug/L
	4,4´-DDT (Dichlorodiphenyltrichloroethane)	50-29-3	0.0010	1.1	0.0010	1.00E-03	0.02	0.004	ug/L
	Aldrin	309-00-2	0.30	3.0	0.30	0.02	0.02	0.003	ug/L
	alpha-BHC (alpha-Hexachlorocyclohexane or alpha-HCH)	319-84-6	2.2	39	2.2	12.40	0.02	0.007	ug/L
	beta-BHC (beta-Hexachlorocyclohexane or beta-HCH)	319-85-7	2.2	39	2.2	0.50	0.02	0.004	ug/L
	Chlordane	57-74-9	0.0043	2.4	0.0043	4.30E-03	#N/A	#N/A	#N/A
	Chlorobenzilate	510-15-6	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	cis-Chlordane	5103-71-9	0.0043	2.4	0.0043	No ESC	#N/A	#N/A	#N/A
	gamma-BHC (Lindane)	58-89-9	0.010	0.95	0.095	0	0.02	0.012	ug/L
	delta-BHC (delta-Hexachlorocyclohexane or delta-HCH)	319-86-8	2.2	39	2.2	No ESC	0.02	0.005	ug/L
	Diallate	2303-16-4	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	No ESV	No ESV	No ESV	No ESC	1	0.376	ug/L
	Dieldrin	60-57-1	0.056	0.24	0.056	0	0.02	0.003	ug/L
	Endosulfan I	959-98-8	0.0030	0.22	0.056	No ESC	0.02	0.002	ug/L
	Endosulfan II	33213-65-9	0.0030	0.22	0.056	No ESC	0.02	0.004	ug/L
	Endosulfan sulfate	1031-07-8	0.0030	0.22	0.056	2	0.02	0.006	ug/L
	Endrin	72-20-8	0.036	0.086	0.036	0	0.02	0.004	ug/L
	Endrin aldehyde	7421-93-4	0.036	0.086	0.036	0	0.02	0.008	ug/L
	Endrin ketone	53494-70-5	0.036	0.086	0.036	No ESC	0.02	0.008	ug/L
	Heptachlor	76-44-8	0.0038	0.52	0.0038	0	0.02	0.003	ug/L
	Heptachlor epoxide	1024-57-3	0.0038	0.52	0.0038	0	0.02	0.005	ug/L
	Isodrin	465-73-6	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	Methoxychlor	72-43-5	0.019	0.70	0.030	0	0.02	0.004	ug/L
	Toxaphene	8001-35-2	2.0E-04	0.73	2.0E-04	0	0.5	0.11	ug/L
	trans-Chlordane	5103-74-2	0.0043	2.4	0.0043	No ESC	#N/A	#N/A	#N/A

Table 15-2
Reference Limits and Evaluation Table for Surface Water, Pore Water and Groundwater

Category	Analyte	CASRN	SLERA COPEC Selection ESV (µg/L) [1]	Refined SLERA ESVs[2]		NJDEP ESC	Eurofins SW846 Water		
				Acute ESV (µg/L)	Chronic ESV (µg/L)	Freshwater (ug/L)	Reporting Limit (µg/L)	Detection Limit (µg/L)	Units
Polychlorinated Biphenyls (PCBs) (EPA Method 8082B)	Aroclor 1016	12674-11-2	0.014	No ESV	0.014	No ESC	0.4	0.119	ug/L
	Aroclor 1221	11104-28-2	0.014	5.0	0.28	No ESC	0.4	0.119	ug/L
	Aroclor 1232	11141-16-5	0.014	10	0.58	No ESC	0.4	0.119	ug/L
	Aroclor 1242	53469-21-9	0.014	1.2	0.053	No ESC	0.4	0.119	ug/L
	Aroclor 1248	12672-29-6	0.014	1.4	0.081	No ESC	0.4	0.119	ug/L
	Aroclor 1254	11097-69-1	0.014	0.60	0.033	No ESC	0.4	0.107	ug/L
	Aroclor 1260	11096-82-5	0.014	1,700	94	No ESC	0.4	0.107	ug/L
	PCBs/Total PCBs	1336-36-3	0.014	No ESV	0.014	0	0.4	0.119	ug/L

Table 15-2
Reference Limits and Evaluation Table for Surface Water, Pore Water and Groundwater

Category	Analyte	CASRN	SLERA COPEC Selection ESV (µg/L) [1]	Refined SLERA ESVs[2]		NJDEP ESC	Eurofins SW846 Water		
				Acute ESV (µg/L)	Chronic ESV (µg/L)	Freshwater (ug/L)	Reporting Limit (µg/L)	Detection Limit (µg/L)	Units
Dioxin/Furans (EPA Method 8290A)	Individual dioxin/furan congeners	--	[c]	[c]	[c]	No ESC	#N/A	#N/A	#N/A
	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)	1746-01-6	2E-06	No ESV	2E-06	0	10	0.0000012	ug/L

Table 15-2
Reference Limits and Evaluation Table for Surface Water, Pore Water and Groundwater

Category	Analyte	CASRN	SLERA COPEC Selection ESV (µg/L) [1]	Refined SLERA ESVs[2]		NJDEP ESC	Eurofins SW846 Water		
				Acute ESV (µg/L)	Chronic ESV (µg/L)	Freshwater (ug/L)	Reporting Limit (µg/L)	Detection Limit (µg/L)	Units
Volatile Organic Compounds (VOCs) (EPA Method 8260B)	1,1,1,2-Tetrachloroethane	630-20-6	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	1,1,1-Trichloroethane	71-55-6	11	200	11	76	1	0.238	ug/L
	1,1,2,2-Tetrachloroethane	79-34-5	610	2,100	610	380	1	0.367	ug/L
	1,1,2-Trichloroethane	79-00-5	1,200	5,200	1,200	500	1	0.433	ug/L
	1,1-Dichloroethane	75-34-3	47	830	47	No ESC	1	0.264	ug/L
	1,1-Dichloroethene	75-35-4	25	450	25	65	1	0.264	ug/L
	1,1-Dichloropropene	563-58-6	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	1,2,3-Trichlorobenzene	87-61-6	8.0	No ESV	8.0	0	1	0.357	ug/L
	1,2,3-Trichloropropane	96-18-4	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	1,2,3,4-Diepoxybutene	1464-53-5	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	1,2,4-Trichlorobenzene	120-82-1	24	700	110	30	1	0.365	ug/L
	1,2,4-Trimethylbenzene	95-63-6	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	1,2-Dibromo-3-chloropropane	96-12-8	No ESV	No ESV	No ESV	No ESC	1	0.376	ug/L
	1,2-Dibromoethane	106-93-4	No ESV	No ESV	No ESV	No ESC	1	0.498	ug/L
	1,2-Dichlorobenzene	95-50-1	0.70	260	14	No ESC	1	0.431	ug/L
	1,2-Dichloroethane	107-06-2	100	8,800	910	910	1	0.43	ug/L
	1,2-Dichloroethene (total)	540-59-0	590	1,100	590	No ESC	#N/A	#N/A	#N/A
	1,2-Dichloropropane	78-87-5	No ESV	No ESV	No ESV	360	1	0.353	ug/L
	1,3,5-Trimethylbenzene	108-67-8	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	1,3-Dichloro-2-propanol	96-23-1	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	1,3-Dichlorobenzene	541-73-1	71	630	71	38	1	0.342	ug/L
	1,3-Dichloropropane	142-28-9	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	1,4-Dichlorobenzene	106-46-7	15	180	15	9	1	0.334	ug/L
	1,4-Dioxane	123-91-1	No ESV	No ESV	No ESV	No ESC	50	28.2	ug/L
	1-Chlorobutane	109-69-3	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	1-Chlorohexane	544-10-5	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	1-Propanol	71-23-8	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	2,2-Dichloropropane	594-20-7	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	2-Butanone (Methyl ethyl ketone) (MEK)	78-93-3	7,200	240,000	14,000	No ESC	5	1.85	ug/L
	2-Chloroethanol	107-07-3	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	2-Chloroethyl vinyl ether	110-75-8	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	2-Chlorotoluene	95-49-8	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	2-Hexanone	591-78-6	99	1,800	99	No ESC	5	1.14	ug/L
	2-Hydroxypropionitrile	78-97-7	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	2-Nitropropane	79-46-9	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	2-Pentanone	107-87-9	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	2-Picoline	109-06-8	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	2-Propanol	67-63-0	7.5	130	7.5	No ESC	#N/A	#N/A	#N/A
	3-Chloropropionitrile	542-76-7	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	4-Chlorotoluene	106-43-4	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	4-Methyl-2-pentanone (MIBK)	108-10-1	170	2,200	170	No ESC	5	1.3	ug/L
	Acetone	67-64-1	1,500	28,000	1,500	No ESC	5	4.42	ug/L
	Acetonitrile	75-05-8	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	Acrolein (Propenal)	107-02-8	3.0	3.0	3.0	0	#N/A	#N/A	#N/A

Table 15-2
Reference Limits and Evaluation Table for Surface Water, Pore Water and Groundwater

Category	Analyte	CASRN	SLERA COPEC Selection ESV (µg/L) [1]	Refined SLERA ESVs[2]		NJDEP ESC	Eurofins SW846 Water		
				Acute ESV (µg/L)	Chronic ESV (µg/L)	Freshwater (ug/L)	Reporting Limit (µg/L)	Detection Limit (µg/L)	Units
Volatile Organic Compounds (VOCs) (EPA Method 8260B)	Acrylonitrile	107-13-1	No ESV	No ESV	No ESV	66	#N/A	#N/A	#N/A
	Allyl Alcohol	107-18-6	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	Allyl chloride	107-05-1	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	Benzene	71-43-2	46	2,300	130	114	1	0.203	ug/L
	Benzyl chloride	100-44-7	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	beta-Propiolactone	57-57-8	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	Bis(2-chloroethyl)sulfide	505-60-2	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	Bromoacetone	598-31-2	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	Bromobenzene	108-86-1	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	Bromochloromethane	74-97-5	No ESV	No ESV	No ESV	No ESC	1	0.412	ug/L
	Bromodichloromethane	75-27-4	No ESV	No ESV	No ESV	0	1	0.343	ug/L
	Bromoform	75-25-2	320	2,300	320	230	1	0.536	ug/L
	Bromomethane	74-83-9	1,300	1,300	No ESV	16	1	0.55	ug/L
	Carbon disulfide	75-15-0	0.92	17	0.92	No ESC	1	0.821	ug/L
	Carbon tetrachloride	56-23-5	9.8	180	9.8	240	1	0.208	ug/L
	Chloral hydrate	302-17-0	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	Chloroacetonitrile	107-14-2	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	Chlorobenzene	108-90-7	1.3	1,100	64	47	1	0.377	ug/L
	Chloroethane	75-00-3	No ESV	No ESV	No ESV	No ESC	1	0.32	ug/L
	Chloroform	67-66-3	1.8	490	28	140	1	0.326	ug/L
	Chloromethane	74-87-3	No ESV	No ESV	No ESV	No ESC	1	0.402	ug/L
	Chloroprene	126-99-8	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	Crotonaldehyde	4170-30-3	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	cis-1,2-Dichloroethene	156-59-2	590	1,100	590	No ESC	1	0.219	ug/L
	cis-1,3-Dichloropropene	10061-01-5	0.055	0.99	0.055	No ESC	1	0.222	ug/L
	cis-1,4-Dichloro-2-butene	1476-11-5	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	Dibromochloromethane (Chlorodibromomethane)	124-48-1	No ESV	No ESV	No ESV	0	1	0.281	ug/L
	Dibromofluoromethane	1868-53-7	No ESV	No ESV	No ESV	No ESC			ug/L
	Dibromomethane	74-95-3	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	Dichlorodifluoromethane	75-71-8	No ESV	No ESV	No ESV	No ESC	1	0.311	ug/L
	Diethyl ether	60-29-7	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	Epichlorohydrin	106-89-8	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	Ethanol	64-17-5	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	Ethyl acetate	14178-6	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	Ethylbenzene	100-41-4	7.3	130	7.3	14	1	0.298	ug/L
	Ethylene oxide	75-21-8	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	Ethyl methacrylate	97-63-2	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	Hexachloroethane	67-72-1	12	210	12	8	2	0.803	ug/L
	Iodomethane	74-88-4	285	285	No ESV	No ESC	#N/A	#N/A	#N/A
	Isobutyl alcohol	78-83-1	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	Isopropylbenzene	98-82-8	No ESV	No ESV	No ESV	No ESC	1	0.336	ug/L
	Malononitrile	109-77-3	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	Methacrylonitrile	126-98-7	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	Methanol	67-56-1	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A

Table 15-2
Reference Limits and Evaluation Table for Surface Water, Pore Water and Groundwater

Category	Analyte	CASRN	SLERA COPEC Selection ESV (µg/L) [1]	Refined SLERA ESVs[2]		NJDEP ESC	Eurofins SW846 Water		
				Acute ESV (µg/L)	Chronic ESV (µg/L)	Freshwater (ug/L)	Reporting Limit (µg/L)	Detection Limit (µg/L)	Units
Volatile Organic Compounds (VOCs) (EPA Method 8260B)	Methyl acetate	79-20-9	No ESV	No ESV	No ESV	No ESC	5	0.785	ug/L
	Methyl methacrylate	80-62-6	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	Methyl tert-butyl ether (MTBE)	1634-04-4	10,000	No ESV	10,000	51000	1	0.465	ug/L
	Methylene chloride	75-09-2	98	26,000	2,200	940	1	0.315	ug/L
	n-Butanol	71-36-3	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	n-Butylbenzene	104-51-8	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	N-Nitroso-di-n-butylamine	924-16-3	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	n-Propylamine	107-10-8	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	n-Propylbenzene	103-65-1	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	p-Isopropyltoluene	99-87-6	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	Paraldehyde	123-63-7	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	Pentachloroethane	76-01-7	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	Pentafluorobenzene	363-72-4	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	Propargyl alcohol	107-19-7	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	Propionitrile (ethyl cyanide)	107-12-0	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	o-Toluidine	95-53-4	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	sec-Butylbenzene	135-98-8	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	Styrene	100-42-5	72	No ESV	72	32	1	0.415	ug/L
	tert-Butylbenzene	98-06-6	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	Tetrachloroethene	127-18-4	50	830	98	45	1	0.249	ug/L
	Toluene	108-88-3	2.0	120	9.8	253	1	0.379	ug/L
	trans-1,2-Dichloroethene	156-60-5	590	1,100	590	970	1	0.235	ug/L
	trans-1,3-Dichloropropene	10061-02-6	0.055	0.99	0.055	No ESC	1	0.485	ug/L
	trans-1,4-Dichloro-2-butene	110-57-6	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A
	Trichloroethene	79-01-6	21	440	47	47	1	0.314	ug/L
	Trichlorofluoromethane	75-69-4	No ESV	No ESV	No ESV	No ESC	1	0.32	ug/L
	Vinyl acetate	108-05-4	16	280	16	No ESC	#N/A	#N/A	#N/A
	Vinyl chloride	75-01-4	No ESV	No ESV	No ESV	930	1	0.171	ug/L
	Xylene, m-	108-38-3	1.8	32	1.8	No ESC	#N/A	#N/A	#N/A
	Xylenes, m+p-	179601-23-1	1.8	32	1.8	No ESC	1	0.296	ug/L
	Xylene, o-	95-47-6	13	230	13	No ESC	1	0.361	ug/L
	Xylene, p-	106-42-3	1.8	32	1.8	No ESC	#N/A	#N/A	#N/A
	Xylenes, Total	1330-20-7	13	No ESV	62,308	27	#N/A	#N/A	#N/A

Table 15-2
Reference Limits and Evaluation Table for Surface Water, Pore Water and Groundwater

Category	Analyte	CASRN	SLERA COPEC Selection ESV (µg/L) [1]	Refined SLERA ESVs[2]		NJDEP ESC	Eurofins SW846 Water		
				Acute ESV (µg/L)	Chronic ESV (µg/L)	Freshwater (ug/L)	Reporting Limit (µg/L)	Detection Limit (µg/L)	Units
Polycyclic Aromatic Hydrocarbons (PAHs) (EPA Method 8270D)	***LOW MOLECULAR WEIGHT***		*LOW MOLECULAR WEIGHT*			0	#N/A	#N/A	#N/A
	2-Methylnaphthalene	91-57-6	330	No ESV	330	330	#N/A	#N/A	ug/L
	Acenaphthene	83-32-9	5.8	No ESV	5.8	38	0.05	0.0142	ug/L
	Acenaphthylene	208-96-8	4,800	No ESV	4,800	4840	0.05	0.0146	ug/L
	Anthracene	120-12-7	0.012	13	0.73	3.50E-02	0.05	0.0092	ug/L
	Fluoranthene	206-44-0	0.040	No ESV	0.040	2	0.05	0.039	ug/L
	Fluorene	86-73-7	3.0	70	3.9	19	0.05	0.0118	ug/L
	Naphthalene	91-20-3	1.1	190	12	13	0.2	0.124	ug/L
	Phenanthrene	85-01-8	0.40	No ESV	0.40	4	0.05	0.0219	ug/L
	HIGH MOLECULAR WEIGHT		*HIGH MOLECULAR WEIGHT*			0.00E+00	#N/A	#N/A	#N/A
	Benzo(a)anthracene	56-55-3	0.018	0.49	0.027	2.50E-02	0.05	0.0156	ug/L
	Benzo(g,h,i)perylene	191-24-2	7.6	No ESV	7.6	8	0.05	0.0351	ug/L
	Benzo(a)pyrene	50-32-8	0.014	0.24	0.014	1.40E-02	0.05	0.0216	ug/L
	Benzo(b)fluoranthene	205-99-2	9.0	No ESV	9.0	9	0.05	0.024	ug/L
	Benzo(k)fluoranthene	207-08-9	0.0041	No ESV	0.0041	0.00E+00	0.05	0.0278	ug/L
	Chrysene	218-01-9	0.0018	No ESV	0.0018	0.00E+00	0.05	0.0299	ug/L
	Indeno(1,2,3-cd)pyrene	193-39-5	4.3	No ESV	4.3	4	0.05	0.0362	ug/L
	Pyrene	129-00-0	0.025	No ESV	0.025	3.00E-01	0.05	0.0314	ug/L
Semi-volatile Organic Chemicals (SVOCs) (EPA Method 8270D)	2,2'-oxybis (1-chloropropane)	108-60-1	No ESV	No ESV	No ESV	0.00E+00	10	0.629	ug/L
	2,4,5-Trichlorophenol	95-95-4	18	No ESV	18	No ESC	10	0.88	ug/L
	2,4,6-Trichlorophenol	88-06-2	18	No ESV	18	5	10	0.857	ug/L
	2,4-Dichlorophenol	120-83-2	0.20	No ESV	0.20	11	10	1.07	ug/L
	2,4-Dimethylphenol	105-67-9	No ESV	No ESV	No ESV	100	10	0.619	ug/L
	2,4-Dinitrophenol	51-28-5	No ESV	No ESV	No ESV	19	20	14.4	ug/L
	2,4-Dinitrotoluene	121-14-2	65	No ESV	65	44	2	0.997	ug/L
	2,6-Dinitrotoluene	606-20-2	230	No ESV	230	No ESC	2	0.826	ug/L
	2-Chloronaphthalene	91-58-7	No ESV	No ESV	No ESV	No ESC	10	1.18	ug/L
	2-Chlorophenol	95-57-8	7.0	No ESV	7.0	No ESC	10	0.377	ug/L
	2-Methylphenol	95-48-7	13	230	13	No ESC	10	0.671	ug/L
	2-Nitroaniline	88-74-4	No ESV	No ESV	No ESV	No ESC	10	0.474	ug/L
	2-Nitrophenol[k]	88-75-5	60	1,200	300	No ESC	10	0.747	ug/L
	3,3'-Dichlorobenzidine	91-94-1	No ESV	No ESV	No ESV	No ESC	10	1.43	ug/L
	3-Nitroaniline	99-09-2	No ESV	No ESV	No ESV	No ESC	10	1.94	ug/L
	4,6-Dinitro-2-methylphenol	534-52-1	No ESV	No ESV	No ESV	0.00E+00	20	13.3	ug/L
	4-Bromophenyl phenyl ether	101-55-3	1.5	No ESV	1.5	No ESC	10	0.745	ug/L
	4-Chloro-3-methylphenol	59-50-7	No ESV	No ESV	No ESV	No ESC	10	0.575	ug/L
	4-Chloroaniline	106-47-8	No ESV	No ESV	No ESV	No ESC	10	1.88	ug/L
	4-Chlorophenyl phenyl ether	7005-72-3	No ESV	No ESV	No ESV	No ESC	10	1.28	ug/L
	4-Methylphenol	106-44-5	No ESV	No ESV	No ESV	No ESC	10	0.651	ug/L
	4-Nitroaniline	100-01-6	No ESV	No ESV	No ESV	No ESC	10	1.22	ug/L
	4-Nitrophenol	100-02-7	60	1,200	300	60	20	3.98	ug/L
	bis(-2-chloroethoxy)Methane	111-91-1	No ESV	No ESV	No ESV	No ESC	10	0.589	ug/L
	bis(-2-chloroethyl)Ether	111-44-4	No ESV	No ESV	No ESV	1900	1	0.633	ug/L
	bis(2-ethylhexyl)Phthalate	117-81-7	3.0	27	3.0	3.00E-01	2	1.7	ug/L

Table 15-2
Reference Limits and Evaluation Table for Surface Water, Pore Water and Groundwater

Category	Analyte	CASRN	SLERA COPEC Selection ESV (µg/L) [1]	Refined SLERA ESVs[2]		NJDEP ESC	Eurofins SW846 Water		
				Acute ESV (µg/L)	Chronic ESV (µg/L)	Freshwater (ug/L)	Reporting Limit (µg/L)	Detection Limit (µg/L)	Units
Semi-volatile Organic Chemicals (SVOCs) (EPA Method 8270D)	Butylbenzylphthalate	85-68-7	19	No ESV	19	238	10	0.854	ug/L
	Carbazole	86-74-8	No ESV	No ESV	No ESV	No ESC	10	0.679	ug/L
	Dibenzo(a,h)anthracene	53-70-3	0.0034	No ESV	0.0034	0.00E+00	1	0.72	ug/L
	Dibenzofuran	132-64-9	3.7	66	3.7	No ESC	10	1.1	ug/L
	Di-n-butylphthalate (butyl phthalate)	84-74-2	19	190	35	10	10	0.84	ug/L
	Di-n-octyl phthalate	117-84-0	3.0	No ESV	708	No ESC	10	4.75	ug/L
	Diethylphthalate	84-66-2	210	1,800	210	110	10	0.976	ug/L
	Dimethylphthalate	131-11-3	3.0	No ESV	3.0	No ESC	10	0.766	ug/L
	Hexachlorobenzene	118-74-1	No ESV	No ESV	No ESV	3.00E-04	1	0.396	ug/L
	Hexachlorobutadiene	87-68-3	1.3	No ESV	1.3	5.30E-02	1	0.78	ug/L
	Hexachlorocyclopentadiene	77-47-4	No ESV	No ESV	No ESV	77	10	3.64	ug/L
	Hexachloroethane	67-72-1	12	210	12	8	2	0.803	ug/L
	Isophorone	78-59-1	No ESV	No ESV	No ESV	920	10	0.798	ug/L
	N-Nitrosodi-n-propylamine	621-64-7	No ESV	No ESV	No ESV	0.00E+00	1	0.43	ug/L
	N-Nitrosodiphenylamine	86-30-6	40	3,800	210	0.00E+00	10	0.891	ug/L
	Nitrobenzene	98-95-3	550	No ESV	550	No ESC	1	0.567	ug/L
	Pentachlorophenol	87-86-5	0.50	19	15	(b)	20	1.45	ug/L
	Phenol	108-95-2	4.0	No ESV	4.0	180	10	0.292	ug/L
	Pyridine	110-86-1	No ESV	No ESV	No ESV	No ESC	#N/A	#N/A	#N/A

Table 15-2
Reference Limits and Evaluation Table for Surface Water, Pore Water and Groundwater

Category	Analyte	CASRN	SLERA COPEC Selection ESV (µg/L) [1]	Refined SLERA ESVs[2]		NJDEP ESC	Eurofins SW846 Water		
				Acute ESV (µg/L)	Chronic ESV (µg/L)	Freshwater (ug/L)	Reporting Limit (µg/L)	Detection Limit (µg/L)	Units
Anions	Cyanide (Methods 9010B/9013/9014)	57-12-5	5.0	22	5.2	5	0.01	0.004	mg/L

-- = indicates that no ESV is available from the designated source

[1] The lowest available ESV from approved sources should be selected as the SLERA ESL to select COPECs and be protective of all receptors.

[2] Acute and chronic ESVs are selected based on the hierarchy described in Section 2.1.2. Where appropriate, species-specific ESVs may be obtained from toxicity literature or other additional sources with prior approval of NPS.

1 -- EcoSSL
2 -- LANL ECORISK Database
3 -- Sample et al. (1996) food-based value

Notes:
[a] Use hardness-dependant equations to calculate site-specific ESVs, assumes a hardness of 40 mg/L
[b] EPA recommends use of the Biotic Ligand Model (BLM) to assess copper toxicity (EPA 2007). See: http://water.epa.gov/scitech/swguidance/standards/criteria/aqlife/copper/2007_index.cfm
[c] Individual congeners should be evaluated in terms of toxicity equivalence relative to TCDD.

µg/L = micrograms per liter
BERA = baseline ecological risk assessment
BLM = Biotic Ligand Model
$CaCO_3$ = calcium carbonate
CASRN = Chemical Abstracts Service Registry Number
CCC = Criterion Continuous Concentration
CCME = Canadian Council of Ministers of the Environment
CMC = Criteria Maximum Concentration
COPEC = chemical of potential ecological concern
DOC = dissolved organic carbon
EC20 = effect concentration for 20% of exposed organisms
EPA = Environmental Protection Agency
ESL = ecological screening level
ESV = ecological screening value
GLWQI = Great Lakes Water Quality Initiative
HQ = hazard quotient
LANL = Los Alamos National Laboratory
LC50 = concentration lethal to 50% of the test population
LCV = Lowest Chronic Value
mg/L = milligrams per liter

0.011 Highlighted standard is lower than the laboratory method detection limit
#NA Not Analyzed

16. QAPP WORKSHEET #16 – PROJECT SCHEDULE/TIMELINE

This worksheet shows the schedule of all project activities to be performed.

TBD

17. QAPP WORKSHEET #17 -PROJECT SAMPLE DESIGN AND RATIONALE

The main objectives of the sample design and rationale presented in this worksheet are to meet the CERCLA RI requirements, which include the following items.

1. Develop a comprehensive hydrogeological CSM from data collected at the sampling sites.
2. Determine the nature and extent of contamination that may threaten human health and the environment, with definitive data.
3. Identify and quantify existing or potential contaminant fate parameters and transport pathways, and bioavailability.
4. Screen environmental sampling results against project screening levels for current and future receptors that may become exposed to contaminants within the Study Area.
5. Collect sufficient data to choose a remedial alternative in the Feasibility Study.

Analytical data to be gathered at each site is to augment data provided in the previous CERCLA phases. The RI sampling approach focused on source areas with a limited number of soil, sediment, surface water, pore water and groundwater sampling points located on the Refuge.

17.1 Soil Sampling

Figure 7 shows the site location, site boundaries, and proposed soil for subsurface soil sampling locations.

Soil samples will be collected by hand auger and direct push technology (DPT) rig in a systematic manner to delineate landfill debris laterally and vertically within the Refuge, and to delineate COPC/COPEC horizontally and vertically within the Refuge.

Surface soil samples and subsurface soil samples will be collected at 30 direct push boring locations. Surface samples will be collected with a hand auger at each boring location. Subsurface soil samples will be collected continuously through the Landfill material to the total depth of each boring using direct push technology in dedicated, disposable clear acetate macro-core samplers for borehole logging. Soil cores will be logged for lithology following the ASTM (2009) Visual-Manual procedure and those data will be recorded on the appropriate Field Form (Appendix C) in accordance with the SOP (Appendix B). The entire core will be screened visually and with the project photo ionization detector (PID)/flame ionization detector (FID) and maximum readings for every foot will be recorded on the project bore log.

Samples will be analyzed at the project laboratory for target analytes:

- VOCs by SW846 Method 8260C;
- SVOCs by SW846 Method 8270D;
- Pesticides by SW846 Method 8081B;
- PCB (Aroclors) by SW846 Method 8082A;

- Selected samples for PCB congeners (approximately 30%);
- Selected samples for dioxins/furans by SW846 Method 8280A(approximately 30%);
- TAL metals by SW846 Methods 8020B and 7471B for mercury; and
- SW846 Method 9012B for cyanide.

A maximum of 10 DPT soil borings will be sampled within the Northeastern Refuge Study Area (N, approximately 10 acres) and 20 DPT soil borings will be sampled in the Southern Refuge Study Area (S, approximately 20 acres). Surface soil samples will be collected from the DPT location with a decontaminated hand auger from 0 to 0.5 ft bgs at all boring locations.

Subsurface soil (landfill material) will be collected to the total depth of the landfill material in the following intervals:

- A composite subsurface soil sample will be collected from 0.5 ft to the total depth of the Landfill from each boring;
- If evidence of elevated contamination is present at a specific interval within each boring, that interval will be discretely sampled, with a maximum one discrete sample per boring;
- A discrete subsurface soil sample will be analyzed from the native soil below the Landfill in each boring to determine if leaching has occurred.

Each sampling interval will be composited and the composite sample will be analyzed in the laboratory by the following methods:

- VOCs;
- SVOCs;
- Pesticides;
- PCBs (Aroclors);
    - If PCBs are detected but do not conform to Aroclor mixture,
        - PCB congeners will be analyzed by Method 1668;
            - Maximum of 24 samples (33%) will be analyzed by Method 1668.
        - Dioxins/Furans will be analyzed by Method 8290A
            - Maximum of 24 samples will be analyzed by8290A.
- TAL Metals, including mercury; and
- Cyanide.

17.2 Pore-Water Samples

Sediment pore-water (i.e., interstitial water) samples will be collected from areas adjacent to the Site where contaminated groundwater is expected to discharge into Refuge wetlands. Pore-water samples will be collocated with sediment and surface-water samples to evaluate relationships between sediment, pore water, and surface water.

Biota within the transition zone (the zone where groundwater and surface water mix) may be adversely impacted by contaminated groundwater discharging through the transition zone into overlying surface-waters. The hyporheic zone (transition zone between groundwater and surface water) is constantly in flux; therefore, pore-water samples will be targeted from depths below the groundwater interface to minimize entrainment of surface water. Metals data collected from monitoring wells MW-3 (terrestrial) and X-3 (wetland), as part to the Geosyntec (2017) monitored natural attenuation investigation suggests that the transition zone extending from the terrestrial ecosystem into the vegetative wetlands is less than 500 feet wide.

The pore water data will be used to determine locations where contaminated groundwater discharges to the wetlands and if needed, to evaluate the bioavailability and potential effects of contaminants discharging to surface water. The freely dissolved fraction of nonionic organic chemicals in sediment (i.e., that fraction that partitions into pore water) represents the bioavailable fraction. Therefore, the pore water concentration data can be used to predict sediment toxicity.

Pore-water samples will be collected from up to 50 locations within the geochemical transition zone on the Refuge property within the wetland area. As shown in Figures 8a and 8b, the pore water sampling locations are oriented along 25 transects, each transect consists of a minimum of 3 pore-water sampling locations. The objective of this sampling is to identify likely areas where mildly reducing pore water is discharging to the wetlands. In these areas, dissolved metals could concentrate within the pore water, and/or precipitate from the pore water upon entering more aerobic conditions thereby potentially impacting sediment and surface water.

The first sampling location at each transect will be located near the interface of the terrestrial ecosystem and vegetative wetlands as mapped during the RI (Figures 8a and 8b). It is anticipated that this pore-water sample will be collected from a depth of about 5 ft below the water table, if field conditions permit.

At each pore-water sampling location, HACH test kits will be used to detect concentrations of arsenic, manganese, lead, and iron in both filtered and unfiltered pore water. Measurements will also be made of temperature, ORP, turbidity, pH, and specific conductance. Field testing of porewater for ferrous iron using a HACH portable colorimeter test kit will also be performed to evaluate the existence of reducing (i.e., anaerobic) conditions. Detectable concentrations of ferrous Fe^{2+} (above 0.01 mg/L) will confirm the existence of anaerobic conditions.

Based on the HACH test kit and field measurement results, a second sampling point will be selected along the transect. For example, if high concentrations of dissolved metals are observed and/or low ORP values at the first sampling location then the second point would be set about 250 feet from the first sampling point. If dissolved metals concentrations are relatively low then the second sampling point would be located about 100 feet from the first point. The sampling depth for this sample would be about 2 to 3 feet below the water table surface but may be revised based on field screening results. Once a sufficient number of points have been sampled

to establish the geochemical gradient and likely discharge points, pore-water samples from two of the sampling locations along each transect will be sent to the Project laboratory for analysis of the following:

- VOCs;
- SVOCs;
- Pesticides;
- DOC
- TOC
- Alkalinity;
- Hardness as C_aCO_3, and
- TAL Metals (filtered and unfiltered), including mercury; and cyanide.

Pore-water will also be collected from approximately 10 locations within the terrestrial portions of the Refuge property adjacent to additional wetland areas, that may be identified during the field investigation. Pore-water samples in the terrestrial areas will be collected at a depth of about 2-3 feet below the water table and field screened for the same parameters as the pore-water samples collected from the geochemical transition zone. These pore-water samples would assist in selecting sediment and surface water samples as described in Sections 17.4 and 17.5, respectively. Based on field screening results of the 10 locations, up to 5 pore-water samples would be sent to the Project laboratory and analyzed for the same constituents as the geochemical transition zone pore-water samples. All 5 of pore-water samples will also be analyzed for PFOA and PFOS and all porewater samples will be analyzed for 1,4-dioxane.

All pore water samples will be collected in accordance with the SOPs identified in Worksheet #21.

17.3 Surface Water Samples

Figure 7, Figure 8a and Figure 8b show the site location, site boundaries, and proposed surface water and sediment sampling locations. Surface water samples will be collected from locations within the Refuge, adjacent to the Refuge property, and upgradient of the Refuge portion of the Site. Surface water samples will be collocated with sediment and pore water samples. A maximum of 10 surface-water samples will be collected and analyzed.

Surface water contaminant concentrations from within the Refuge portion of the Site and adjacent to the Refuge property will be used to evaluate potential migration pathways, the spatial extent and relative concentrations of Site-related contaminants. Surface water contaminant concentrations from upgradient of the Refuge portion of the Site will be used to evaluate potential upgradient surface water contaminant contributions. Surface water flow measurements from upgradient, within the Refuge, and adjacent to the Refuge property will be used to evaluate the relative contributions of groundwater to surface water flow.

Surface-water samples will be analyzed in the project laboratory and will include unfiltered aliquots and filtered aliquots (0.45 micron) for TAL metals:

- SVOCs;
- Pesticides;
- PCBs;
- TAL Metals, including mercury and cyanide;
- DOC; and
- Hardness.

Surface-water sample locations will be collocated with selected pore-water and sediment samples based on availability and professional judgement. Data for the placement of pore-water samples will include the observation of seeps and surface water bodies within the study area and field measurements in soil and groundwater from DPT and along embankments, including:

- Elevated FID readings in soil and headspace;
- Staining on soil cores and on embankments; and
- Field test kit results for dissolved iron.

17.4 Sediment Samples

Figure 7, Figure 8a and Figure 8b show the site location, site boundaries, and proposed surface water and sediment sampling locations. Sediment samples will be collected from open surface water bodies and/or surface water drainage swales within the Refuge portion of the Site, adjacent to the Refuge property, and upgradient of the Refuge portion of the Site. Sediment sample locations will be collocated with pore water and surface water samples when practical. A maximum of 20 sediment samples will be collected and analyzed.

The sediment contaminant concentrations within the Refuge portion of the Site and adjacent Refuge property will be used to evaluate potential migration pathways, the spatial extent and relative concentrations of Site-related contaminants. Sediment contaminant concentrations from upgradient of the Refuge portion of the Site will be used to evaluate potential upgradient sediment contaminant contributions. Upgradient sediment samples will not be evaluated for bioavailability parameters described in the next paragraphs.

Sediment acid-volatile sulfides (AVS), grain size, and TOC may vary substantially over relatively short distances and have been shown to influence contaminant mobility, bioavailability, and toxicity. Potential toxicity of the sediment is evaluated by calculating the sum of the simultaneously extracted metals (SEM) on a molar basis (µmol/g dry weight) divided by the AVS concentration (in the same units). If AVS exceeds SEM then the metals are likely to precipitate to sulfide and this should limit bioavailability, based on the hypothesis that metals will react with the sulfide to form insoluble compounds that are not bioavailable to benthic organisms.

Organic carbon normalization of AVS/SEM ([ΣSEM-AVS]/f_{oc}) has been shown to reduce the variability associated with prediction sediment toxicity, where f_{oc} is the fraction of organic carbon. Both the difference between the ΣSEM and AVS [ΣSEM–AVS] and the organic carbon-normalized difference between SEM and AVS ([ΣSEM-AVS]/f_{oc}) models will be used as parameters to evaluate potential bioavailability of metals in sediment. According to U.S. EPA (2005):

1) Any sediment with AVS > 0.1 µmol/g OC will not cause adverse biological effects due to chromium or silver.
2) Any sediment in which (SEM-AVS)/f_{OC} < 130 µmol/g OC should pose low risk of adverse biological effects due to cadmium, copper, lead, nickel and zinc.
3) Any sediment in which 130 µmol/g OC < (SEM-AVS)/f_{OC} < 3,000 µmol/g OC may have adverse biological effects due to cadmium, copper, lead, nickel or zinc.
4) In any sediment in which (SEM-AVS)/f_{OC} > 3,000 µmol/g OC adverse biological effects due to cadmium, copper, lead, nickel or zinc may be expected.

Proper sample collection, preservation, and storage are critical for sediment samples collected for AVS/SEM. This is because the sulfide ion is unstable in the presence of oxygen and therefore samples must be protected from exposure to oxygen during sample collection and storage. Sediment samples should be collected in wide mouth jars with zero headspace. The samples should be cooled to 4°C as soon as possible after collection.

Sediment samples will be analyzed in the Project laboratory for:

- VOCs;
- SVOCs;
- Pesticides;
- PCBs (Aroclors);
    - If PCBs are detected but do not conform to Aroclor mixture,
    - PCB congeners will be analyzed by Method 1668;
    - Maximum of 7samples (33%) will be analyzed by Method 1668.
    - Dioxins/Furans will be analyzed by Method 8290A
    - Maximum of 7 samples will be analyzed by8290A. and
- TAL Metals, plus total mercury and cyanide.

Bioavailability parameters will be analyzed in sediments collected within the Refuge:

- TOC;
- Grain size;
- pH;
- ORP;
- AVS/SEM;
- Moisture content.

17.5 Geotechnical Samples

Geotechnical samples will be collected during the Data Gap Investigation to evaluate the geotechnical properties of the clay layer below the Landfill on the Refuge portion of the Site (Attachment 1) and assess its suitability as cover and surrounding material for an onsite repository located on the private portion of the Landfill. This data will be available as proof of concept, with Site-specific geotechnical data of repository parameters to be part of the remedial design that is out of the scope of this investigation. The rationale for using the clay beneath the Site for capping and encasing material is based on the geotechnical properties of the clay layer incorporated into the nearby Asbestos Dump Superfund Site remedial design.

Ten geotechnical samples will be collected from the clay layer illustrated in Attachment 1. Sample locations will be determined in the field under the direction of the engineer of record. Samples will be analyzed by an accredited geotechnical laboratory for the following American Society for Testing and Materials (ASTM) methods:

- Compaction – ASTM D698 Laboratory Compaction, Standard Effort
- Moisture - ASTM D 2216 Standard Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass
- Permeability - ASTM D5084 Standard Test Method for Permeability of Fine Soils (Constant Head)
- Compressibility – ASTM D2435 One-Dimensional Consolidation Test
- Triaxial Stress – ASTM D2850 Unconsolidated, Undrained Pore Pressure
- Atterberg Limits - ASTM D 4318 Standard Test Methods for Liquid Limit, Plastic limit, and Plasticity Index of Soils
- Density - ASTM D7263 Standard Test Methods for Laboratory Determination of Density (Unit Weight) of Soil Specimens.

18. QAPP WORKSHEET #18 - SAMPLE LOCATIONS AND METHODS / SOP REQUIREMENTS

This worksheet summarizes the investigative samples to be collected. Field QC samples are identified in Worksheet #20.

Table 18-1. Surface Soil Sampling Locations and Methods/SOP Requirements

Sample Location	Sample ID[1]	Matrix	Depth Interval (ft)	Analytical Group	Sample Type	Sampling SOP / Laboratory
Northeastern Refuge Study Area						
GS-N-SS1	GS-N-SS1	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-06 / Appendix E
GS-N-SS1	GS-N-SS100	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Duplicate	AI-W-ES-06 / Appendix E
GS-N-SS2	GS-N-SS2	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal, MS/MSD	AI-W-ES-06 / Appendix E
GS-N-SS3	GS-N-SS3	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-06 / Appendix E
GS-N-SS4	GS-N-SS4	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-06 / Appendix E
GS-N-SS5	GS-N-SS5	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-06 / Appendix E
GS-N-SS6	GS-N-SS6	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-06 / Appendix E
GS-N-SS7	GS-N-SS7	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-06 / Appendix E
GS-N-SS8	GS-N-SS8	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-06 / Appendix E

Table 18-1. Surface Soil Sampling Locations and Methods/SOP Requirements						
Sample Location	**Sample ID[1]**	**Matrix**	**Depth Interval (ft)**	**Analytical Group**	**Sample Type**	**Sampling SOP / Laboratory**
Northeastern Refuge Study Area						
GS-N-SS9	GS-N-SS9	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as	Normal	AI-W-ES-06 / Appendix E
GS-N-SS10	GS-N-SS10	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-06 / Appendix E

ft – foot
MS/MSD – Matrix Spike/Spike Duplicate
N – Northeastern Refuge Study Area
PCBs – Polychlorinated Biphenyls
SOP – Standard Operating Procedure
SS – Surface Soil
SVOCs – Semi-volatile Organic Compounds
TAL – Target Analyte List
TCLP - Toxicity Characteristic Leaching Procedure
VOCs - Volatile Organic Compounds

Table 18-2. Surface Soil Sampling Locations and Methods/SOP Requirements						
Sample Location	**Sample ID[1]**	**Matrix**	**Depth Interval (ft)**	**Analytical Group**	**Sample Type**	**Sampling SOP / Laboratory SOP**
Northeastern Refuge Study Area						
GS-S-SS11	GS-S-SS11	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-06 / Appendix E
GS-S-SS11	GS-S-SS102	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Duplicate	AI-W-ES-06 / Appendix E
GS-S-SS12	GS-S-SS12	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-06 / Appendix E
GS-S-SS13	GS-S-SS13	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-06 / Appendix E
GS-S-SS14	GS-S-SS14	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-06 / Appendix E
GS-S-SS15	GS-S-SS15	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-06 / Appendix E
GS-S-SS16	GS-S-SS16	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-06 / Appendix E
GS-S-SS17	GS-S-SS17	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-06 / Appendix E
GS-S-SS18	GS-S-SS18	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-06 / Appendix E
GS-S-SS19	GS-S-SS19	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-06 / Appendix E

Table 18-2. Surface Soil Sampling Locations and Methods/SOP Requirements						
Sample Location	**Sample ID[1]**	**Matrix**	**Depth Interval (ft)**	**Analytical Group**	**Sample Type**	**Sampling SOP / Laboratory SOP**
Northeastern Refuge Study Area						
GS-S-SS20	GS-S-SS20	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal, MS/MSD	AI-W-ES-06 / Appendix E
GS-S-SS21	GS-S-SS21	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-06 / Appendix E
GS-S-SS21	GS-S-SS103	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Duplicate	AI-W-ES-06 / Appendix E
GS-S-SS22	GS-S-SS22	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-06 / Appendix E
GS-S-SS23	GS-S-SS23	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-06 / Appendix E
GS-S-SS24	GS-S-SS24	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-06 / Appendix E
GS-S-SS25	GS-S-SS25	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-06 / Appendix E
GS-S-SS26	GS-S-SS26	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-06 / Appendix E
GS-S-SS27	GS-S-SS27	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-06 / Appendix E
GS-S-SS28	GS-S-SS28	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-06 / Appendix E
GS-S-SS29	GS-S-SS29	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-06 / Appendix E

Table 18-2. Surface Soil Sampling Locations and Methods/SOP Requirements						
Sample Location	**Sample ID[1]**	**Matrix**	**Depth Interval (ft)**	**Analytical Group**	**Sample Type**	**Sampling SOP / Laboratory SOP**
Northeastern Refuge Study Area						
GS-S-SS30	GS-S-SS30	Soil Grab	0 to 0.5	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal, MS/MSD	AI-W-ES-06 / Appendix E

ft – foot
MS/MSD – Matrix Spike/Spike Duplicate
N – Northeastern Refuge Study Area
PCBs – Polychlorinated Biphenyls
SOP – Standard Operating Procedure
SS – Surface Soil
SVOCs – Semi-volatile Organic Compounds
TAL – Target Analyte List
TCLP - Toxicity Characteristic Leaching Procedure
VOCs - Volatile Organic Compounds

Table 18-3. Subsurface Soil Sampling Locations and Methods/SOP Requirements						
Sample Location	**Sample ID[1]**	**Matrix**	**Depth Interval (ft)**	**Analytical Group**	**Sample Type**	**Sampling SOP / Laboratory SOP**
Northeastern Refuge Study Area						
GS-N-DPT1	GS-N-DPT1-SO-0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-N-DPT1	GS-N-DPT100-SO-0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Duplicate	AI-W-ES-07/ Appendix E
GS-N-DPT1	GS-N-DPT1-SO-X-X	Soil Macro-core (Optional)	X to X	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-N-DPT1	GS-N-DPT1-SO-X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-N-DPT1	GS-N-DPT101-SO-X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Duplicate	AI-W-ES-07/ Appendix E
GS-N-DPT2	GS-N-DPT2-SO-0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal and MS/MSD	AI-W-ES-07/ Appendix E
GS-N-DPT2	GS-N-DPT2-SO- X-X	Soil Macro-core (Optional)	X to X	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal and MS/MSD	AI-W-ES-07/ Appendix E
GS-N-DPT2	GS-N-DPT2-SO- X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal and MS/MSD	AI-W-ES-07/ Appendix E
GS-N-DPT3	GS-N-DPT3-SO-0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-N-DPT3	GS-N-DPT3-SO- X-X	Soil Macro-core (Optional)	X to X	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E

Table 18-3. Subsurface Soil Sampling Locations and Methods/SOP Requirements						
Sample Location	**Sample ID[1]**	**Matrix**	**Depth Interval (ft)**	**Analytical Group**	**Sample Type**	**Sampling SOP / Laboratory SOP**
Northeastern Refuge Study Area						
GS-N-DPT3	GS-N-DPT3-SO- X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-N-DPT4	GS-N-DPT4-SO-0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-N-DPT4	GS-N-DPT4-SO-0-100	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Duplicate	AI-W-ES-07/ Appendix E
GS-N-DPT4	GS-N-DPT4-SO-X-X	Soil Macro-core (Optional)	X to X	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-N-DPT4	GS-N-DPT4-SO-X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-N-DPT5	GS-N-DPT5-SO-0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-N-DPT5	GS-N-DPT5-SO- SO-X-X	Soil Macro-core (Optional)	X to X	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-N-DPT5	GS-N-DPT5-SO- SO-X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-N-DPT6	GS-N-DPT6-SO-0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-N-DPT6	GS-N-DPT6- SO-X-X	Soil Macro-core (Optional)	X to X	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E

Table 18-3. Subsurface Soil Sampling Locations and Methods/SOP Requirements						
Sample Location	**Sample ID[1]**	**Matrix**	**Depth Interval (ft)**	**Analytical Group**	**Sample Type**	**Sampling SOP / Laboratory SOP**
Northeastern Refuge Study Area						
GS-N-DPT6	GS-N-DPT6- SO-X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-N-DPT7	GS-N-DPT7-SO-0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-N-DPT7	GS-N-DPT7- SO-X-X	Soil Macro-core (Optional)	X to X	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-N-DPT7	GS-N-DPT7- SO-X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-N-DPT8	GS-N-DPT8-SO-0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-N-DPT8	GS-N-DPT8- SO-X-X	Soil Macro-core (Optional)	X to X	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-N-DPT8	GS-N-DPT8- SO-X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-N-DPT9	GS-N-DPT9-SO-0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-N-DPT9	GS-N-DPT9-SO-X-X	Soil Macro-core (Optional)	X to X	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-N-DPT9	GS-N-DPT9-SO-X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E

Table 18-3. Subsurface Soil Sampling Locations and Methods/SOP Requirements						
Sample Location	**Sample ID[1]**	**Matrix**	**Depth Interval (ft)**	**Analytical Group**	**Sample Type**	**Sampling SOP / Laboratory SOP**
Northeastern Refuge Study Area						
GS-N-DPT10	GS-N-DPT10-SO-0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-N-DPT10	GS-N-DPT10-SO-X-X	Soil Macro-core (Optional)	X to X	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-N-DPT10	GS-N-DPT10-SO-X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E

BLTD – Below Landfill Total Depth
ft – foot
LTD – Landfill Total Depth
MS/MSD – Matrix Spike/Spike Duplicate
N – Northeastern Refuge Study Area
PCBs – Polychlorinated Biphenyls
SOP – Standard Operating Procedure
SVOCs – Semi-volatile Organic Compounds
TAL – Target Analyte List
TCLP - Toxicity Characteristic Leaching Procedure
VOCs - Volatile Organic Compounds
X – Depth to be determined in the field

Table 18-4. Subsurface Soil Sampling Locations and Methods/SOP Requirements						
Sample Location	**Sample ID[1]**	**Matrix**	**Depth Interval (ft)**	**Analytical Group**	**Sample Type**	**Sampling SOP / Laboratory SOP**
Southern Refuge Study Area						
GS-S-DPT11	GS-S-DPT11-SO--0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT11	GS-S-DPT101-SO--0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Duplicate	AI-W-ES-07/ Appendix E
GS-S-DPT11	GS-S-DPT11-SO-X-X	Soil Macro-core (Optional)	X to X	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT11	GS-S-DPT11-SO-X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT11	GS-S-DPT101-SO-X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Duplicate	AI-W-ES-07/ Appendix E
GS-S-DPT12	GS-S-DPT12-SO-0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal and MS/MSD	AI-W-ES-07/ Appendix E
GS-S-DPT12	GS-S-DPT12-SO-X-X	Soil Macro-core (Optional)	X to X	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal and MS/MSD	AI-W-ES-07/ Appendix E
GS-S-DPT12	GS-S-DPT12-SO-X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal and MS/MSD	AI-W-ES-07/ Appendix E
GS-S-DPT13	GS-S-DPT13-SO-0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT13	GS-S-DPT13-X-X	Soil Macro-core (Optional)	X to X	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT13	GS-S-DPT13-X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E

Table 18-4. Subsurface Soil Sampling Locations and Methods/SOP Requirements						
Sample Location	**Sample ID[1]**	**Matrix**	**Depth Interval (ft)**	**Analytical Group**	**Sample Type**	**Sampling SOP / Laboratory SOP**
Southern Refuge Study Area						
GS-S-DPT14	GS-S-DPT14-SO -0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT14	GS-S-DPT14-SO -X-X	Soil Macro-core (Optional)	X to X	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT14	GS-S-DPT14-SO -X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT15	GS-S-DPT15-SO -0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT15	GS-S-DPT15-SO -X-X	Soil Macro-core (Optional)	X to X	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT15	GS-S-DPT15-SO -X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT16	GS-S-DPT16-SO -0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT6	GS-S-DPT6-SO -X-X	Soil Macro-core (Optional)	X to X	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT6	GS-S-DPT6-SO -X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT7	GS-S-DPT7-SO -0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT7	GS-S-DPT7-SO -X-X	Soil Macro-core (Optional)	X to X	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E

Table 18-4. Subsurface Soil Sampling Locations and Methods/SOP Requirements						
Sample Location	**Sample ID[1]**	**Matrix**	**Depth Interval (ft)**	**Analytical Group**	**Sample Type**	**Sampling SOP / Laboratory SOP**
Southern Refuge Study Area						
GS-S-DPT7	GS-S-DPT7-SO -X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT18	GS-S-DPT18-SO -0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT18	GS-S-DPT18-SO -X-X	Soil Macro-core (Optional)	X to X	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT18	GS-S-DPT18-SO -X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT19	GS-S-DPT19-SO -0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT19	GS-S-DPT19-SO-X-X	Soil Macro-core (Optional)	X to X	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT19	GS-S-DPT19-SO-X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT20	GS-S-DPT120-SO-0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT20	GS-S-DPT20-SO-X-X	Soil Macro-core (Optional)	X to X	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT20	GS-S-DPT20-SO -X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT21	GS-S-DPT102-SO--0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Duplicate	AI-W-ES-07/ Appendix E

Table 18-4. Subsurface Soil Sampling Locations and Methods/SOP Requirements						
Sample Location	**Sample ID[1]**	**Matrix**	**Depth Interval (ft)**	**Analytical Group**	**Sample Type**	**Sampling SOP / Laboratory SOP**
Southern Refuge Study Area						
GS-S-DPT21	GS-S-DPT12-SO--0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT21	GS-S-DPT21-SO-X-X	Soil Macro-core (Optional)	X to X	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Duplicate	AI-W-ES-07/ Appendix E
GS-S-DPT21	GS-S-DPT21-SO-X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT22	GS-S-DPT22-SO-0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT22	GS-S-DPT22-SO-X-X	Soil Macro-core (Optional)	X to X	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT22	GS-S-DPT22-SO-X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT23	GS-S-DPT23-SO-0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT23	GS-S-DPT23-SO-X-X	Soil Macro-core (Optional)	X to X	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT23	GS-S-DPT23-SO-X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT24	GS-S-DPT24-SO-0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT24	GS-S-DPT24-SO-X-X	Soil Macro-core (Optional)	X to X	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E

Table 18-4. Subsurface Soil Sampling Locations and Methods/SOP Requirements						
Sample Location	**Sample ID[1]**	**Matrix**	**Depth Interval (ft)**	**Analytical Group**	**Sample Type**	**Sampling SOP / Laboratory SOP**
Southern Refuge Study Area						
GS-S-DPT24	GS-S-DPT24-SO-X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT25	GS-S-DPT25-SO-0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT25	GS-S-DPT25-SO-X-X	Soil Macro-core (Optional)	X to X	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT25	GS-S-DPT25-SO-X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT26	GS-S-DPT26-SO-0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT26	GS-S-DPT26-SO-X-X	Soil Macro-core (Optional)	X to X	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT26	GS-S-DPT26-SO-X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT27	GS-S-DPT27-SO-0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT27	GS-S-DPT27-SO-X-X	Soil Macro-core (Optional)	X to X	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT27	GS-S-DPT27-SO-X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT28	GS-S-DPT28-SO-0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E

Table 18-4. Subsurface Soil Sampling Locations and Methods/SOP Requirements						
Sample Location	**Sample ID[1]**	**Matrix**	**Depth Interval (ft)**	**Analytical Group**	**Sample Type**	**Sampling SOP / Laboratory SOP**
Southern Refuge Study Area						
GS-S-DPT28	GS-S-DPT28-SO-X-X	Soil Macro-core (Optional)	X to X	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT28	GS-S-DPT28-SO-X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT29	GS-S-DPT29-SO-0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT29	GS-S-DPT29-SO-X-X	Soil Macro-core (Optional)	X to X	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT29	GS-S-DPT29-SO-X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT30	GS-S-DPT30-SO-0-X	Soil Macro-core	0 to LTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT30	GS-S-DPT30-SO-X-X	Soil Macro-core (Optional)	X to X	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E
GS-S-DPT30	GS-S-DPT30-SO-X-X	Soil Macro-core	LTD to BLTD	VOCs, SVOCs, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans,	Normal	AI-W-ES-07/ Appendix E

BLTD – Below Landfill Total Depth
ft – foot
LTD – Landfill Total Depth
MS/MSD – Matrix Spike/Spike Duplicate
PCBs – Polychlorinated Biphenyls
SOP – Standard Operating Procedure
S – Southern Refuge Study Area
SVOCs – Semi-volatile Organic Compounds
TAL – Target Analyte List
TCLP - Toxicity Characteristic Leaching Procedure

VOCs - Volatile Organic Compounds
X – Depth to be determined in the field

Table 18-5. Pore water Sampling Locations and Methods/SOP Requirements						
Sample Location	**Sample ID[1]**	**Matrix**	**Depth Interval (ft)**	**Analytical Group**	**Sample Type**	**Sampling SOP / Laboratory SOP**
Northeastern Refuge Study Area						
GS-N-PW1	GS-N-PW1T-X-X GS-N-PW1D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal, MS/MSD	SESDPROC-513-R3/ Appendix E
GS-N-PW2	GS-N-PW2T-X-X GS-N-PW2D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-N-PW2	GS-N-PW100T-X-X GS-N-PW100D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Duplicate	SESDPROC-513-R3/ Appendix E
GS-N-PW3	GS-N-PW3T-X-X GS-N-PW3D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-N-PW4	GS-N-PW4T-X-X GS-N-PW4D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-N-PW5	GS-N-PW5T-X-X GS-N-PW5D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-N-PW6	GS-N-PW6T-X-X GS-N-PW6D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-N-PW7	GS-N-PW7T-X-X GS-N-PW7D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E

Table 18-5. Pore water Sampling Locations and Methods/SOP Requirements						
Sample Location	**Sample ID[1]**	**Matrix**	**Depth Interval (ft)**	**Analytical Group**	**Sample Type**	**Sampling SOP / Laboratory SOP**
Northeastern Refuge Study Area						
GS-N-PW8	GS-N-PW8T-X-X GS-N-PW8D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-N-PW9	GS-N-PW9T-X-X GS-N-PW9D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-N-PW10	GS-N-PW10T-X-X GS-N-PW10D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-N-PW11	GS-N-PW11T-X-X GS-N-PW11D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-N-PW12	GS-N-PW12T-X-X GS-N-PW12D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-N-PW12	GS-N-PW101T-X-X GS-N-PW101D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Duplicate	SESDPROC-513-R3/ Appendix E
GS-N-PW13	GS-N-PW13T-X-X GS-N-PW13D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-N-PW14	GS-N-PW14T-X-X GS-N-PW14D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E

Table 18-5. Pore water Sampling Locations and Methods/SOP Requirements						
Sample Location	**Sample ID[1]**	**Matrix**	**Depth Interval (ft)**	**Analytical Group**	**Sample Type**	**Sampling SOP / Laboratory SOP**
Northeastern Refuge Study Area						
GS-N-PW15	GS-N-PW15T-X-X GS-N-PW15D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-N-PW16	GS-N-PW16T-X-X GS-N-PW16D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-N-PW17	GS-N-PW17T-X-X GS-N-PW17D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-N-PW18	GS-N-PW18T-X-X GS-N-PW18D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-N-PW19	GS-N-PW19T-X-X GS-N-PW19D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-N-PW20	GS-N-PW20T-X-X GS-N-PW20D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-N-PW21	GS-N-PW21T-X-X GS-N-PW21D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal, MS/MSD	SESDPROC-513-R3/ Appendix E
GS-N-PW22	GS-N-PW22T-X-X GS-N-PW22D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E

Table 18-5. Pore water Sampling Locations and Methods/SOP Requirements						
Sample Location	**Sample ID[1]**	**Matrix**	**Depth Interval (ft)**	**Analytical Group**	**Sample Type**	**Sampling SOP / Laboratory SOP**
Northeastern Refuge Study Area						
GS-N-PW22	GS-N-PW102T-X-X GS-N-PW102D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Duplicate	SESDPROC-513-R3/ Appendix E
GS-N-PW23	GS-N-PW23T-X-X GS-N-PW23D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-N-PW24	GS-N-PW24T-X-X GS-N-PW24D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-N-PW25	GS-N-PW25T-X-X GS-N-PW25D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E

DOC – Dissolved Organic Carbon
MS/MSD – Matrix Spike/Spike Duplicate

N – Northeastern Refuge Study Area
PCBs – Polychlorinated Biphenyls
PW – Pore water
SOP – Standard Operating Procedure
S – Southern Refuge Study Area
SVOCs – Semi-volatile Organic Compounds
TAL – Target Analyte List
TCLP - Toxicity Characteristic Leaching Procedure
VOCs - Volatile Organic Compounds
X – Depth to be determined in the field

Table 18-6. Pore-water Sampling Locations and Methods/SOP Requirements						
Sample Location	**Sample ID[1]**	**Matrix**	**Depth Interval (ft)**	**Analytical Group**	**Sample Type**	**Sampling SOP / Laboratory SOP**
Southern Refuge Study Area						
GS-S-PW26	GS-S-PW26T-X-X GS-S-PW26D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-S-PW27	GS-S-PW27T-X-X GS-S-PW27D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-S-PW28	GS-S-PW28T-X-X GS-S-PW28D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-S-PW29	GS-S-PW29T-X-X GS-S-PW29D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-S-PW30	GS-S-PW30T-X-X GS-S-PW30D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-S-PW31	GS-S-PW31T-X-X GS-S-PW31D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-S-PW31	GS-S-PW103T-X-X GS-S-PW103D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Duplicate	SESDPROC-513-R3/ Appendix E
GS-S-PW32	GS-S-PW32-X-X GS-S-PW32D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E

Table 18-6. Pore-water Sampling Locations and Methods/SOP Requirements						
Sample Location	**Sample ID[1]**	**Matrix**	**Depth Interval (ft)**	**Analytical Group**	**Sample Type**	**Sampling SOP / Laboratory SOP**
Southern Refuge Study Area						
GS-S-PW33	GS-S-PW33T-X-X GS-S-PW33D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-S-PW34	GS-S-PW34T-X-X GS-S-PW34D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-S-PW35	GS-S-PW35T-X-X GS-S-PW35D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-S-PW36	GS-S-PW36T-X-X GS-S-PW36D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-S-PW37	GS-S-PW37T-X-X GS-S-PW37D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-S-PW38	GS-S-PW38-X-X GS-S-PW38D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal,	SESDPROC-513-R3/ Appendix E
GS-S-PW39	GS-S-PW39T-X-X GS-S-PW39D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-S-PW40	GS-S-PW40T-X-X GS-S-PW40D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal, MS/MSD	SESDPROC-513-R3/ Appendix E

Table 18-6. Pore-water Sampling Locations and Methods/SOP Requirements						
Sample Location	**Sample ID[1]**	**Matrix**	**Depth Interval (ft)**	**Analytical Group**	**Sample Type**	**Sampling SOP / Laboratory SOP**
Southern Refuge Study Area						
GS-S-PW41	GS-S-PW41T-X-X GS-S-PW41D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-S-PW41	GS-S-PW104T-X-X GS-S-PW402D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Duplicate	SESDPROC-513-R3/ Appendix E
GS-S-PW42	GS-S-PW42T-X-X GS-S-PW42D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-S-PW43	GS-S-PW43T-X-X GS-S-PW43D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-S-PW44	GS-S-PW44T-X-X GS-S-PW44D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-S-PW45	GS-S-PW45T-X-X GS-S-PW45D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-S-PW46	GS-S-PW46T-X-X GS-S-PW46D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-S-PW47	GS-S-PW47T-X-X GS-S-PW47D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E

Table 18-6. Pore-water Sampling Locations and Methods/SOP Requirements						
Sample Location	**Sample ID[1]**	**Matrix**	**Depth Interval (ft)**	**Analytical Group**	**Sample Type**	**Sampling SOP / Laboratory SOP**
Southern Refuge Study Area						
GS-S-PW48	GS-S-PW48T-X-X GS-S-PW48D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-S-PW49	GS-S-PW49T-X-X GS-S-PW49D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-S-PW50	GS-S-PW50T-X-X GS-S-PW50D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-S-PW50	GS-S-PW105T-X-X GS-S-PW105D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Duplicate	SESDPROC-513-R3/ Appendix E
GS-S-PW51	GS-S-PW51T-X-X GS-S-PW51D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-S-PW52	GS-S-PW52T-X-X GS-S-PW52D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-S-PW53	GS-S-PW53T-X-X GS-S-PW53D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E
GS-S-PW54	GS-S-PW54T-X-X GS-S-PW54D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E

Table 18-6. Pore-water Sampling Locations and Methods/SOP Requirements						
Sample Location	**Sample ID[1]**	**Matrix**	**Depth Interval (ft)**	**Analytical Group**	**Sample Type**	**Sampling SOP / Laboratory SOP**
Southern Refuge Study Area						
GS-S-PW55	GS-S-PW55T-X-X GS-S-PW55D-X-X	Pore Water	X to X	Hach Kit (As, Mn, Fe, Pb); Laboratory analysis for: VOCs, SVOCs, TOC, Pesticides, DOC, alkalinity, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	SESDPROC-513-R3/ Appendix E

DOC – Dissolved Organic Carbon
MS/MSD – Matrix Spike/Spike Duplicate
PCBs – Polychlorinated Biphenyls
PW – Pore water
SOP – Standard Operating Procedure
S – Southern Refuge Study Area
SVOCs – Semi-volatile Organic Compounds
TAL – Target Analyte List
TCLP - Toxicity Characteristic Leaching Procedure
VOCs - Volatile Organic Compounds
X – Depth to be determined in the field

Table 18-7 Surface Water Sampling Locations and Methods/SOP Requirements						
Sample Location	**Sample ID[1]**	**Matrix**	**Water Depth (ft)**	**Analytical Group**	**Sample Type**	**Sampling SOP / Laboratory SOP**
Northeast Refuge Study Area						
GS-N-SWSD1	GS-N-SW3T-1 GS-N-SW3D-1	Surface Water	X	SVOCs, Pesticides, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	AI-W-ES-03/ Appendix E
GS-N-SWSD1	GS-N-SW300T-1 GS-N-SW300D-1	Surface Water	X	SVOCs, Pesticides, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Duplicate	AI-W-ES-03/ Appendix E
GS-N-SWSD2	GS-N-SW3T -1 GS-N-SW3D -1	Surface Water	X	SVOCs, Pesticides, hardness, TAL Metals & Cyanide (filtered and unfiltered)	MS/MSD	AI-W-ES-03/ Appendix E
GS-N-SWSD3	GS-N-SW3T-1 GS-N-SW3D -1	Surface Water	X	SVOCs, Pesticides, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	AI-W-ES-03/ Appendix E
GS-N-SWSD4	GS-NSW4T-1 GS-N-SW4D -1	Surface Water	X	SVOCs, Pesticides, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	AI-W-ES-03/ Appendix E
GS-N-SWSD5	GS-N-SW5T-1 GS-N-SW5D -1	Surface Water	X	SVOCs, Pesticides, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	AI-W-ES-03/ Appendix E

DOC – Dissolved Organic Carbon
FWS – US Fish and Wildlife
MS/MSD – Matrix Spike/Spike Duplicate
PCBs – Polychlorinated Biphenyls
SOP – Standard Operating Procedure
SVOCs – Semi-volatile Organic Compounds
SWSD – Surface water, sediment sampling location
SW – Surface water
TAL – Target Analyte List
TCLP - Toxicity Characteristic Leaching Procedure
VOCs - Volatile Organic Compounds
X – Depth to be determined in the field

Table 18-8 Surface Water Sampling Locations and Methods/SOP Requirements						
Sample Location	**Sample ID[1]**	**Matrix**	**Water Depth (ft)**	**Analytical Group**	**Sample Type**	**Sampling SOP / Laboratory SOP**
Southern Refuge Study Area						
GS-S-SWSD6	GS-S-SW6T-1 GS-S-SW6D-1	Surface Water	X	SVOCs, Pesticides, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	AI-W-ES-03/ Appendix E
GS-S-SWSD7	GS-S-SW7T -1 GS-S-SW7D -1	Surface Water	X	SVOCs, Pesticides, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	ES-02 / GCMSV-003
GS-S-SWSD8	GS-S-SW8T-1 GS-S-SW8D -1	Surface Water	X	SVOCs, Pesticides, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	ES-02 / GCMSV-003
GS-S-SWSD9	GS-S-SW9T-1 GS-S-SW9D -1	Surface Water	X	SVOCs, Pesticides, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	ES-02 / GCMSV-003
GS-S-SWSD10	GS-S-SW30T-1 GS-S-SW30D -1	Surface Water	X	SVOCs, Pesticides, hardness, TAL Metals & Cyanide (filtered and unfiltered)	Normal	ES-02 / GCMSV-003

DOC – Dissolved Organic Carbon
FWS – US Fish and Wildlife
MS/MSD – Matrix Spike/Spike Duplicate
PCBs – Polychlorinated Biphenyls
SOP – Standard Operating Procedure
S – Southern Refuge Study Area
SVOCs – Semi-volatile Organic Compounds
SWSD – Surface water, sediment sampling location
SW – Surface water
TAL – Target Analyte List
TCLP - Toxicity Characteristic Leaching Procedure
VOCs - Volatile Organic Compounds
X – Depth to be determined in the field

Table 18-9 Sediment Sampling Locations and Methods/SOP Requirements						
Sample Location	**Sample ID[1]**	**Matrix**	**Water Depth (ft)**	**Analytical Group**	**Sample Type**	**Sampling SOP / Laboratory SOP**
Northeast Refuge Study Area						
GS-N-SWSD1	GS-N-SD1-1	Sediment	X	VOCs, SVOCs, Pesticides, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans, Bioavailability parameters: TOC; Grain size, pH, ORP, AVS/SEM, Moisture content.	Normal	AI-W-ES-10/ Appendix E
GS-N-SWSD1	GS-N-SD100-1	Sediment	X	VOCs, SVOCs, Pesticides, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans, Bioavailability parameters: TOC; Grain size, pH, ORP, AVS/SEM, Moisture content.	Duplicate	AI-W-ES-10/ Appendix E
GS-N-SWSD2	GS-N-SD2-1	Sediment	X	VOCs, SVOCs, Pesticides, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans, Bioavailability parameters: TOC; Grain size, pH, ORP, AVS/SEM, Moisture content.	Normal, MS/MSD	AI-W-ES-10/ Appendix E
GS-N-SWSD3	GS-N-SD3-1	Sediment	X	VOCs, SVOCs, Pesticides, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans, Bioavailability parameters: TOC; Grain size, pH, ORP, AVS/SEM, Moisture content.	Normal	AI-W-ES-10/ Appendix E
GS-N-SWSD4	GS-N-SD4-1	Sediment	X	VOCs, SVOCs, Pesticides, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans, Bioavailability parameters: TOC; Grain size, pH, ORP, AVS/SEM, Moisture content.	Normal	AI-W-ES-10/ Appendix E
GS-N-SWSD5	GS-N-SD5-1	Sediment	X	VOCs, SVOCs, Pesticides, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans, Bioavailability parameters: TOC; Grain size, pH, ORP, AVS/SEM, Moisture content.	Normal	AI-W-ES-10/ Appendix E

Table 18-9 Sediment Sampling Locations and Methods/SOP Requirements						
Sample Location	**Sample ID[1]**	**Matrix**	**Water Depth (ft)**	**Analytical Group**	**Sample Type**	**Sampling SOP / Laboratory SOP**
Northeast Refuge Study Area						
GS-N-SWSD6	GS-N-SD6-1	Sediment	X	VOCs, SVOCs, Pesticides, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans, Bioavailability parameters: TOC; Grain size, pH, ORP, AVS/SEM, Moisture content.	Normal	AI-W-ES-10/ Appendix E
GS-N-SWSD7	GS-N-SD7-1	Sediment	X	VOCs, SVOCs, Pesticides, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans, Bioavailability parameters: TOC; Grain size, pH, ORP, AVS/SEM, Moisture content.	Normal	AI-W-ES-10/ Appendix E
GS-N-SWSD8	GS-N-SD8-1	Sediment	X	VOCs, SVOCs, Pesticides, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans, Bioavailability parameters: TOC; Grain size, pH, ORP, AVS/SEM, Moisture content.	Normal	AI-W-ES-10/ Appendix E
GS-N-SWSD9	GS-N-SD9-1	Sediment	X	VOCs, SVOCs, Pesticides, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans, Bioavailability parameters: TOC; Grain size, pH, ORP, AVS/SEM, Moisture content.	Normal	AI-W-ES-10/ Appendix E
GS-N-SWSD10	GS-N-SD10-1	Sediment	X	VOCs, SVOCs, Pesticides, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans, Bioavailability parameters: TOC; Grain size, pH, ORP, AVS/SEM, Moisture content.	Normal	AI-W-ES-10/ Appendix E

AVS/SEM – acid-volatile sulfides/simultaneously extracted metals
DOC – Dissolved Organic Carbon
FWS – US Fish and Wildlife
MS/MSD – Matrix Spike/Spike Duplicate
ORP – Oxidation Reduction Potential
PCBs – Polychlorinated Biphenyls
pH – Potential hydrogen scale for acidity to alkalinity
SOP – Standard Operating Procedure
N – Northern Refuge Study Area

SVOCs – Semi-volatile Organic Compounds
SD – Sediment
SWSD – Surface water, sediment sampling location
TAL – Target Analyte List
TCLP - Toxicity Characteristic Leaching Procedure
TOC – Total Organic Carbon
VOCs - Volatile Organic Compounds
X – Depth to be determined in the field
VOCs - Volatile Organic Compounds
X – Depth to be determined in the field

Table 18-10. Sediment Sampling Locations and Methods/SOP Requirements						
Sample Location	**Sample ID[1]**	**Matrix**	**Water Depth (ft)**	**Analytical Group**	**Sample Type**	**Sampling SOP / Laboratory SOP**
Southern Refuge Study Area						
GS-S-SWSD11	GS-S-SD11-1	Sediment	X	VOCs, SVOCs, Pesticides, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans, Bioavailability parameters: TOC; Grain size, pH, ORP, AVS/SEM, Moisture content.	Normal	AI-W-ES-10/ Appendix E
GS-S-SWSD11	GS-S-SD101-1	Sediment	X	VOCs, SVOCs, Pesticides, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans, Bioavailability parameters: TOC; Grain size, pH, ORP, AVS/SEM, Moisture content.	Duplicate	AI-W-ES-10/ Appendix E
GS-S-SWSD12	GS-S-SD12-1	Sediment	X	VOCs, SVOCs, Pesticides, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans, Bioavailability parameters: TOC; Grain size, pH, ORP, AVS/SEM, Moisture content.	Normal	AI-W-ES-10/ Appendix E
GS-S-SWSD13	GS-S-SD13-1	Sediment	X	VOCs, SVOCs, Pesticides, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans, Bioavailability parameters: TOC; Grain size, pH, ORP, AVS/SEM, Moisture content.	Normal	AI-W-ES-10/ Appendix E
GS-S-SWSD14	GS-S-SD14-1	Sediment	X	VOCs, SVOCs, Pesticides, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans, Bioavailability parameters: TOC; Grain size, pH, ORP, AVS/SEM, Moisture content.	Normal	AI-W-ES-10/ Appendix E
GS-S-SWSD15	GS-S-SD15-1	Sediment	X	VOCs, SVOCs, Pesticides, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans, Bioavailability parameters: TOC; Grain size, pH, ORP, AVS/SEM, Moisture content.	Normal	AI-W-ES-10/ Appendix E
GS-S-SWSD16	GS-S-SD16-1	Sediment	X	VOCs, SVOCs, Pesticides, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans, Bioavailability parameters: TOC; Grain size, pH, ORP, AVS/SEM, Moisture content.	Normal	AI-W-ES-10/ Appendix E

Table 18-10. Sediment Sampling Locations and Methods/SOP Requirements						
Sample Location	**Sample ID[1]**	**Matrix**	**Water Depth (ft)**	**Analytical Group**	**Sample Type**	**Sampling SOP / Laboratory SOP**
Southern Refuge Study Area						
GS-S-SWSD17	GS-S-SD17-1	Sediment	X	VOCs, SVOCs, Pesticides, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans, Bioavailability parameters: TOC; Grain size, pH, ORP, AVS/SEM, Moisture content.	Normal	AI-W-ES-10/ Appendix E
GS-S-SWSD18	GS-S-SD8-1	Sediment	X	VOCs, SVOCs, Pesticides, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans, Bioavailability parameters: TOC; Grain size, pH, ORP, AVS/SEM, Moisture content.	Normal	AI-W-ES-10/ Appendix E
GS-S-SWSD19	GS-S-SD9-1	Sediment	X	VOCs, SVOCs, Pesticides, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans, Bioavailability parameters: TOC; Grain size, pH, ORP, AVS/SEM, Moisture content.	Normal	AI-W-ES-10/ Appendix E
GS-S-SWSD20	GS-S-SD20-1	Sediment	X	VOCs, SVOCs, Pesticides, PCBs (as Aroclors), TAL Metals & Cyanide If PCBs identified other than Aroclors: analyze PCB (as Congeners), Dioxins, Furans, Bioavailability parameters: TOC; Grain size, pH, ORP, AVS/SEM, Moisture content.	Normal,	AI-W-ES-10/ Appendix E

AVS/SEM – acid-volatile sulfides/simultaneously extracted metals
DOC – Dissolved Organic Carbon
FWS – US Fish and Wildlife
MS/MSD – Matrix Spike/Spike Duplicate
ORP – Oxidation Reduction Potential
PCBs – Polychlorinated Biphenyls
pH – Potential hydrogen scale for acidity to alkalinity
SOP – Standard Operating Procedure
S – Southern Refuge Study Area
SVOCs – Semi-volatile Organic Compounds
SD – Sediment
SWSD – Surface water, sediment sampling location
TAL – Target Analyte List
TCLP - Toxicity Characteristic Leaching Procedure
TOC – Total Organic Carbon
VOCs - Volatile Organic Compounds
X – Depth to be determined in the field

Table 18-11. Geotechnical Sampling Locations and Methods/SOP Requirements						
Sample Location	**Sample ID[1]**	**Matrix**	**Depth Interval (ft)**	**Analytical Group**	**Sample Type**	**Sampling SOP / Laborator**
Northeastern Refuge Study Area						
GS-N-GT1	GS-N-GT1-X-X	Underlying Clay	X to X	ASTM D698; ASTM D2216; ASTM D5084, ASTM D2435, ASTM D2850; ASTM D7263 ASTM-D4318	Normal	AI-W-ES-09/ Appendix E
GS-N-GT2	GS-N-GT2-X-X	Underlying Clay	X to X	ASTM D698; ASTM D2216; ASTM D5084, ASTM D2435, ASTM D2850; ASTM D7263 ASTM-D4318	Normal	AI-W-ES-09/ Appendix E
GS-N-GT3	GS-N-GT3-X-X	Underlying Clay	X to X	ASTM D698; ASTM D2216; ASTM D5084, ASTM D2435, ASTM D2850; ASTM D7263 ASTM-D4318	Normal	AI-W-ES-09/ Appendix E
GS-N-GT4	GS-N-GT4-X-X	Underlying Clay	X to X	ASTM D698; ASTM D2216; ASTM D5084, ASTM D2435, ASTM D2850; ASTM D7263 ASTM-D4318	Normal	AI-W-ES-09/ Appendix E
GS-N-GT5	GS-N-GT5-X-X	Underlying Clay	X to X	ASTM D698; ASTM D2216; ASTM D5084, ASTM D2435, ASTM D2850; ASTM D7263 ASTM-D4318	Normal	AI-W-ES-09/ Appendix E
Southern Refuge Study Area						
GS-N-GT6	GS-S-GT6-X-X	Underlying Clay	X to X	ASTM D698; ASTM D2216; ASTM D5084, ASTM D2435, ASTM D2850; ASTM D7263 ASTM-D4318	Normal	AI-W-ES-09/ Appendix E
GS-N-GT7	GS-S-GT7-X-X	Underlying Clay	X to X	ASTM D698; ASTM D2216; ASTM D5084, ASTM D2435, ASTM D2850; ASTM D7263 ASTM-D4318	Normal	AI-W-ES-09/ Appendix E
GS-N-GT8	GS-S-GT8-X-X	Underlying Clay	X to X	ASTM D698; ASTM D2216; ASTM D5084, ASTM D2435, ASTM D2850; ASTM D7263 ASTM-D4318	Normal	AI-W-ES-09/ Appendix E
GS-N-GT9	GS-S-GT9-X-X	Underlying Clay	X to X	ASTM D698; ASTM D2216; ASTM D5084, ASTM D2435, ASTM D2850; ASTM D7263 ASTM-D4318	Normal	AI-W-ES-09/ Appendix E
GS-N-GT10	GS-S-GT10-X-X	Underlying Clay	X to X	ASTM D698; ASTM D2216; ASTM D5084, ASTM D2435, ASTM D2850; ASTM D7263 ASTM-D4318	Normal	AI-W-ES-09/ Appendix E

ASTM D698 Standard Test Method for Compaction
ASTM D 2216 Standard Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass
ASTM D2434 Standard Test Method for Permeability of Granular Soils (Constant Head)
ASTM D2435 Standard Test Method for One-Dimensional Consolidation Properties of Saturated Cohesive Soil
ASTM D2850 Triaxial Stress with Pore Pressure
ASTM D 4318 Standard Test Methods for Liquid Limit, Plastic limit, and Plasticity Index of Soils
ASTM D7263 Standard Test Methods for Laboratory Determination of Density (Unit Weight) of Soil Specimens.

19. QAPP WORKSHEET #19 – ANALYTICAL SOP REQUIREMENTS

This worksheet summarizes the sample containers, preservation requirements, and holding times for each analytical group.

Table 19-1. Analytical SOP Requirements Table (Soil Samples)

Matrix	Analytical Group	Analytical and Preparation Method/ Lab SOP Reference[1]	Analytical Containers (number, size, type)	Sample Volume Per Container	Sample Preservation Requirements (chemical, temperature, light protected)	Sample Maximum Hold Time (preparation/analysis)
Soil	VOCs	SW-846 8260D/5035B	3 x Terra Core™ samplers, plus 1 x 2 oz. jar for moisture[2]	5 grams	Cool to 4 ± 2 C DI water or Na2S2O3	48 hours for preservation/14 days for analysis
Soil	SVOCs	SW-846 8270E/3546	2 x 4 oz. glass jar	30 grams	Cool to 4 ± 2 C	14 days until extraction/40 days for analysis
Soil	Pesticides	SW846 8081B/3546	1x 4-ounce (oz) glass jar with Teflon®- lined lid	30 grams	Cool to 4 ± 2 C	14 days until extraction/40 days for analysis
Soil	PCBs	SW846 8082A/3546	1x 4-ounce (oz) glass jar with Teflon®- lined lid	30 grams	Cool to 4 ± 2 C	14 days until extraction/40 days for analysis
Soil	TAL Metals	SW 846 6020B/7471B/3050B	1x 4-ounce (oz) glass jar with Teflon®- lined lid	30 grams	Cool to 4 ± 2 C	180 day for 6020C/28 Days 7471B
Soil	Dioxins/Furans	SW 846 8290A/8290_P_SOX	1x 8-ounce glass jar with Teflon®-lined lid or stainless steel liner	60 grams	Cool to 4 ± 2 C	30 days for preparation, 45 days for analysis
Soil	PCBs (Congeners)	Method 1668A / WS-ID-0013 Preparation:WS-IDP-0013	1x 8-ounce glass jar with Teflon®-lined lid or stainless steel liner	30 grams	Cool to 4 ± 2 C	360 days until extraction/365 days for analysis
Soil	TAL Metals	Simultaneously Extracted Metals (SEM) by Acid Volatile Sulfide (AVS)/SEM and EPA Method 6010C	TBD	TBD	Cool to 4 ± 2 C	TBD

Soil	pH	EPA Method 9045D	1x 4-ounce (oz) glass jar with Teflon®- lined lid	30 grams	Cool to 4 ± 2 C	Analyze immediately.
Soil	Total Organic Carbon (TOC)	Lloyd Kahn and EPA Method 9060A	1x 4-ounce (oz) glass jar with Teflon®- lined lid	30 grams	Cool to 4 ± 2 C	14 days for analysis.
IDW Soil	RCRA VOCs Pest/PCB/Dioxin-Furan/TAL Metals	SW-846 1311/8260B	2 x 4 oz. jars	25 grams	Cool to 4 ± 2 C	14 days until TCLP extraction/14 days for analysis
IDW Soil	RCRA SVOCs	SW-846 1311/8270D	2 x 4 oz. jars	100 grams	Cool to 4 ± 2 C	14 days until TCLP extraction/7 days until preparative extraction/40 days for analysis
Clay Layer	Geotechnical Analysis	ASTM D698	4 - 2.8 x 24 inch Shelby Tubes	0.25 ft^3	Handle carefully	Keep intact, hand deliver.
Clay Layer	Geotechnical Analysis	ASTM D2850	1 - 2.8 x 24 inch Shelby Tubes	0.05 ft^3	Handle carefully	Keep intact, hand deliver.
Clay Layer	Geotechnical Analysis	ASTM D2216, ASTM D2434, ASTM D4318, ASTM D7263, ASTM D5084	1 – 0.5 Gal Ziplock Bag	0.5 gallons	None	None

[1] Laboratory SOPs are listed on Worksheet #23.
[2] If VOCs is the only analysis requested, a separate 2 oz. jar will be submitted for percent moisture analysis.
°C – Degrees Celsius
< – Less Than
ASTM – American Society for Testing and Materials
DI – Deionized
IDW – Investigation Derived Waste
oz – ounce
PCB – Polychlorinated Biphenyls
RCRA – Resource Conservation and Recovery Act
SIM – Selective Ion Monitoring
SOP – Standard Operating Procedure/Practice
SVOC- Semi-volatile Organic Compound
SW-846 – Test Methods for Evaluating Solid Waste, Physical/Chemical Methods
TAL – Target Analyte List
TCLP- Toxicity Characteristic Leaching Procedure
VOC – Volatile Organic Compound

Table 19-2. Analytical SOP Requirements Table (Water Samples)					
Matrix	**Analytical Group**	**Analytical and Preparation Method/ Lab SOP Reference[1]**	**Analytical Containers (number, size, type)**	**Sample Preservation Requirements (chemical, temperature, light protected)**	**Sample Maximum Hold Time (preparation/analysis)**
Water	VOCs	SW-846 8260D/5035B	3 x 40-mL vials	No headspace; HCl to pH <2; Cool to 4 ± 2 C	14 days for analysis
Water	SVOCs	SW-846 8270E/3510C	2 x 250 ml amber bottles	Cool to 4 ± 2 C	7 days until extraction/40 days for analysis
Water	Pesticides	SW846 8081B/3510C	2 x 250 ml amber bottles	Cool to 4 ± 2 C	7 days until extraction/40 days for analysis
Water	PCBs	SW846 8082A/3510C	2 x 250 ml amber bottles	Cool to 4 ± 2 C	7 days until extraction/40 days for analysis
Water	TAL Metals	SW 846 6020B/3010A	1 x 500-mL high density polyethylene (HDPE)	HNO_3 to pH <2; Cool to 4 ± 2 C	180 day for analysis
	Mercury LL	SW846 7470A	1 x 500-mL high density polyethylene (HDPE)	HNO_3 to pH <2; Cool to <6°C	Preserved sample – 28 days
Water	Dioxins/Furans	SW 846 8290A/8290_P_SOX	2 x 1-liter (L) amber bottles	Cool to 4 ± 2 C	30 days for preparation, 45 days for analysis
Water	PCBs (Congeners)	Method 1668A / WS-ID-0013 Preparation:WS-IDP-0013	1 x 1-liter (L) amber bottles	Cool to 4 ± 2 C	365 days until extraction/365 days for analysis
Water	PFAs	Method 533	1 x 1-liter (L) (HDPE)	Cool to 4 ± 2 C	TBD

[1] Laboratory SOPs are listed on Worksheet #23.
°C – Degrees Celsius
< – Less than
H_2SO_4 – Sulfuric Acid
HCl – Hydrochloric Acid
IDW – Investigation Derived Waste

L – Liter
MCAWW – Methods for Chemical Analysis of Water and Wastes
mL – milliliter
°C – Degrees Celsius
< – Less Than
PCB – Polychlorinated Biphenyls
PFAs - Per- and Polyfluoroalkyl Substances
SOP – Standard Operating Procedure/Practice
SVOC- Semi-volatile Organic Compound
SW-846 – Test Methods for Evaluating Solid Waste, Physical/Chemical Methods
TAL – Target Analyte List
TCLP- Toxicity Characteristic Leaching Procedure
VOC – Volatile Organic Compound

20. QAPP WORKSHEET #20 – FIELD QUALITY CONTROL SAMPLE SUMMARY

This worksheet summarizes the field QC samples to be collected and their collection frequency. The frequency of field QC samples is calculated with respect to each site, and not a percent of the total number of samples collected

Table 20-1 Field Quality Control Summary [1]

Matrix	Analytical Group	No. of Normal Samples	No. of Field Duplicates	No. of Equipment Blanks	No. of MS/MSDs	No. of Trip Blanks	No. of Temperature Blanks	Total No. of Samples
Sampling Frequency:			Minimum 10%	1 per day per site	Minimum 5%	1 per cooler with VOCs	1 per cooler	N/A
Surface Soil	VOCs	30	3	1	2	2	N/A	36
Surface Soil	SVOCs	30	3	1	2	N/A	N/A	34
Surface Soil	Pesticides	30	3	1	2	N/A	N/A	34
Surface Soil	PCBs (Aroclor)	30	3	1	2	N/A	N/A	34
Surface Soil	TAL Metals/Mercury/ Cyanide	30	3	1	2	N/A	N/A	34
Surface Soil	Dioxin/Furans	10	1	1	1	N/A	N/A	12
Surface Soil	PCBs (Congeners)	10	1	1	1	N/A	N/A	12
Subsurface Soil	VOCs	90	9	8	5	10	N/A	117
Subsurface Soil	SVOCs	90	9	8	5	N/A	N/A	107
Subsurface Soil	Pesticides	90	9	8	5	N/A	N/A	107
Subsurface Soil	PCBs (Aroclor)	90	9	8	5	N/A	N/A	107
Subsurface Soil	TAL Metals/Mercury/ Cyanide	90	9	8	5	N/A	N/A	107
Subsurface Soil	Dioxin/Furans	30	3	8	2	N/A	N/A	41
Subsurface Soil	PCBs (Congeners)	30	3	8	2	N/A	N/A	41
Pore Water	VOCs (Total)	55	6	10	3	6	N/A	77
Pore Water	SVOCs (Total)	55	6	10	3	N/A	N/A	71
Pore Water	Pesticides (Total)	55	6	10	3	N/A	N/A	71
Pore Water	PCBs (Aroclor) (Total)	55	6	10	3	N/A	N/A	71
Pore Water	TAL Metals/Mercury/ Cyanide (Total)	55	6	10	3	N/A	N/A	71
Pore Water	TAL Metals/Mercury/ Cyanide (Dissolved)	55	6	10	3	N/A	N/A	71

Table 20-1 Field Quality Control Summary [1]								
Matrix	**Analytical Group**	**No. of Normal Samples**	**No. of Field Duplicates**	**No. of Equipment Blanks**	**No. of MS/MSDs**	**No. of Trip Blanks**	**No. of Temperature Blanks**	**Total No. of Samples**
Sampling Frequency:			Minimum 10%	1 per day per site	Minimum 5%	1 per cooler with VOCs	1 per cooler	N/A
Pore Water	PFAs	5	1	1	1	N/A	N/A	7
Pore Water	DOC, TOC, Alkalinity, Hardness	30	NA	NA	NA	N/A	N/A	30
Surface Water	VOCs (Total)	10	1	N/A	1	2	N/A	13
Surface Water	SVOCs (Total)	10	1	N/A	1	N/A	N/A	11
Surface Water	Pesticides (Total)	10	1	N/A	1	N/A	N/A	11
Surface Water	TAL Metals/Mercury /Cyanide(Total)	10	1	N/A	1	N/A	N/A	11
Surface Water	TAL Metals/Mercury /Cyanide(Dissolved)	10	1	N/A	1	N/A	N/A	11
Sediment	VOCs	20	2	N/A	1	2	N/A	24
Sediment	SVOCs	20	2	N/A	1	N/A	N/A	22
Sediment	Pesticides	20	2	N/A	1	N/A	N/A	22
Sediment	PCBs (Aroclor)	20	2	N/A	1	N/A	N/A	22
Sediment	TAL Metals/Mercury/ Cyanide	20	2	N/A	1	N/A	N/A	22
Sediment	Dioxin/Furans	7	1	N/A	1	N/A	N/A	8
Sediment	PCBs (Congeners)	7	1	N/A	1	N/A	N/A	8
Geotechnical Clay	ASTM D698; ASTM D2216; ASTM D5084, ASTM D2435, ASTM D2850; ASTM D7263 ASTM-D4318	10	N/A	N/A	N/A	N/A	N/A	10

[1] The number of sampling locations is subject to change based on changing field conditions.
MS/MSD – Matrix Spike/Matrix Spike Duplicate
N/A – Not applicable
No. – Number
PCBs – Polychlorinated Biphenyls
PFAs - Per- and Polyfluoroalkyl Substances
SVOCs – semi-volatile organic compounds

TAL – Target Analyte List
VOCs – Volatile Organic Compounds

21. QAPP WORKSHEET #21 – PROJECT SAMPLING SOPREFERENCE

This worksheet summarizes the SOPs used for sampling and other field activities that may occur on the project.

Table 21-1. Project Sampling SOP References

Reference Number[1]	Title, Revision Date and/or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	SOP Location
FO-01	Geologic Standards SOP	AI SOPs and FWS Investigation Guidance[2]	See SOP	N	Appendix B Section 1.1
FO-02	Site Reconnaissance, Preparation, and Restoration SOP	AI SOPs and FWS Investigation Guidance[2]	See SOP	N	Appendix B Section 1.2
FO-03	Utilities Location SOP	AI SOPs and FWS Investigation Guidance[2]	See SOP	N	Appendix B Section 1.3
FO-06	General Drilling SOP	AI SOPs and FWS Investigation Guidance[2]	See SOP	N	Appendix B Section 1.6
FO-07	Lithologic Logging SOP	AI SOPs and FWS Investigation Guidance[2]	See SOP	N	Appendix B Section 1.7
FO-08	Borehole Abandonment SOP	AI SOPs and FWS Investigation Guidance[2]	See SOP	N	Appendix B Section 1.8
FO-09	Hollow-Stem Auger Drilling and Sampling SOP	AI SOPs and FWS Investigation Guidance[2]	See SOP	N	Appendix B Section 1.9
FO-10	Direct Push Drilling and Sampling SOP	AI SOPs and FWS Investigation Guidance[2]	See SOP	N	Appendix B Section 1.10
FO-15	Test Pit Excavation SOP	AI SOPs and FWS Investigation Guidance[2]	See SOP	N	Appendix B Section 1.15
FO-16	Equipment Decontamination SOP	AI SOPs and FWS Investigation Guidance[2]	See SOP	N	Appendix b Section 1.16
FO-17	Waste Handling SOP	AI SOPs and FWS Investigation Guidance[2]	See SOP	N	Appendix b Section 1.17
FO-18	Surveying SOP	AI SOPs and FWS Investigation Guidance[2]	See SOP	N	Appendix B Section 1.18

Table 21-1. Project Sampling SOP References					
Reference Number[1]	Title, Revision Date and/or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	SOP Location
ES-01	General Water Sampling SOP	AI SOPs and FWS Investigation Guidance[2]	See SOP	N	Appendix B Section 2.1
ES-03	Surface Water Sampling SOP	AI SOPs and FWS Investigation Guidance[2]	See SOP	N	Appendix B Section 2.3
ES-04	Method 5035 Sampling for VOCs in Soil and Sediments SOP	AI SOPs and FWS Investigation Guidance[2]	See SOP	N	Appendix B Section 2.4
ES-05	Soil Sampling SOP	AI SOPs and FWS Investigation Guidance[2]	See SOP	N	Appendix B Section 2.5
ES-06	Subsurface Soil Sampling SOP	AI SOPs and FWS Investigation Guidance[2]	See SOP	N	Appendix B Section 2.6
ES-07	Field Headspace Screening SOP	AI SOPs and FWS Investigation Guidance[2]	See SOP	N	Appendix B Section 2.8
ES-08	Geotechnical Sampling SOP	AI SOPs and FWS Investigation Guidance[2]	See SOP	N	Appendix B Section 2.9
ES-9	Sediment Sampling SOP	AI SOPs and FWS Investigation Guidance[2]	See SOP	N	Appendix B Section 2.10
ES-10	Sample Handling SOP	AI SOPs and FWS Investigation Guidance[2]	See SOP	N	Appendix B Section 2.11
ES-11	Field Quality Control Samples SOP	AI SOPs and FWS Investigation Guidance[2]	See SOP	N	Appendix B Section 2.12
FM-01	General Field Measurement Parameter SOP	AI SOPs and FWS Investigation Guidance[2]	See SOP	N	Appendix B Section 3.1
FM-02	Field Equipment Calibration and Quality Control SOP	AI SOPs and FWS Investigation Guidance[2]	See SOP	N	Appendix B Section 3.2
FM-03	Equipment Maintenance and Decontamination SOP	AI SOPs and FWS Investigation Guidance[2]	See SOP	N	Appendix B Section 3.3
FM-04	Organic and Explosive Vapor Measurement SOP	AI SOPs and FWS Investigation Guidance[2]	See SOP	N	Appendix B Section 3.4

Table 21-1. Project Sampling SOP References					
Reference Number[1]	**Title, Revision Date and/or Number**	**Originating Organization of Sampling SOP**	**Equipment Type**	**Modified for Project Work? (Y/N)**	**SOP Location**
FM-07	Electrical Conductivity, pH, Temperature, Oxidation/Reduction Potential and Dissolved Oxygen Measurement SOP	AI SOPs and FWS Investigation Guidance[2]	See SOP	N	Appendix B Section 3.7
FM-08	Water Turbidity Measurement SOP	AI SOPs and FWS Investigation Guidance[2]	See SOP	N	Appendix B Section 3.8
RK-01	General Record Keeping SOP	AI SOPs	See SOP	N	Appendix B Section 4.1
RK-02	Logbook SOP	AI SOPs and FWS Investigation Guidance[2]	See SOP	N	Appendix B Section 4.2
RK-03	Field Data Forms SOP	AI SOPs and FWS Investigation Guidance[2]	See SOP	N	Appendix B Section 4.3

[1]Reference Number for SOP included in Appendix B, Standard Operating Procedures.
AI – Applied Intellect, LLC
ES – Environmental Sampling
FM – Field Measurements
FO – Field Operations
N – No
RK – Record Keeping
SOP – Standard Operating Procedure/Practice
Y – Yes

22. QAPP WORKSHEET #22 - FIELD EQUIPMENT CALIBRATION, MAINTENANCE, TESTING, AND INSPECTION

This worksheet summarizes commonly used field equipment and details calibration, maintenance, testing and inspections.

Table 22-1. Field Equipment Calibration, Maintenance, Testing, and Inspection

Field Equipment	Activity	Frequency	Acceptance Criteria	Corrective Action	Resp. Person	SOP Reference	Comments
Water Level Sounder	Test for sound	Prior to use	Notable Beep	Check battery condition	Field Team Lead	User Manual provided by manufacturer; SOPs FM-02, FM-03, FM-05 Appendix B Section 3	No additional comments.
Turbidity Meter	Calibration of Turbidity	Daily Turbidity calibration	Within the specified range for Turbidity in the SOP	If sensor fails to calibrate, the sensor will be cleaned and a span calibration will be performed, or the unit will be returned to a qualified service representative for repairs.	Field Team Lead	User Manual provided by manufacturer; SOPs FM-02, FM-03, FM-08 Appendix B Section 3	Calibration should be performed at the beginning of each day, prior to any sampling
Multi-Parameter Water Quality Meter	Calibration of pH, Conductivity, Temperature, DO, ORP	Daily pH calibration	Within the specified range for pH in the SOP	If sensor fails to calibrate, the sensor will be cleaned and a span calibration will be performed, or the unit will be returned to a qualified service representative for repairs.	Field Team Lead	User Manual provided by manufacturer; SOPs FM-02, FM-03, FM-07 Appendix B Section 3	Calibration should be performed at the beginning of each day, prior to any sampling
		Daily Conductivity calibration	Within the specified range for Conductivity in the SOP				
		Daily Temperature calibration	Within the specified range for Temperature in the SOP				
		Daily DO calibration	Within the specified range for DO in the SOP				
		Daily ORP calibration	Within the specified range for ORP in the SOP				

Table 22-1. Field Equipment Calibration, Maintenance, Testing, and Inspection							
Field Equipment	**Activity**	**Frequency**	**Acceptance Criteria**	**Corrective Action**	**Resp. Person**	**SOP Reference**	**Comments**
Photoionization Detector[1]	Calibrates to 100 ppm Isobutylene	Daily Calibration	Isobutylene ppm ±1%	If standards do not pass, contact technical support. If problem cannot be corrected, a new meter will be obtained.	Field Team Lead	User Manual provided by manufacturer; SOPs FM-02, FM-03, FM-04 Appendix B Section 3	Calibration should be performed at the beginning of each day, prior to any sampling

[1] A function check is first performed on the equipment. The calibration gas is tested to ensure that it falls within manufacturer's criteria. If the function check is acceptable, then the instrument is ready for use. Otherwise, the instrument will be calibrated according to manufacturer's instructions.
% – Percent
± – Plus or Minus
ORP – Oxidation-Reduction Potential
pH – Measure of acidity or basicity of an aqueous solution
ppm – parts per million
SOP – Standard Operating Procedure/Practice

23. QAPP WORKSHEET #23 - ANALYTICAL SOP REFERENCE

This worksheet summarizes the laboratory's analytical SOPs for the project. The laboratory's proprietary SOPs are not included in this UFP-QAPP; however, they are available (as confidential business information) upon request.

Table 23-1. Analytical SOP References

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work (Y/N)
ED-MT-017	Mercury Analysis for Water and Wastewater Samples by 245.1, 7470A using the Leeman	Definitive	Water, Metals	CVAA	TestAmerica Edison	N
ED-MT-034	SW-846 Method 6020A and 6020B Trace Metals Analysis of Water, Wastewater, Soil, Sediment and	Definitive	Water, Solid, Metals	ICP-MS	TestAmerica Edison	N
ED-MT-035	Mercury Analysis for Solid and Semisolid Waste Samples using the Leeman Mercury Analyzer (Cold	Definitive	Solid, Metals	CVAA	TestAmerica Edison	N
ED-MTP-003	Digestion of Water and Wastewater Samples for Analysis by ICP using Method 3010A	Definitive	Water, Metals	Hot Block	TestAmerica Edison	N
ED-MTP-005	Hot Block Digestion of Sediments, Sludges, and Soils using SW846 Method 3050B	Definitive	Solid, Metals	Hot Block	TestAmerica Edison	N
ED-ORP-002	SW846 Method 3510C-Extraction of Semi-Volatile Organic Compounds in Water by Separatory Funnel	Definitive	Water, SVOA	Sep Funnel	TestAmerica Edison	N
ED-ORP-014	SW846 Method 3510C-Extraction of Pesticides and PCBs in Water by Separatory Funnel	Definitive	Water, Pest/PCB	Sep Funnel	TestAmerica Edison	N
ED-ORP-044	SW846 Method 3546: Microwave Extraction of Solids	Definitive	Soil, SVOA, Pest/PCB	Microwave	TestAmerica Edison	N
ED-GCS-016	SW846 Method 8081B, Analysis of Organochlorine Pesticides by Gas Chromatography	Definitive	Water, Soil, Pes	GC/ECD	TestAmerica Edison	N
ED-GCS-017	SW846 Method 8082A, Analysis of PCBs by Gas Chromatography	Definitive	Water, Soil, PCB	GC/ECD	TestAmerica Edison	N
ED-MSS-009	SW8270D, Semivolatile Organic Compounds by Gas Chromatography/ Mass Spectrometry (GC/MS)	Definitive	Water, Soil, SVOA	GC/MS	TestAmerica Edison	N
ED-MSV-001	Purge and Trap for Aqueous Samples Method 5030, SW846	Definitive	Water, VOA	Purge and Trap	TestAmerica Edison	N
ED-MSV-002	SW846 Method 5035A, Closed System Purge and Trap and Extraction for Volatile Organics in Soil	Definitive	Solid, VOA	Purge and Trap	TestAmerica Edison	N
ED-MSV-014	SW846 Method 8260C Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry	Definitive	Water, Solid, VOA	GC/MS	TestAmerica Edison	N

ED-WET-076	TOC, The Determination of Total Organic Carbon in Solid Samples (Lloyd Kahn) by Flash EA 1112 Analyzer	Definitive	Solid, Wet Chem	TOC Analyzer	TestAmerica Edison	N
ED-WET-061	TPH SOIL, Analysis of pH for Soils and Organic Samples Electrochemically (SW846 9045C/9045D)	Definitive	Solid, Wet Chem	pH meter	TestAmerica Edison	N
WS-LC-0025, Rev. 3.8	SW846 Method 537 for PFAs - Per- and Polyfluoro-alkyl Substances Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS)	Definitive	Water	(LC/MS/MS)	TestAmerica Sacramento	N

BNA – Base, Neutrals, Acids
FIA – Flow Injection Analysis
GC/FID – Gas Chromatography/Flame Ionization Detector
GC/MS – Gas Chromatography/Mass Spectrometry
IC – Ion Chromatography
LC/MS/MS - Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry
N – No
N/A – Not Applicable
pH – Measure of acidity or basicity in an aqueous solution
SIM – Selective Ion Monitoring
SOP – Standard Operating Procedure/Practice
USEPA – United States Environmental Protection Agency
VOC – Volatile Organic Compound

24. QAPP WORKSHEET #24 - ANALYTICAL INSTRUMENT CALIBRATION

This worksheet describes the calibration procedures and schedule for the analytical instruments used on this project. Values provided in this table were derived from the Project Laboratory SOPs.

Table 24-1. Analytical Instrument Calibration

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	Analytes/ Method
GC/MS	ICAL	As needed; when CCV out of criteria	• Option 1: Average RRF if RSD for each target analyte < 20%; ≤50%D for designated poor performers • Option 2: linear least squares regression r ≥ 0.995	Correct problem; Repeat calibration	Analyst/ Supervisor	Volatile Organics by 8260D
	ICV	After each ICAL	• Value of all target analytes within ± 20% of expected value	Reanalyze ICV; upon 2nd failure, repeat calibration		
	CCV	Daily; every 12 hours	• The initial CCV: All target analytes ≤ 20%D; ≤50%D for designated poor performers	Reanalyze CCV; if failure, repeat ICAL		
	BFB Tune	initially (prior to ICAL))	• Mass Criteria: • Mass 50 15.0-40.0 percent of mass 95 • Mass 75 30.0-60 percent of mass 95 • Mass 95 Base peak, 100 percent relative abundance • Mass 96 5.0 to 9.0 percent of mass 95 • Mass 173 Less than 2.0 percent of mass 174 • Mass 174 Greater than 50.0 percent of mass 95 • Mass 175 5.0 to 9.0 percent of mass 174 • Mass 176 95.0 to 101 percent of mass 174 • Mass 177 5.0 to 9.0 percent of mass 176	Retune and/or clean source		

Table 24-1. Analytical Instrument Calibration						
Instrument	**Calibration Procedure**	**Frequency of Calibration**	**Acceptance Criteria**	**Corrective Action**	**Person Responsible for CA**	**Analytes/ Method**
GC/MS	ICAL	As needed; when CCV out of criteria	• Option 1: Average RRF if RSD for each target analyte < 20% • Option 2: linear least squares regression r ≥ 0.995 • Option 3: non-linear regression COD r^2 ≥ 0.99	Correct problem; Repeat calibration	Analyst/ Supervisor	Semivolatile Organics by 8270E
	ICV	After each initial calibration	• Value of all target analytes within ± 20% of expected value	Reanalyze ICV; upon 2nd failure, repeat calibration		
	CCV	Daily; every 12 hours	• The initial CCV: All target analytes ≤ 20%D; ≤50%D for designated poor performers • RRF criteria as listed in the method.	Reanalyze CCV; if failure, repeat ICAL		
	DFTPP Tune	Initially (prior to ICAL)	• Mass Criteria: • Mass 51 30.0-60.0% of mass 198 • Mass 68 <2% of mass 69 • Mass 70 <2% of mass 69 • Mass 127 40.0 to 60.0 percent of mass 198 • Mass 197 Less than 1.0%of mass 198 • Mass 198 Base peak, 100% relative abundance • Mass 199 5.0 to 9.0%of mass 198 • Mass 275 10.0 to 30.0%of mass 198 • Mass 365 >1% of mass 198 • Mass 441 Present but less than mass 443 • Mass 442 >40% of mass 198 • Mass 443 17.0-23.0% of mass 442	Retune and/or clean source		

Table 24-1. Analytical Instrument Calibration						
Instrument	**Calibration Procedure**	**Frequency of Calibration**	**Acceptance Criteria**	**Corrective Action**	**Person Responsible for CA**	**Analytes/ Method**
GC	ICAL	Initially and upon failure of two consecutive CCVs	• RSD <20% for all target compounds or linear or quadratic calibration $r^2 > 0.990$	Correct problem; Repeat calibration	Analyst/ Supervisor	Pesticides by 8081B, PCBs by 8082A
	ICV	After each initial calibration	• 80-120% Recovery	Reanalyze ICV; if failure, repeat ICAL		
	CCV	Prior to sequence, every 10 samples thereafter	• % Drift ± 20%	Reanalyze CCV; if failure, repeat ICAL		
ICP-MS	ICAL	Daily	•Correlation Coefficient of ≥0.995	Correct problem; Repeat calibration	Analyst/ Supervisor	Metals by 6020
	ICV	After each initial calibration	90-110% Recovery	Reanalyze ICV; if failure, repeat ICAL		
	CCV	Prior to sequence, every 10 samples and end of	90-110% Recovery	Reanalyze CCV; if failure, repeat ICAL and all associated samples		
Mercury Analyzer	ICAL	Daily	•Correlation Coefficient of ≥0.995	Correct problem; Repeat calibration	Analyst/ Supervisor	Mercury by 7470/7471
	ICV	After each initial calibration	90-110% Recovery	Reanalyze ICV; if failure, repeat ICAL		
	CCV	Prior to sequence, every 10 samples and end of sequence	90-110% Recovery	Reanalyze CCV; if failure, repeat ICAL and all associated samples		

%D – Percent Difference
≥ – Greater Than or Equal to
< – Less Than
≤ – Less Than or Equal to
± – Plus or Minus
BFB – 4-Bromofluorobenzene
CCV – Continuing Calibration Verification
COD – Coefficient of Determination
DFTPP – Decafluorotriphenylphosphine
GC – Gas Chromatograph
GC/MS – Gas Chromatograph/Mass Spectrometer
IC – Ion Chromatograph
ICAL – Initial Calibration
ICV – Initial Calibration Verification
PAH – Polycyclic Aromatic Hydrocarbon r – Linear Regression Value

RRF – Relative Response Factor
RSD – Relative Standard Deviation
VOC – Volatile Organic Compound

Table 24-2. Analytical Instrument Calibration (Dioxin/Furans, PCB congeners)						
Instrument	**Calibration Procedure**	**Frequency of Calibration**	**Acceptance Criteria**	**Corrective Action**	**Person Responsible for CA**	**Analytes/ Method**
GC/HRMS	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.	Lab Manager / Analyst [b]	Dioxins/ Furans WS-ID-0005
HRGC/HRMS	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%.	Lab Manager / Analyst [b]	Dioxins/ Furans WS-ID-0005
GC/HRMS	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 & 9 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.	Lab Manager / Analyst [b]	Dioxins/ Furans WS-ID-0005

Table 24-2. Analytical Instrument Calibration (Dioxin/Furans, PCB congeners)						
Instrument	**Calibration Procedure**	**Frequency of Calibration**	**Acceptance Criteria**	**Corrective Action**	**Person Responsible for CA**	**Analytes/ Method**
GC/HRMS	Second-source calibration verification	Immediately following ICAL.	Ion abundance ratios in accordance with SOP; and RF (unlabeled standards) within ± 20%D of average RF from ICAL; and RF (labelled standards) 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	Lab Manager / Analyst [b]	Dioxins/ Furans WS-ID-0005
GC/HRMS	Calibration Verification (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 RF (unlabeled standards) within ± 20%D of average RF from ICAL; and RF (labelled standards) within ± 30%D of average RF from ICAL.	Correct problem repeat calibration verification. If fails, repeat ICAL and reanalyze all samples analyzed since last successful CCV End of Run CCV: If RF (unlabeled standards) > ± 20%D and ≤ ± 25%D and/or RF (labelled standards) > ± 30%D and ≤ ± 35%D of the average RF from ICAL use mean RF from bracketing CCVs to quantitate impacted samples. If bracketing CCVs differ by more than 25% RPD (unlabeled) or 35% RPD (labelled), run a new ICAL within 2 hours, and requantitate samples. Otherwise, reanalyze samples with positive detections.	Lab Manager / Analyst [b]	Dioxins/ Furans WS-ID-0005

Table 24-2. Analytical Instrument Calibration (Dioxin/Furans, PCB congeners)						
Instrument	**Calibration Procedure**	**Frequency of Calibration**	**Acceptance Criteria**	**Corrective Action**	**Person Responsible for CA**	**Analytes/ Method**
GC/HRMS	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. End of run check must be ≥ 5,000	Retune instrument & verify. Assess data for impact if end resolution is less than 10,000 narrate or reinject, as necessary.	Lab Manager / Analyst [b]	PCB Congeners WS-ID-0013
HRGC/HRMS	GC Column Performance Check (CPSM/WDM per method)	Prior to ICAL or calibration verification.	The congener pairs 23/34 and 182/187 are checked for chromatographic resolution. The valley between each pair must be less than 40% of the shorter of the two peaks . The CS-3 (CCV) is used to define chromatographic windows. First and last eluter must be present in each window.	1) Readjust windows. 2) Evaluate system. 3) Perform maintenance. 4) Reanalyze CPSM.	Lab Manager / Analyst [b]	PCB Congeners WS-ID-0013
GC/HRMS	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 Toxic/LOC compounds [a]	Evaluate standard and instrument response. If problem with instrument (autosampler failure, response poor, etc.) or standards, correct as appropriate, then repeat initial calibration.	Lab Manager / Analyst [b]	PCB Congeners WS-ID-0013
GC/HRMS	Second-source calibration verification	Immediately following ICAL.	All project analytes within ± 30% of the expected value from the ICAL.	Evaluate standards and instrument response. If standard issue, repeat or remake then repeat standard as appropriate. If still fails, repeat initial calibration	Lab Manager / Analyst [b]	PCB Congeners WS-ID-0013

Table 24-2. Analytical Instrument Calibration (Dioxin/Furans, PCB congeners)						
Instrument	**Calibration Procedure**	**Frequency of Calibration**	**Acceptance Criteria**	**Corrective Action**	**Person Responsible for CA**	**Analytes/ Method**
GC/HRMS	Calibration Verification (CCV)	At the beginning of each 12-hour period.	Ion abundance ratios in	Correct problem repeat calibration verification. If fails, repeat ICAL and reanalyze all samples analyzed since last successful CCV	Lab Manager / Analyst [b]	PCB Congeners WS-ID-0013

a The toxics/LOCs are the 27 congeners that are calibrated by a multipoint curve. They encompass the WHO list toxic congeners and the first and last eluter

for each level of chlorination (LOC). All other congeners are quantified off of a daily single point standard.
[b] The analyst initiates the corrective action and the lab manager and analyst are responsible for the corrective action.

%D – Percent Difference
≥ – Greater Than or Equal to
< – Less Than
≤ – Less Than or Equal to
± – Plus or Minus
BFB – 4-Bromofluorobenzene
CCV – Continuing Calibration Verification
COD – Coefficient of Determination
DFTPP – Decafluorotriphenylphosphine
GC – Gas Chromatograph
GC/MS – Gas Chromatograph/Mass Spectrometer
IC – Ion Chromatograph
ICAL – Initial Calibration
ICV – Initial Calibration Verification
PAH – Polycyclic Aromatic Hydrocarbon r – Linear Regression Value
RRF – Relative Response Factor
RSD – Relative Standard Deviation
VOC – Volatile Organic Compound

Table 24-3 Analytical Instrument Calibration (PFAs)						
Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	Analytes/ Method
LC/MS/MS PFAs	TBD					

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

This worksheet describes the maintenance, testing, and inspection procedures for commonly used analytical instruments. The laboratory's SOP associated with each instrument/analysis is listed in Worksheet #23.

Table 25-1. Analytical Instrument and Equipment Maintenance, Testing, and Inspection

Instrument/ Equipment	Maintenance Activity	Analytical Group	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person
GC/MS	Clean mass spectrometer source, change trap, clip column, change purge and trap ferrules, bake out column	VOCs	Purge lines, purge flow, trap, ion source, column	Prior to calibration check and/or as necessary	Acceptable ICAL or CCV	Correct problem and repeat ICAL or CCV	Analyst / Supervisor
GC/MS	Clean mass spectrometer source, Check for leaks, replace gas line filters, replace column, clean injection port/liner	SVOCs	Purge lines, purge flow, trap, ion source, column	Prior to calibration check and/or as necessary	Acceptable ICAL or CCV	Correct problem and repeat ICAL or CCV	Analyst / Supervisor
GC	Change/check helium, clip column, bake column; change septa and liner	Pest/PCB	Check gases; peak shapes and drift	Prior to calibration check and/or as necessary; change septa each day of use and liner weekly	Acceptable ICAL or CCV	Correct problem and repeat ICAL or CCV	Analyst / Supervisor
ICP-MS	Check sample waste container level. Check quartz torch condition. Measure quartz torch for proper alignment. Check oil level of roughing pumps. Check peristaltic pump: proper roller pressure, sample introduction tubing, correct pump rotation, and condition of drain tubing. Check condition of sampler and skimmer cones. Check and drain oil mist eliminator on roughing pumps.	Metals	Inspect waste container, quartz torch, pump, sampler and skimmer cones.	Maintenance is performed prior to initial calibration or as necessary.	The acceptance criteria for the continuing calibration standard are 90 to110% of true value.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/Supervisor

Table 25-1. Analytical Instrument and Equipment Maintenance, Testing, and Inspection							
Instrument/ Equipment	**Maintenance Activity**	**Analytical Group**	**Inspection Activity**	**Frequency**	**Acceptance Criteria**	**Corrective Action**	**Responsible Person**
CVAA Mercury	Change the tubing. Check the reagents and standards.	Mercury	Inspect the tubing, filter, and optical cell.	Maintenance is performed prior to initial calibration or as necessary.	The acceptance criteria are 80 to120% of the true value.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/ Supervisor
GC/HRMS	Parameter Setup Testing Activity: Physical check	Dioxin/ Furan	Physical check	Initially; prior to DCC	Correct Parameters	Reset if incorrect	TestAmerica Chemist
GC/HRMS	Tune Check Testing Activity: Instrument Performance	Dioxin/ Furan	Conformance to instrument tuning.	Initially; prior to DCC	Compliance to ion abundance criteria	Correct the problem and repeat tune check	TestAmerica Chemist
GC/HRMS	Parameter Setup Testing Activity: Physical check	PCB Congeners	Physical check	Initially; prior to DCC	Correct Parameters	Reset if incorrect	TestAmerica Chemist
GC/HRMS	Tune Check Testing Activity: Instrument Performance	PCB Congeners	Conformance to instrument tuning.	Initially; prior to DCC	Compliance to ion abundance criteria	Correct the problem and repeat tune check	TestAmerica Chemist
LC/MS/MS	TBD						

CCV – Continuing Calibration Verification
GC/MS – Gas Chromatograph/Mass Spectrometer
ICAL – Initial Calibration
LC/MS/MS - Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry
nm – Nanometer
PAH – Polycyclic Aromatic Hydrocarbon

TBD – To Be Determined
VOC – Volatile Organic Compound

26. QAPP WORKSHEET #26 - SAMPLE HANDLING SYSTEM

This worksheet lists all personnel who are primarily responsible for ensuring proper handling, custody, and storage of field samples from the time of collection, to laboratory delivery, to sample disposal.

Table 26-1. Sample Handling System

	Responsible Person	Organization
SAMPLE COLLECTION, PACKAGING, AND SHIPMENT		
Sample Collection (Personnel/Organization):	Installation Lead (or designee)	AI
Sample Packaging (Personnel/Organization):	Installation Lead (or designee)	AI
Coordination of Shipment (Personnel/Organization):	Installation Lead (or designee)	AI
Type of Shipment/Carrier:	FedEx, UPS, or Courier Service	
SAMPLE RECEIPT AND ANALYSIS		
Sample Receipt (Personnel/Organization):	Laboratory Project Manager	Eurofins/TestAmerica
Sample Custody and Storage (Personnel/Organization):	Laboratory Project Manager	Eurofins/TestAmerica
Sample Preparation (Personnel/Organization):	Laboratory Project Manager	Eurofins/TestAmerica
Sample Determinative Analysis (Personnel/Organization):	Laboratory Project Manager	Eurofins/TestAmerica
SAMPLE ARCHIVING		
Field Sample Storage (No. of days from sample collection):	N/A	N/A
Sample Extract/Digestate Storage (No. of days from extraction/digestion):	Laboratory Project Manager	Eurofins/TestAmerica
Biological Sample Storage (No. of days from sample collection):	N/A	N/A
SAMPLE DISPOSAL		
Personnel/Organization:	Laboratory Project Manager	Eurofins/TestAmerica
Number of Days from Analysis:	60 days	

Note: Project Personnel are identified in Worksheet #7
AI – Applied Intellect, LLC
Eurofins/TestAmerica
N/A – not applicable

27. QAPP WORKSHEET #27 - SAMPLE CUSTODY REQUIREMENTS

This worksheet presents the sample ID procedures. Sample documentation (labels, COC, field log book, etc.) and sample shipment protocols are presented in Appendix B. Appendix B presents the procedures used to maintain sample custody and integrity (e.g., labels, COCs, field logbook) and protocols for sample shipment.

27.1 SAMPLE IDENTIFICATION

Each sample collected will be given a unique sample ID. A record of all sample IDs will be kept with the field records and recorded on a COC form. In addition, the sample IDs will be used to identify and retrieve analytical results from the laboratory, validation, and upload into the project database. Sample IDs will be formatted as follows:

- "GS-" Installation Acronym (e.g., GS = Great Swamp National Wildlife Refuge)
- "N" Study Area Acronym
    - N = Northeastern Refuge Study Area;
    - S = Southern Refuge Study Area;
    - O = Off-Refuge Study Area.
- "SS1" is an example of the matrix acronym
    - SS1 = surface soil followed by a serial number to identify the sample location in each study area
    - SO-0-3= subsurface soil sample collected in the 0 to 3 ft bgs depth interval;
    - PW3T = pore water followed by a serial number to identify the sample location in each study area, unfiltered;
    - PW3D = pore water, followed by a serial number to identify the sample location in each study area filtered;
    - SW3T = surface water, followed by a serial number to identify the sample location in each study area unfiltered; and
    - SW3D = surface water, filtered.
    - SD = sediment
- "DP1" is a direct push boring location that may be associated with subsurface soil samples (SO) or HydroPunch groundwater samples (GW)
    - DP locations will be serially numbered in each study area.
- "MW3" is the monitoring well identifier for groundwater samples collected from monitoring wells.

For example:

- GS-N-DPT4-SO-X-X represents a subsurface soil sample from the LTD to BLTD ft depth interval at the DPT4 direct push location in the N of the Great Swamp National Refuge.
- Field duplicates will be designated by a serially increasing number starting at "100" for each sample location:

  - For normal surface water sample GS-N-SW7T-1, the first duplicate surface water sample in the N would be GS-N-SW3**00**T-1; the second duplicate would be GS-N-SW3**01**T-1.
- Trip blanks will be associated with a specific cooler; therefore trip blanks will be assigned a sequential number identical to the cooler number (e.g., GS-TB-1 for cooler #1, GS-TB-2 for cooler #2, etc.). To insure correct association, and electronic documentation, the sample manager will write the cooler number in the notes section of the COC.
- Equipment blanks will be collected at a rate of one per day, when subsurface soil samples are collected. Equipment blanks will be collected at the start of each field day. The site number where the equipment blank is collected will be identified in the sample ID followed by a sequential number for each day of sampling (e.g., GS-N-EB-1 for the first day of sampling, GS-N-EB-2 for the second day of sampling, etc.).
- MS/MSDs will be generated in the laboratory from samples specified in the field. Triple sample volume will be collected and submitted to the laboratory for analysis; however, MS/MSDs will not have a separate sample ID. The COCs will designate which sample locations are associated with the extra volume for MS/MSD analysis.

28. QAPP WORKSHEET #28 - LABORATORY QC SAMPLES

This worksheet identifies the QC samples and their respective acceptance limits for commonly used analytical groups.

Table 28-1. Laboratory QC Samples Table for VOCs

Matrix	Soil and Water
Analytical Group	VOCs
Analytical Method/ SOP Reference[1]	SW-846 8260

QC Sample	Frequency / Number	Method/SOP QC Acceptance Limits[2]	CA	Person(s) Responsible for CA	DQI	Measurement Performance Criteria
Method Blank	Daily and/or 1 per batch of 20 samples	No analytes detected > 1/2 LOQ 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 > LOQ.	If all samples below 1/2 LOQ, no CA. Otherwise, reprep and reanalyze all samples processed with contaminated blank.	Analyst/Supervisor	Bias/ Contamination	No analytes detected > 1/2 LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). For common laboratory contaminants, no analytes detected > LOQ.
LCS	Daily and/or 1 per batch of 20 samples	See limits in Worksheet #15	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.		Accuracy/Bias	See limits in Worksheet #15
MS/MSD	1 per batch of 20 samples if client designated	See limits in Worksheet #15	No action required.		Accuracy/Bias	See limits in Worksheet #15
Internal Standards (IS)	Every sample	Retention times ± 30 seconds from retention time of midpoint standard in the ICAL; and EICP area within -50 to + 100% of ICAL midpoint standard	Inspect GC/MS for malfunctions or interferences. Reanalyze sample		Accuracy/Bias	Retention times ± 30 seconds from retention time of midpoint standard in the ICAL; and EICP area within -50 to + 100% of ICAL midpoint standard
Surrogates	Every sample	See limits in Worksheet #12	Reanalyze sample		Accuracy/Bias	See limits in Worksheet #12

Table 28-2. Laboratory QC Samples Table for SVOCs

Matrix	Soil and Water
Analytical Group	SVOCs
Analytical Method/ SOP	SW-846 8270E

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits[2]	CA	Person(s) Responsible for CA	DQI	Measurement Performance Criteria
Method Blank	1 per batch of 20 samples	No analytes detected > 1/2 LOQ 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 > LOQ.	If all samples below 1/2 LOQ, no CA. Otherwise, reprep and reanalyze all samples processed with contaminated blank.	Analyst/ Supervisor	Bias/ Contamination	No analytes detected > 1/2 LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). For common laboratory contaminants, no analytes detected > LOQ.
LCS	1 per batch of 20 samples	See limits in Worksheet #15	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.		Accuracy/Bias	See limits in Worksheet #15
MS/MSD	1 per batch of 20 samples	See limits in Worksheet #15	No action required.		Accuracy/Bias	See limits in Worksheet #15
IS	Every sample	Retention times ± 30 seconds from retention time of midpoint standard in the ICAL; and EICP area within -50 to + 100% of ICAL midpoint standard	Inspect GC/MS for malfunctions or interferences. Reanalyze sample		Accuracy/Bias	Retention times ± 30 seconds from retention time of midpoint standard in the ICAL; and EICP area within -50 to + 100% of ICAL midpoint standard
Surrogates	Every sample	See limits in Worksheet #12	Reanalyze sample		Accuracy/Bias	See limits in Worksheet #12

Table 28-3. Laboratory QC Samples Table for Metals by ICPMS

	Soil and Water					
Analytical Group	Metals by ICP/MS					
Analytical Method/ SOP	EPA 6020B					
QC Sample	**Frequency/ Number**	**Method/SOP QC Acceptance Limits[2]**	**CA**	**Person(s) Responsible for CA**	**DQI**	**Measurement Performance Criteria**
Method Blank (MB)	1/ Preparatory Batch (20 samples)	No Target Compounds> ½ LOQ and greater than 1/10 the amount measured in any sample or greater than project specific requirements.	If sufficient sample is available, reanalyze samples. Qualify data as needed. Report results if sample results >10x blank result or sample results ND	Analyst	Accuracy/Bias-Contamination	No Target Compounds>1/2 LOQ
Laboratory Control Sample (LCS)	1/Preparatory Batch (20 samples)	Water: %Recoveries 80 to 120%	all samples prepared in association with the LCS must be redigested and reanalyzed		Accuracy/Bias	Laboratory % Recovery Limits
Matrix Spike / (MS)	1 pair/Preparatory Batch (20 samples)	Percent recoveries: 75-125% RPD: RPD between MS and	Evaluate LCS, unspiked sample and qualify data		Accuracy/Bias/	Laboratory % Recovery
Matrix Duplicate	1 /Preparatory Batch (20 samples)	RPD ≤ 20%	Qualify Data		Precision	Laboratory control limits
Dilution Test	One per preparatory batch Only applicable for samples with concentrations >25 x LOQ.	Five-fold dilution must agree within ± 20% of the original determination	If dilution test fails re-analyze, if it fails dilute QC samples to higher dilution to eliminate possible matrix interference.		Accuracy/Bias/ Precision	Within 20% of original determination
Post Digestion Spike (PDS)	One per analytical batch	Recovery within 75-125% of expected results	Lab narrates outlier; qualify data if acceptance criteria are not met.		Accuracy/Bias	Within +/-25% of expected results

Table 28-4. Laboratory QC Samples Table for Mercury in Soil

Matrix	Soil
Analytical Group	Mercury by
Analytical Method/ SOP	EPA 7470A

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits[2]	CA	Person(s) Responsible for CA	DQI	Measurement Performance Criteria
Method Blank (MB)	1/Preparatory Batch (20 samples)	< Reporting limit	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank. Report results if sample results >10x blank result or sample results ND.	Analyst / Section	Accuracy/Bias-Contamination	No Target Compounds>RL
Laboratory Control Sample (LCS)	1/Preparatory Batch (20 samples)	Vendor's certified limits Current limits: 67-130%	Correct any problems, then re-prep and reanalyze LCS and associated samples for failed analytes in all samples in the associated batch.		Accuracy/Bias	Vendor Control Limits
Matrix Spike (MS)	1/Preparatory Batch (20 samples)	Percent Recoveries: 75-125%	Evaluate LCS, qualify data		Accuracy/Bias	Laboratory % Recovery
Matrix Duplicate	1/Preparatory Batch (20 samples)	RPD ≤ 20%	Qualify Data		Precision	Laboratory control limits

Table 28-5. Laboratory QC Samples Table for Mercury in Water

Matrix	Water
Analytical Group	Mercury by
Analytical Method/ SOP	EPA 7470A

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits[2]	CA	Person(s) Responsible for CA	DQI	Measurement Performance Criteria
Method Blank (MB)	1/Preparatory Batch (20 samples)	< Reporting limit	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank. Report results if sample results >10x blank result or sample results ND.	Analyst / Section Supervisor	Accuracy/Bias-Contamination	No Target Compounds> RL
Laboratory Control Sample (LCS)	1/Preparatory Batch (20 samples)	Percent Recoveries: 80-120%	Correct any problems, then re-prep and reanalyze LCS and associated samples for failed analytes in all samples in the associated batch.		Accuracy/Bias	Laboratory % Recovery Control Limits
Matrix Spike (MS)	1/Preparatory Batch (20 samples)	Percent Recoveries: 75-125%	Evaluate LCS, qualify data		Accuracy/Bias	Laboratory % Recovery
Matrix Duplicate	1/Preparatory Batch (20 samples)	RPD: ≤ 20%	Qualify Data		Precision	Laboratory Control Limits

Table 28-6. Laboratory QC Samples Table for Pesticides

Matrix	Soil/Water
Analytical Group	Pesticides
Analytical Method/ SOP	SW846 8081B

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits[2]	CA	Person(s) Responsible for CA	DQI	Measurement Performance Criteria
Method Blank (MB)	1/ Preparatory Batch (20 samples)	No Target Compounds> LOQ	Reanalyze and, if necessary, reextract. Report non-conformance in narrative; compounds present in blank should be flagged "B" in samples, if	Analyst	Accuracy/Bias-Contamination	No Target Compounds>LOQ
Laboratory Control Sample (LCS)	1/Preparatory Batch (20 samples)	Must contain all single-component target analytes, Recovery for all target analytes within lab generated limits	Reanalyze, if necessary, qualify data and narrate issues of nonconformance		Accuracy/Bias	Recovery for all target analytes within lab generated limits
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	1 /Preparatory Batch (20 samples)	Must contain all single-component target analytes, Recovery for all target analytes within lab	Reanalyze, if necessary, qualify data and narrate issues of non-		Accuracy/Bias	Recovery for all target analytes within lab generated limits
Surrogates	Every sample including QC.	Minimum of two recommended (DCB and TCMX). Recovery within lab generated limits on both columns.	Reanalyze if necessary. Qualify data.		Accuracy/Bias	Minimum of two recommended (DCB and TCMX). Recovery within lab generated limits on both columns

Table 28-7. Laboratory QC Samples Table for PCBs

Matrix	Soil/Water
Analytical Group	PCBs
Analytical Method/ SOP	SW846 8082A

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits[2]	CA	Person(s) Responsible for CA	DQI	Measurement Performance Criteria
Method Blank (MB)	1/ Preparatory Batch (20 samples)	No Target Compounds> LOQ	Reanalyze and, if necessary, reextract. Report non-conformance in narrative; compounds present in blank should be flagged "B" in samples, if detected..	Analyst	Accuracy/Bias-Contamination	No Target Compounds>LOQ
Laboratory Control Sample (LCS)	1/Preparatory Batch (20 samples)	Must contain all single-component target analytes, Recovery for all target analytes within lab generated limits	Reanalyze, if necessary, qualify data and narrate issues of nonconformance		Accuracy/Bias	Recovery for all target analytes within lab generated limits
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	1 /Preparatory Batch (20 samples)	Must contain all single-component target analytes, Recovery for all target analytes within lab	Reanalyze, if necessary, qualify data and narrate issues of non-		Accuracy/Bias	Recovery for all target analytes within lab generated limits
Surrogates	Every sample including QC.	Minimum of two recommended (DCB and TCMX). Recovery within lab generated limits on both columns.	Reanalyze if necessary. Qualify data.		Accuracy/Bias	Minimum of two recommended (DCB and TCMX). Recovery within lab generated limits on both columns

Table 28-8. Laboratory QC Samples Table for PCBs (1668A)

Matrix	Soil/Water
Analytical Group	PCBs
Analytical Method/ SOP Reference[1]	EPA Method 1668A / WS-ID-0013

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits[2]	CA	Person(s) Responsible for CA	DQI	Measurement Performance Criteria
Method Blank	One per preparation batch	Project specific criteria, if available. Otherwise, no target analytes detected ≥RL or ≥ 20% of the associated regulatory limit or ≥ 5% of the sample result for the analyte, 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. "Totals" are not considered "target analytes" – no corrective action or flagging is necessary for "totals".	Lab Manager / Analyst	Accuracy/Bias Contamination	No target analytes ≥ RL
Isotope Dilution Analyte (IDA)	Every field sample, standard and QC sample	% recovery for each IDA in the original sample (prior to dilutions) should be limits in Table per method.	If IDA out check for errors and correct. If IDA still out high: ND samples, report and narrate; if detections, but IDA<200% report and narrate; if detections and IDA>200%, dilute, re-analyze, report and narrate. If IDA still out low; ND samples evaluate S/N. If S/N > 10:1, report and narrate; if detections report and narrate. If IDA < 10% re-extract.		Precisions and Accuracy/Bias	Meets all EPA Method requirements (25-150% Recovery for samples, 30-140 for LCS)
Laboratory Control Sample (LCS)	One per sample preparation batch	Method OPR Limits from Table 6 of method.	Reanalyze LCS once. If acceptable, report. Otherwise, evaluate and reprep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.		Accuracy/Bias Contamination	Method OPR Limits (50-150)
MS/MSD	One MS/MSD if requested by program	Method OPR Limits (reproduced in Table 28A) for recovery, 50% RPD	Identify problem; if not related to matrix interference, re-extract and reanalyze MS/MSD.		Precision and Accuracy/Bias	Method OPR Limits (50-150) for recovery,

Table 28-9. Laboratory QC Samples Table for Dioxin/Furans (8290)

Matrix	Soil/Water
Analytical Group	Dioxins/Furans
Analytical Method/ SOP Reference[1]	EPA Method 8290 / WS-ID-0005

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits[2]	CA	Person(s) Responsible for CA	DQI	Measurement Performance Criteria
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 QSM requirements. "Totals" are not considered "target analytes" – no corrective action or flagging is necessary for "totals".	Lab Manager / Analyst	Accuracy/Bias Contamination	No target analytes ≥ 1/2 LOQ
Isotope Dilution Analyte (IDA)	Every field sample, standard and QC sample	% recovery for each IDA in the original sample (prior to dilutions) should be within 40-135%	If IDA out check for errors and correct. If IDA still out high: ND samples, report and narrate; if detections, but IDA<200% report and narrate; if detections and IDA>200%, dilute, re-analyze, report and narrate. If IDA still out low; ND samples evaluate S/N. If S/N > 10:1, report and narrate; if detections report and narrate. If IDA < 10% re-extract.		Precisions and Accuracy/Bias	Meets all EPA Method requirements (40-135% Recovery)

Matrix	Soil/Water
Analytical Group	Dioxins/Furans
Analytical Method/ SOP Reference[1]	EPA Method 8290 / WS-ID-0005

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits[2]	CA	Person(s) Responsible for CA	DQI	Measurement Performance Criteria
Laboratory Control Sample (LCS)	One per sample preparation batch	QSM or laboratory statistically derived control limits	Reanalyze LCS once. If acceptable, report. Otherwise, if exceedance is not a critical chemical of concern (CCoC) 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 CCoC, evaluate and reprep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.		Accuracy/Bias Contamination	QSM or laboratory statistically derived control limits
MS/MSD	One MS/MSD per analytical/preparation batch	QSM or laboratory statistically derived control limits, RPD ≤ 20%.	If not related to matrix interference, re-extract and reanalyze MS/MSD, if sufficient sample material available.		Precision and Accuracy/Bias	QSM or laboratory statistically derived control limits

Table 28-10. Laboratory QC Samples Table for PFAS

Matrix	Soil/Water
Analytical Group	PFAs
Analytical Method/ SOP Reference[1]	EPA Method E357

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits[2]	CA	Person(s) Responsible for CA	DQI	Measurement Performance Criteria
Method Blank	One per preparation batch	TBD				

29. QAPP WORKSHEET #29 - PROJECT DOCUMENTS AND RECORDS

This worksheet identifies project documents and records that will be generated for every aspect of the project.

Table 29-1. Project Documents and Records

Sample Collection Documents and Records	Off-site Analysis Documents and Records	Data Assessment Documents and Records
Field data collection sheets	COC records	Field sampling audit checklists
COC records	Sample receipt forms and sample tracking forms	Analytical audit checklists
Airbills	Preparation and analysis forms and/or logbooks	Data review reports
Communication logs	Tabulated data summary forms and raw data for field samples, standards, QC checks, and QC samples	Telephone logs
Corrective action reports	Case narrative	Corrective action reports
Documentation of corrective action results	Sample chronology (time of receipt, extraction, and analysis)	Laboratory assessment
Documentation of deviation from methods	Identification of QC samples	Laboratory QA plan
	Communication logs	MDL study information
	Corrective action reports	DoD ELAP accreditation
	Definitions of laboratory qualifiers	Hard copy of analytical and raw data
	Documentation of corrective action results	Validated data
	Documentation of laboratory method deviations	
	Electronic data deliverables	
	Instrument calibration reports	
	Laboratory name	
	Laboratory sample identification numbers	
	Reporting forms, completed with actual results	
	Signatures for laboratory sign-off (e.g., laboratory QA manager)	
	Standards traceability records	

COC – Chain of Custody
DoD – Department of Defense
ELAP – Environmental Laboratory Accreditation Program
MDL – Method Detection Limit
QA – Quality Assurance
QC – Quality Control

30. QAPP WORKSHEET #30 - ANALYTICAL SERVICES

This worksheet identifies the laboratories that will provide the analytical services for this project. The laboratories shown below are ELAP-accredited and New Jersey-certified.

Table 30-1. Analytical Services

Matrix	Analytical Group	Sample Locations / ID Number	Analytical Method/SOP	Data Package Turnaround Time (business days)	Laboratory/Organization (name and address, contact person, and telephone number)	Backup Laboratory/ Organization [1] (name and address, contact person and telephone number)
Soil, Water	VOCs SVOCs, Pesticides PCBs (as Aroclors), PCB (as Congeners) Dioxins/Furans TAL Metals Mercury Cyanide	See Worksheet #18	8260C/5035A-FW 8270D/3546 8081B 3546 8082A 3546 1668A/HRMS_Sox_P 8290A/8290_P_Sox 6020B/3050B 7471B/7471B_Prep 9012B/9012B_Prep	15 days	Eurofins/TestAmerica (name and address, contact person, and telephone number)	Eurofins/TestAmerica (name and address, contact person, and telephone number)

[1] Laboratories providing definitive data are accredited by the ELAP and New Jersey.
[2] Fate and transport Parameters include anions (chloride, sulfate) by Method 9056A and nitrate+nitrite by Method 353.2; dissolved gases (methane, ethane, and ethene) by RSK 175; and alkalinity by SM 2320B.
[3] Includes Bulk density by ASTM Method D2937; porosity; moisture content by ASTM Method D2216; and fraction organic carbon content by ASTM Method D5084.
ID - Identifier
PAH – Polycyclic Aromatic Hydrocarbon
SIM – Selective Ion Monitoring
SOP – Standard Operating Procedure/Practice
VOC – Volatile Organic Compound

31. QAPP WORKSHEET #31 - PLANNED PROJECT ASSESSMENT

This worksheet identifies the assessments/audits planned for the project.

Table 31-1. Planned Project Assessments [1]

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment	Person(s) Responsible for Responding to Assessment Findings	Person(s) Responsible for Identifying and Implementing CA	Person(s) Responsible for Monitoring Effectiveness of CA
Laboratory Verification	Prior to identifying a laboratory for the project	Internal	AI	Project Chemist	Laboratory Project Manager or designee	Laboratory Quality Assurance Manager or designee	Project Chemist
Facility Notification	1 month and 48 hours prior to start of field work	Internal	AI	Installation Lead	N/A	N/A	N/A
Subcontractor Notifications	1 month and 48 hours prior to start of sampling	Internal	AI	Project Manager	N/A	N/A	N/A
Field Audit	May occur during field work	Internal	AI	Quality Manager (or designee)	Installation Lead	Installation Lead	Installation Lead and Project Manager
Health and Safety Audit	Daily during field work	Internal	AI	Installation Lead or designee	Installation Lead and Project Manager	Installation Lead	Installation Lead and Project Manager

[1] Project Personnel are identified in Worksheet #7
AI – Applied Intellect, LLC
CA – Corrective Action
N/A – not applicable

31.1 LABORATORY VERIFICATION

Contracted laboratories that perform analysis on definitive data (e.g., VOCs and PAHs) must be DoD ELAP-accredited for each method specified. Laboratory verification consists of ensuring that accreditation of primary and secondary laboratories have not expired. In addition, prior to start of sampling, variances requested by the laboratories must be reviewed and verified that they are acceptable and meet the DQOs listed in this UFP-QAPP.

31.2 FACILITY NOTIFICATION

At least 48 hours prior to start of sampling, the Refuge COR or their representative shall be notified of field activities and all required access to enter the facility has been obtained.

31.3 SUBCONTRACTOR NOTIFICATION

Subcontractors will be notified of the start of field work no later than one month before field work commences. They will be required to provide health and safety, and specialty certifications no later than two weeks before field work commences. Subcontractors will be provided with the Final UFP-QAPP and a HASP for review and sign-off no later than two weeks before field work commences. Subcontractors will provide a signature for all employees that will be working on the project that verifies that they have read and understand the requirements of the project.

31.4 FIELD AUDITS

Prior to start of the project, a visit to the project site will be performed to verify site conditions. Throughout the duration of the project, field documentation and sample receipt forms will be reviewed as needed.

Project quality assurance will be a function of the Quality Manager (or designee), who is assigned the authority to inspect all activities and may stop work if activities detrimental to the quality of the work product are detected. Project personnel will evaluate compliance of the laboratory QA program and procedures with the DoD QSM v5.0 requirements. Oversight may include internal and external audits as needed, documentation of findings, and reports of corrective action. The Quality Manager will coordinate a management review of any deficiencies that are noted.

32. QAPP WORKSHEET #32 - ASSESSMENT FINDINGS AND CORRECTIVE ACTION RESPONSES

This worksheet describes the sequence of events that include documentation of deficiencies, notification of findings, request for corrective action, implementation of corrective action, and follow-up assessment of the corrective action's effectiveness for each assessment/audit performed on the project.

Table 32-1. Assessment Findings and Corrective Action Responses [1]

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response	Timeframe for Response
Field Sampling Audit	Logbook or nonconformance report	Project Manager (or designee)	24 hours after audit	Written Letter	Installation Lead	24 hours after notification
Field Documentation Review	Nonconformance report	Quality Manager	24 hours after document review	Written Memorandum	Project Chemist Installation Lead	24 hours after notification
Laboratory Assessment (if significant QA/QC issues are encountered)	Written audit report	Project Manager Quality Manager Installation Points of Contact or their representative Laboratory Project Manager	5 days after audit	Corrective Action Plan	Laboratory Project Manager	Two weeks after receiving notification

[1] Project Personnel are identified in Worksheet #7
QA/QC – Quality Assurance/Quality Control

32.1 Field Sampling Audit

The Quality Manager may schedule surveillance of field activities at any time to evaluate the execution of sample collection, identification, and control in the field. The Quality Manager (or designee) may conduct surveillance of field activities during the project during a scheduled visit. Sampling operations may be reviewed and compared to the requirements listed in this UFP-QAPP. Use of proper sample containers, proper handling of samples, and adequate documentation of the sampling operation will be verified. The surveillance may include observations of COC procedure, field documentations, instrument calibrations, and field measurements.

32.2 Field Documentation Review

Field documents and COC records will be reviewed to ensure that all entries are printed or written in indelible black or blue ink, dated, and signed. The COC will be reviewed daily for completeness by the Project Chemist. A copy of the COC form will be retained by the Project Chemist and kept in the project file until the completion of the project.

32.3 Laboratory Assessment

The primary and secondary laboratories selected for this project are current on the DoD ELAP accreditation. AI may conduct a laboratory assessment if warranted during the project. The scope of the laboratory assessment by AI will be determined based on quality issues encountered.

32.4 Corrective Action Procedures

The Quality Manager or senior technical staff will document problems and the corrective actions to provide a complete record of QA activities and help identify necessary preventive actions. Non-conformances that affect the findings or recommendations of the project or that have impacts to work outside of the project will be reported to Installation Points of Contact or their representative and the Project Team.

If the laboratories encounter issues during the project that may impact data quality, the Laboratory Project Manager will notify the Project Chemist within one business day of discovery to discuss corrective actions. A written corrective action plan shall be provided in a timely manner and implemented immediately by the laboratory.

33. QAPP WORKSHEET #33 - QA MANAGEMENT REPORTS

This worksheet lists the periodic QA management reports ensuring that managers and stakeholders are updated on project status and the results of the QA assessments.

Table 33-1. Planned Project Assessments

Type of Report	Frequency (Daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation	Report Recipient(s)
Daily Field Report	Daily/after start of sampling	Daily/after start of sampling	Field Team Leader	AI Project Manager
Monthly Progress Report	Monthly	Monthly	Project Manager	FWS COR and CO or their representative
Data Usability Assessment Report	After all data are generated and validated	Submitted with RI Report	Project Chemist	Quality Manager, Project Manager

34. QAPP WORKSHEET #34 - VERIFICATION (STEP I) PROCESS

This worksheet presents the Data Review Process for Verification (Step I). Verification is a completeness check performed before the data review process in order to determine whether the required measurements are collected and all data deliverables (the complete data package) are present. It involves a review of all data inputs to ensure that they are present. The column titled **Internal/External** is in relation to the data generator.

Table 34-1. Verification (Step I) Process

Verification Input	Description	Internal/ External	Responsible for Verification
COC forms	COC forms will be reviewed internally upon their completion and verified against the packed sample coolers they represent. The shipper's signature on the COC should be initialed by the reviewer, a copy of the COC retained in the project file, and the original and remaining copies taped inside the cooler for shipment.	Internal	Field Team Leader
Audit reports	Upon report completion, a copy of all audit reports will be placed in the project file. If corrective actions are required, a copy of the documented corrective action taken will be attached to the appropriate audit report in the project file. At the beginning of each week, and at the completion of the site work, project file audit reports will be reviewed internally to ensure that all appropriate corrective actions have been taken and that corrective action reports are attached. If corrective actions have not been taken, the project manager will be notified to ensure action is taken.	Internal	Installation Lead
Field notes/logbook	Field notes will be reviewed internally and placed in the project file. A copy of the field notes will be attached to the RI Report.	Internal	Installation Lead/ Field Team Leader
Sample Receipt	For samples shipped via courier or by air, the Project Chemist will verify receipt of samples by the laboratory.	Internal	Project Chemist
Sample login	Sample login information will be reviewed for completeness in accordance with the COC forms.	Internal	Installation Lead
		External	Laboratory Project Manager
Laboratory data prior to release	Laboratory data will be reviewed and verified for completeness against analyses requested on the COC forms.	External	Laboratory Project Manager
Laboratory data due at turnaround time listed on COC	Laboratory data will be verified that the analyses reported are consistent with the analyses requested on the COC forms.	Internal	Project Chemist
Laboratory data packages	Laboratory data packages will be verified internally by the laboratory performing the work for completeness and technical accuracy prior to submittal.	External	Laboratory Project Manager or designee.

Table 34-1. Verification (Step I) Process			
Verification Input	**Description**	**Internal/ External**	**Responsible for Verification**
Laboratory data packages	All received data packages will be verified externally by the data validator or project chemist for completeness. All screening level data and site evaluation/fate and transport/geochemical conditions parameters will only undergo Verification (Step I), unless otherwise dictated by project requirements. All definitive data will be validated externally according to the data validation procedures specified in	External	Data Validator or Project Chemist
IDW Disposal Manifests	IDW Disposal Manifests will be reviewed for accuracy and included as an appendix in the RI Report.	Internal	Installation Lead
Field and electronic data	One hundred percent of manual entries will be reviewed against the hardcopy information and 10 percent of electronic uploads will be checked against the hardcopy.	Internal	Data Manager

COC – Chain of Custody
IDW – Investigation-Derived Waste
QAPP – Quality Assurance Project Plan

35. QAPP WORKSHEET #35 - VERIFICATION (STEP IIA AND IIB) PROCESS

This worksheet presents the Data Review Process for Validation (Steps IIa and IIb). Validation procedures and criteria ensure that data are evaluated properly, completely, and consistently for use in meeting project goals.

Step IIa Validation activities ensure compliance with methods, procedures, and contracts for both sampling and analytical data. Examples of Step IIa validation activities are listed as follows:

- Data Deliverables and UFP-QAPP
- Analytes
- COC
- Holding Times
- QC acceptance criteria (blanks, surrogates, LCS, MS/MSD, serial dilutions, post digestion spikes)
- Sampling Methods and Procedures
- Field Transcription
- Analytical Methods and Procedures
- Validation Flags
- Sample Handling
- Laboratory Transcription
- Standards
- Communication
- Audits

Step IIb Validation activities ensure compliance with Measurement Performance Criteria in the UFP-QAPP for both sampling and analytical data. Some of the elements have both Step IIa and Step IIb validation activities. Examples of Step IIb validation activities are listed as follows:

- Data Deliverables and UFP-QAPP
- Deviations
- Sampling Plan
- Sampling Procedures
- Co-located Field Duplicates
- Project LOQs
- Confirmatory Analyses
- Validation Flags
- Performance Criteria (ICV, CCV, Method specific instrument performance checks (tunes, breakdown checks, instrument checks, interference checks)

Table 35-1. Validation (Step IIa and IIb) Process

Step IIa/ IIb[1]	Validation Input	Description	Responsible for Validation
IIa	Field logbook	Field logbooks will be reviewed weekly for accuracy and completeness associated with each sampling event. The inspection will be documented in daily quality control report.	Installation Lead
			Quality Manager
IIa	COC forms		Installation Lead

Table 35-1. Validation (Step IIa and IIb) Process			
Step IIa/ IIb[1]	**Validation Input**	**Description**	**Responsible for Validation**
		COC forms will be reviewed daily to ensure that project information, sample analyses requested, number of field QC samples collected, and selection of Stage 2B or 3	Quality Manager
		COC forms will be reviewed by the validator for completeness and that sample preservations are in accordance with this UFP-QAPP.	Data Validator
IIa	Sampling Methods and Procedures	Establish that the required sampling methods were used and that any deviations were noted. Ensure that the sampling procedures and field measurements met performance criteria and that any deviations were documented.	Installation Lead
			Quality Manager
IIa	Sample receipt	The sample cooler will be checked for compliance with temperature and packaging requirements listed in Worksheet #27 of this UFP-QAPP.	Laboratory Project Manager
			Project Chemist or Data Validator
IIa	Sample log-ins	Sample log-in will be reviewed for accuracy against the COC form. Sample log-ins will be reviewed by the project chemist or validator that preservation, temperature, and sample receipt conditions are in accordance with this UFP-QAPP.	Laboratory Project Manager
			Project Chemist or Data Validator
IIa	Laboratory data prior to release	Data reported are compliant with method- and project-specific QC requirements; the reported information is complete; the information in the report narrative is complete and accurate; and results are reasonable.	Laboratory Project Manager or designee
		100 percent of the data comply with the method- and project-specific requirements and that any deviations or failure to meet criteria are documented in the data package case narrative.	
		100 percent of manual entries are free of transcription errors and manual calculations are accurate; computer calculations are spot-checked to verify program validity; data reported are compliant with method- and project- specific QC requirements; raw data and supporting materials are complete; spectral assignments are confirmed; descriptions of deviations from method or project requirements are documented; significant figures and rounding have been appropriately used; reported values include dilution factors; and results are reasonable.	Laboratory Project Manager or designee
IIb	Data validation reports	All data packages will consist of sample results and summary forms for all QC samples. At least 10 percent of the data packages submitted for definitive data will be EPA Level IV (includes raw data). For definitive data results, data packages will be evaluated by undergoing data validation in accordance with USEPA Contract Laboratory Program National Functional Guidelines for Organic and Inorganic Data. Data validation reports will also be reviewed in conjunction with the project DQOs and data quality indicators. Data validation reports will include validation of holding time, sample handling, analytes, analytical methods, and laboratory performance criteria. Field duplicate and field blanks results will be identified, and any outlier will be discussed.	Data Validator

Table 35-1. Validation (Step IIa and IIb) Process			
Step IIa/ IIb[1]	**Validation Input**	**Description**	**Responsible for Validation**
		The following validation flags will be used for all validation: • J – Result is estimated • U – Analyte is not detected at or above the stated MDL • R – Result is rejected and the data are unusable • UJ – Analyte is not detected, but there is an uncertainty concerning the reported value In addition, reason codes will be applied to describe the reason why a validation flag was assigned. Reason codes will be explained in the data validation reports and will be uploaded to the MS Excel EDD.	Data Validator

Note: Project Personnel are identified in Worksheet #7
[1] IIa=compliance with methods, procedures, and contracts; IIb=comparison with measurement performance criteria in this UFP-QAPP
[2] Stages of data validation are defined in Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use (USEPA 2009)
COC – Chain of Custody
DQO – Data Quality Objectives
EDD- Electronic Data Deliverable
LOD – Limit of Detection
QC – Quality Control
UFP-QAPP – Uniform Federal Policy-Quality Assurance Project Plan
USEPA – United States Environmental Protection Agency

36. QAPP WORKSHEET #36 - ANALYTICAL DATA VALIDATION (STEPS IIA AND IIB) SUMMARY

This worksheet identifies the criteria that will be used to validate data under Steps IIa and IIb.

Table 36-1. Analytical Data Validation (Steps IIa and IIb) Summary

Step IIa/ IIb	Matrix	Analytical Group	Validation Criteria	Data Validator
IIa	Soil/Sediment	VOCs	8260C/5035A-FW, and EPA CLP guidelines	LDC
IIa	Soil/Sediment	SVOCs,	8270D/3546, and EPA CLP guidelines	LDC
IIa	Soil/Sediment	Pesticides	8081B, and EPA CLP guidelines	LDC
IIa	Soil/Sediment	PCBs (as Aroclors),	8082A, and EPA CLP guidelines	LDC
IIa	Soil/Sediment	PCB (as Congeners)	1668A/HRMS_Sox_P, and EPA CLP guidelines	LDC
IIa	Soil/Sediment	Dioxins/Furans	8290A/8290_P_Sox, and EPA CLP guidelines	LDC
IIa	Soil/Sediment	TAL Metals	6020B/3050B, and EPA CLP guidelines	LDC
IIa	Soil/Sediment	Mercury	7471B/7471B_Prep, and EPA CLP guidelines	LDC
IIa	Soil/Sediment	Cyanide	9012B/9012B_Prep, and EPA CLP guidelines	LDC
IIb	Surface water/Pore water	VOCs	8260C/5035A-FW, and EPA CLP guidelines	LDC
IIb	Surface water/Pore water	SVOCs,	8270D/3546, and EPA CLP guidelines	LDC
IIb	Surface water/Pore water	Pesticides	8081B, and EPA CLP guidelines	LDC
IIb	Surface water/Pore water	PCBs (as Aroclors),	8082A, and EPA CLP guidelines	LDC
IIb	Surface water/Pore water	PCB (as Congeners)	1668A/HRMS_Sox_P, and EPA CLP guidelines	LDC
IIb	Surface water/Pore water	Dioxins/Furans	8290A/8290_P_Sox, and EPA CLP guidelines	LDC
IIa	Surface water/Pore water	TAL Metals	6020B/3050B, and EPA CLP guidelines	LDC
IIb	Surface water/Pore water	Mercury	7471B/7471B_Prep, and EPA CLP guidelines	LDC
IIb	Surface water/Pore water	Cyanide	9012B/9012B_Prep, and EPA CLP guidelines	LDC

CLP – Contract Laboratory Program; refers to:
National Functional Guidelines for Organic Superfund Methods Data Review (USEPA 2017);
National Functional Guidelines for Inorganic Superfund Methods Data Review (USEPA 2017);
National Functional Guidelines for High Resolution Superfund Methods Data Review (USEPA 2016);
EPA – United States Environmental Protection Agency
LDC – Laboratory Data Consultant, LLC
PCBs – Polychlorinated Biphenyls
SW-846 – Test Methods for Evaluating Solid Waste, Physical/Chemical Methods
SVOCs – Semi-volatile Organic Compounds
TAL – Target Analyte List
VOC – Volatile Organic Carbon

37. QAPP WORKSHEET #37 - USABILITY ASSESSMENT

The usability assessment is an evaluation of data based upon the results of data validation and verification for the decisions being made. In the usability step, reviewers assess whether the process execution and resulting data meet quality objectives based on criteria established in this UFP-QAPP. The usability assessment will consider data from sampling activity, on-site analytical, off-site laboratory, and validation reports. The usability assessment will be performed by the data assessment team and documented in the RI Report by the Project Chemist. The data assessment team will consist of the Project Manager, Quality Manager, Project Chemist, and Database Manager. In addition, other project personnel (e.g., Installation Points of Contact or their representative, state regulator, Installation Lead) may be involved with the determination of whether data meet project quality objectives.

The data assessment team will:

- Identify project requirements and verify field activities were performed in accordance with the SOPs (Appendix B) detailed in Worksheets #14 and #21.
- Review the project DQOs and data validation process detailed in Worksheet #34, #35, and #36.
- Verify that all samples and analytical data collected meet the DQOs.
- Evaluate validated data to assess if it satisfies DQOs (e.g., tolerable limits on decision errors) and is adequate to make the decision regarding additional investigation for the site.
- Provide input on the suitability of the results for the purposes intended.

In the Usability Assessment, determine the impacts of any deviations from the planned procedures documented in this UFP-QAPP, guidance documents, or SOPs, for the following items:

- Sampling Locations;
- Holding Times;
- SOPs and Methods;
- COC; and
- Damaged Samples.

In addition, evaluate the possible effects of outliers or anomalous data from the following:

- QC Samples;
- Comparability;
- Background;
- Matrix;
- Completeness;
- Critical Samples; and
- Meteorological Data and Site Conditions.

These considerations for the Usability Assessment are discussed in detail in Section 5.2.3.2 of the UFP-QAPP Manual (USEPA 2005). The usability assessment will include an evaluation of the DQIs (precision, accuracy/bias, representativeness, comparability, completeness, and sensitivity). The impact of any data gaps or deviations from planned procedures will be evaluated. This includes rejected data based on the results of the data validation process. The usability assessment will evaluate the overall dataset for the entire site and any trends, relationships, or correlations will be described.

After the data usability assessment has been performed, data deemed appropriate for use will be presented in the RI Report. The RI Report will include conclusions and optimization recommendations, as applicable.

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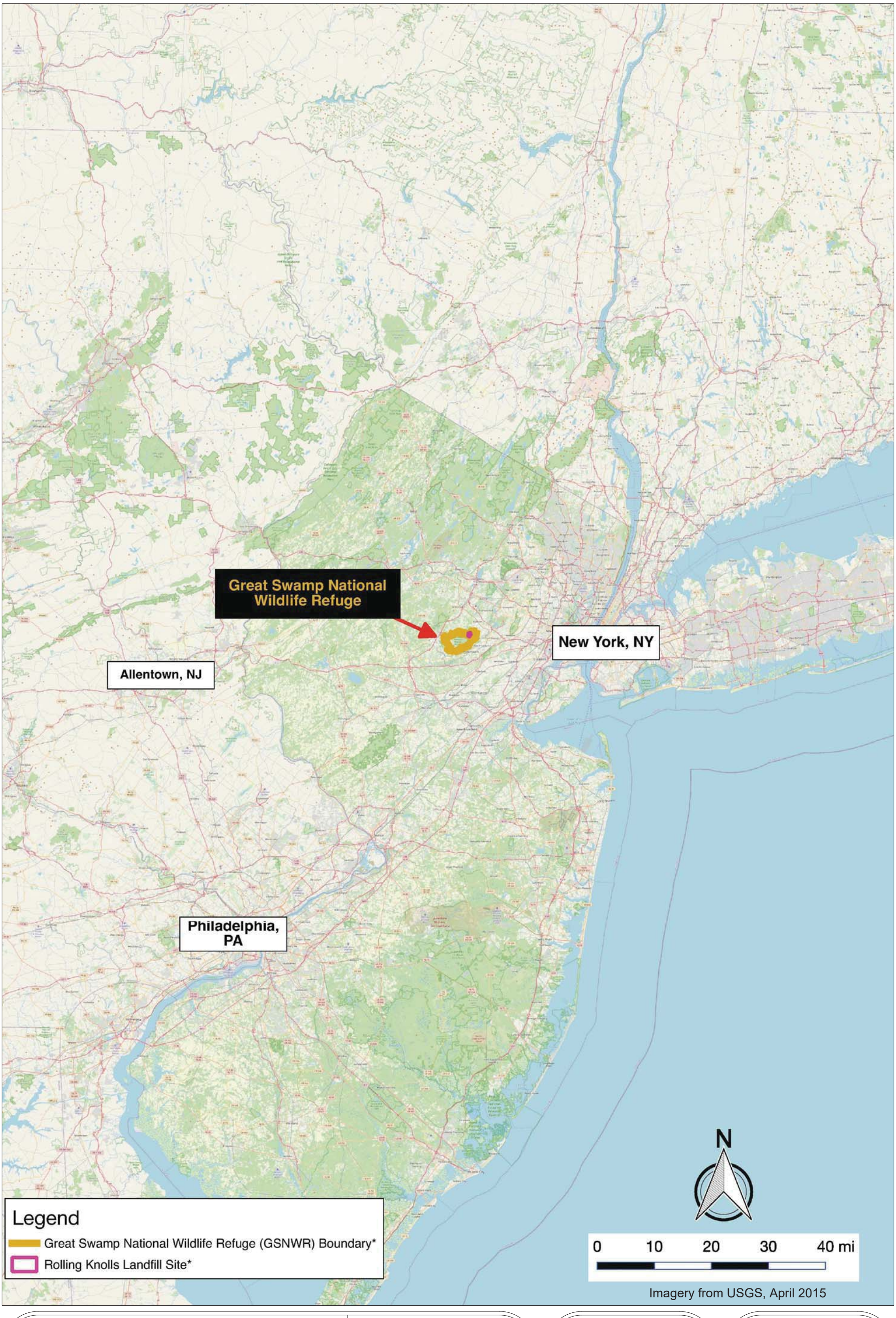
Figures



PROJECT NAME:
ROLLING KNOLLS DATA GAP INVESTIGATION

SECTION:
SITE LOCATION MAP

REVISION DATE:
9/1/2020

FINAL

DRAWING NO.
FIG 1

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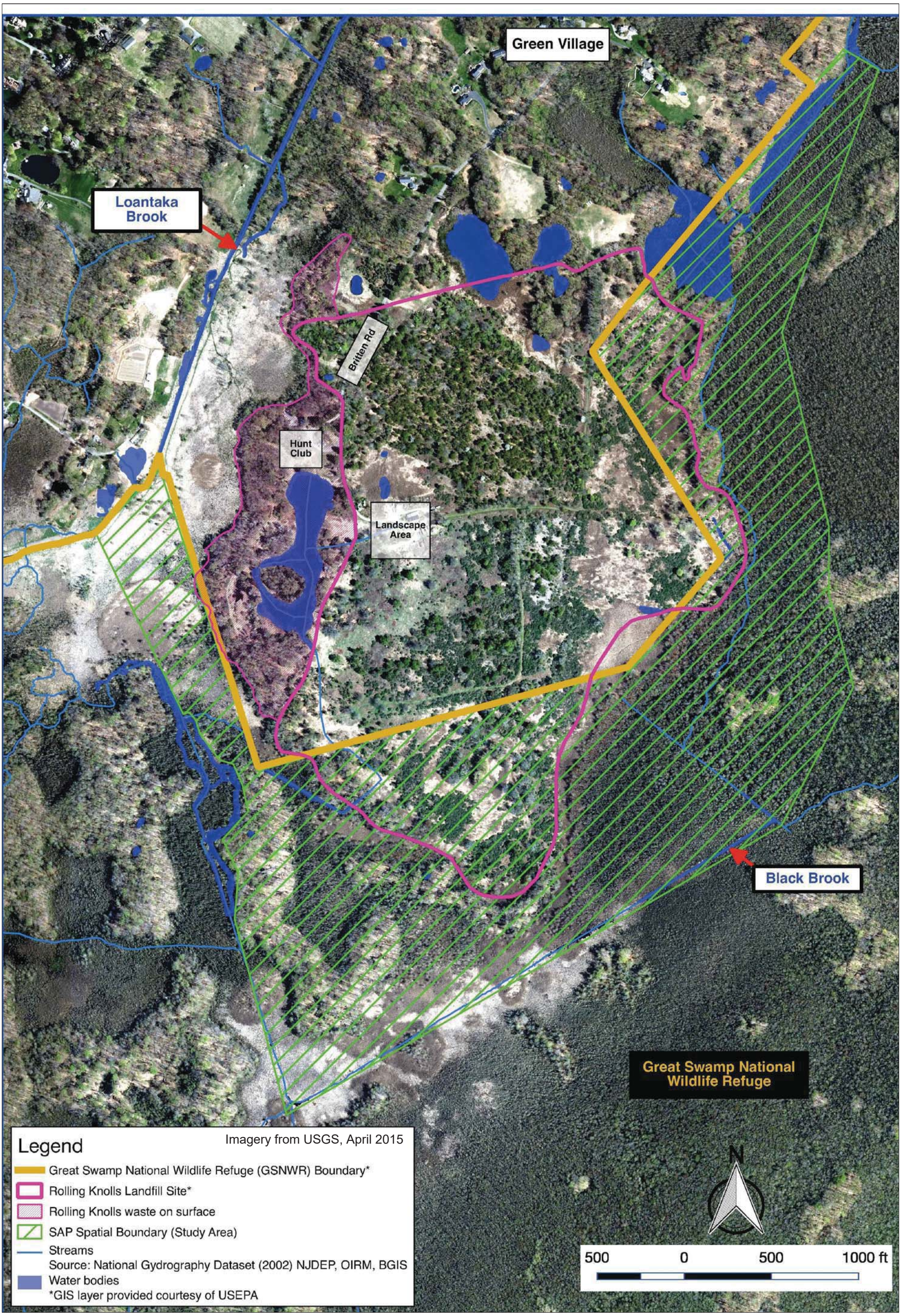
PRESTON SOWELL



PROJECT NAME:
ROLLING KNOLLS DATA GAP INVESTIGATION

SECTION:
SITE MAP

REVISION DATE:
9/1/2020

FINAL

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FIG 2

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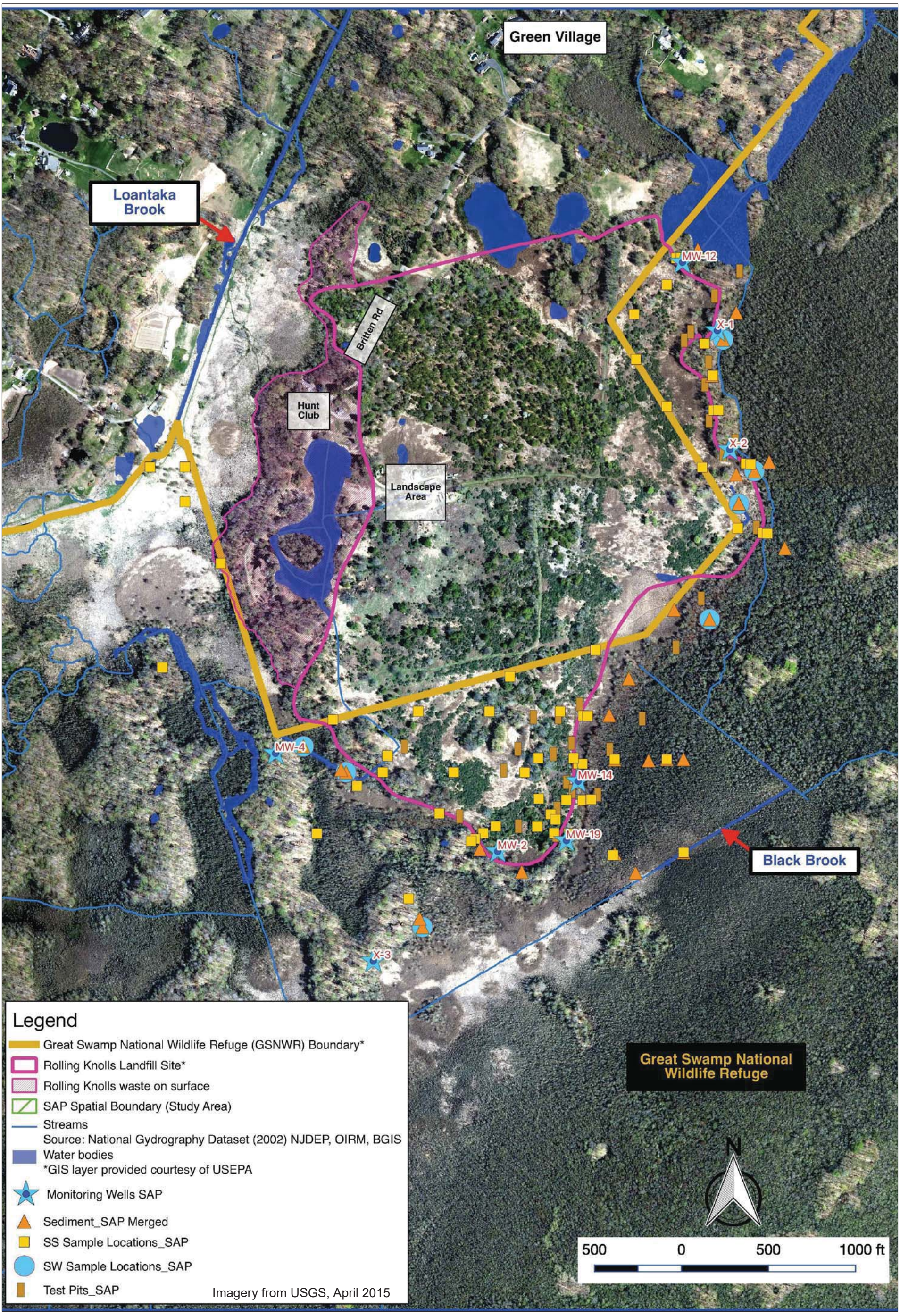

PROJECT NAME:
ROLLING KNOLLS DATA GAP INVESTIGATION

SECTION:
RI AND BERA SAMPLING LOCATIONS IN STUDY AREA

REVISION DATE:
9/1/2020

FINAL

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FIG 3

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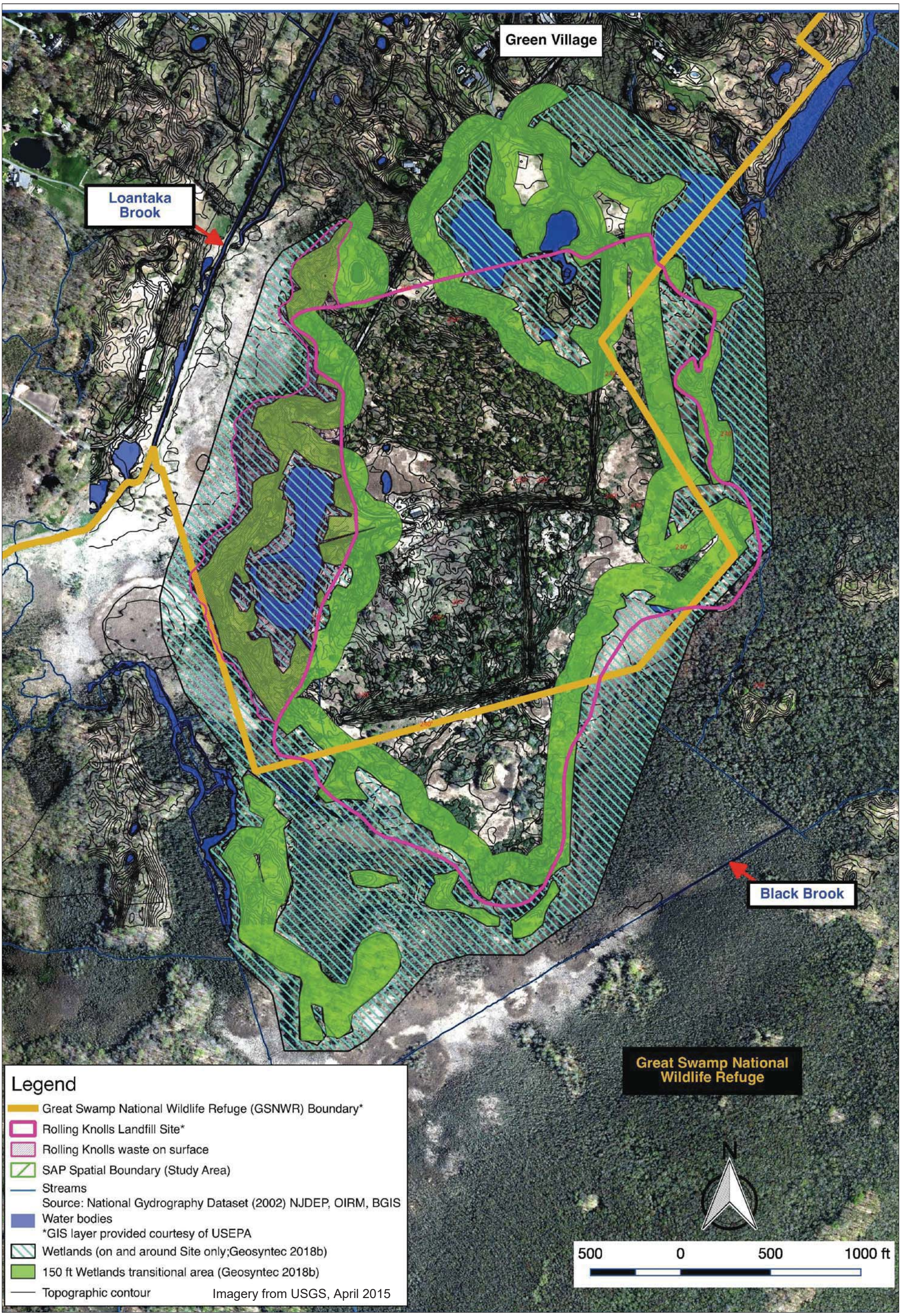

PROJECT NAME:
ROLLING KNOLLS DATA GAP INVESTIGATION

SECTION:
WETLANDS AND TOPOGRAPHY

REVISION DATE:
9/1/2020

FINAL

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FIG 4

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West

East

WILDLIFE REFUGE

LANDFILL WASTE

Infiltration to groundwater

overland flow

contaminated groundwater

contaminated sediments

contaminated Surface water?

impermeable clay

NOT TO SCALE

REVISIONS:

No. 1 _____ DATE _____ INITIALS _____

No. 2 _____ DATE _____ INITIALS _____

No. 3 _____ DATE _____ INITIALS _____

DESIGN: P. SOWELL 9/1/2020 DATE

DRAWN: P. SOWELL 9/1/2020 DATE

CHECKED: - - DATE

PROJECT NAME
ROLLING HILLS SUPP. RI/FS

SECTION
CONCEPTUAL SITE MODEL

REVISION DATE
9/1/2020

FINAL

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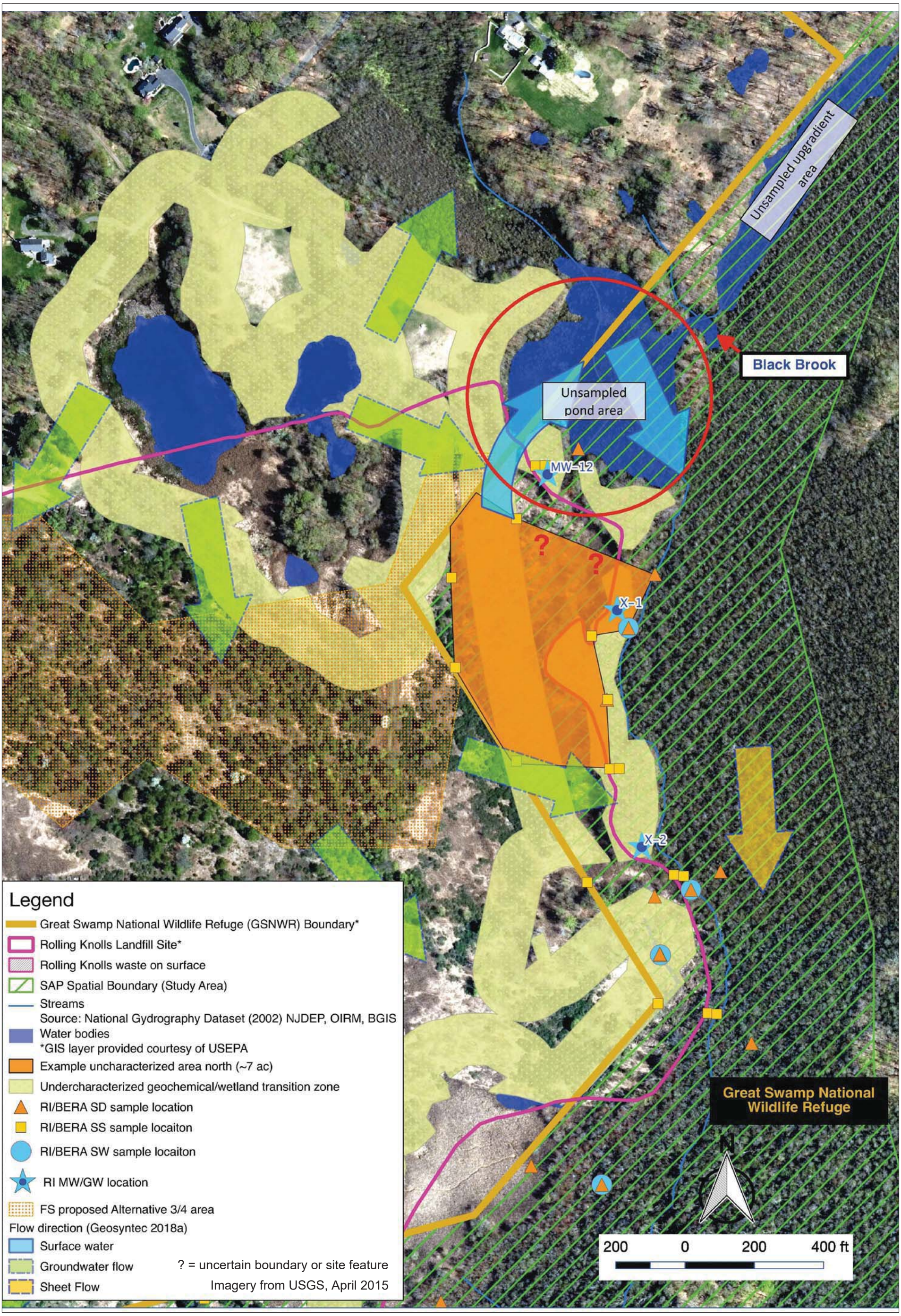
FIG 5

SHEET **1** OF **1**



PROJECT NAME:
ROLLING KNOLLS DATA GAP INVESTIGATION

SECTION:
DATA GAP AREAS - NORTH

REVISION DATE:
9/1/2020

FINAL

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FIG 6A

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Legend

- Great Swamp National Wildlife Refuge (GSNWR) Boundary*
- Rolling Knolls Landfill Site*
- Rolling Knolls waste on surface
- SAP Spatial Boundary (Study Area)
- Streams
  Source: National Gydrography Dataset (2002) NJDEP, OIRM, BGIS
- Water bodies
  *GIS layer provided courtesy of USEPA
- Example uncharacterized area south (~7.5 ac)
- Undercharacterized geochemical/wetland transition zone
- RI/BERA SD sample location
- RI/BERA SS sample locaiton
- RI/BERA SW sample locaiton
- RI MW/GW location

Flow direction (Geosyntec 2018a)

- Surface water
- Groundwater flow
- Sheet Flow

Imagery from USGS, April 2015

PROJECT NAME:
ROLLING KNOLLS DATA GAP INVESTIGATION

SECTION:
DATA GAP AREAS - SOUTH

REVISION DATE:
9/28/2020

FINAL

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FIG 6B

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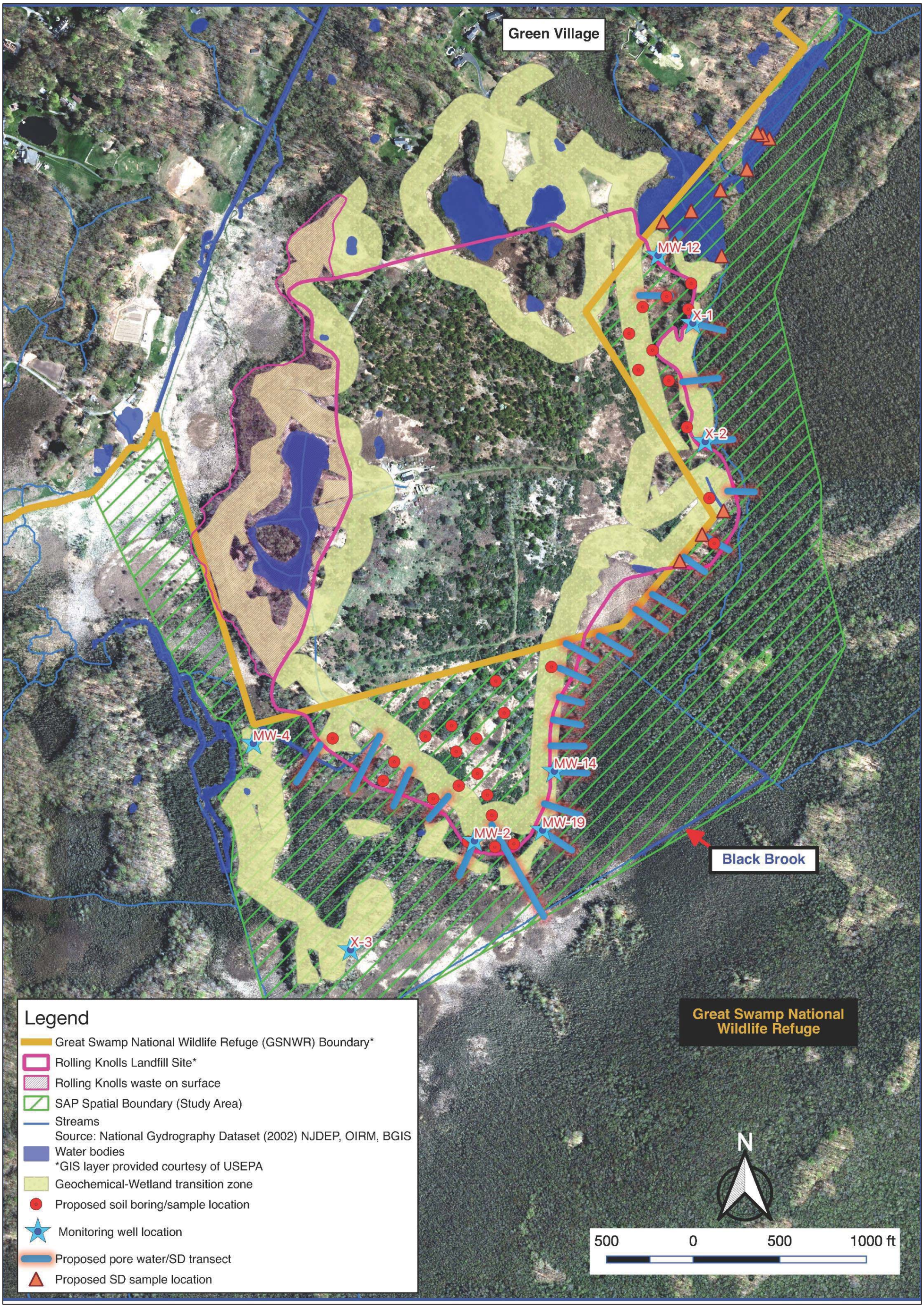

PROJECT NAME:
ROLLING KNOLLS DATA GAP INVESTIGATION

SECTION:
PROPOSED DATA GAP SAMPLING LOCATIONS - OVERVIEW*

REVISION DATE:
9/28/2020

FINAL

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FIG 7

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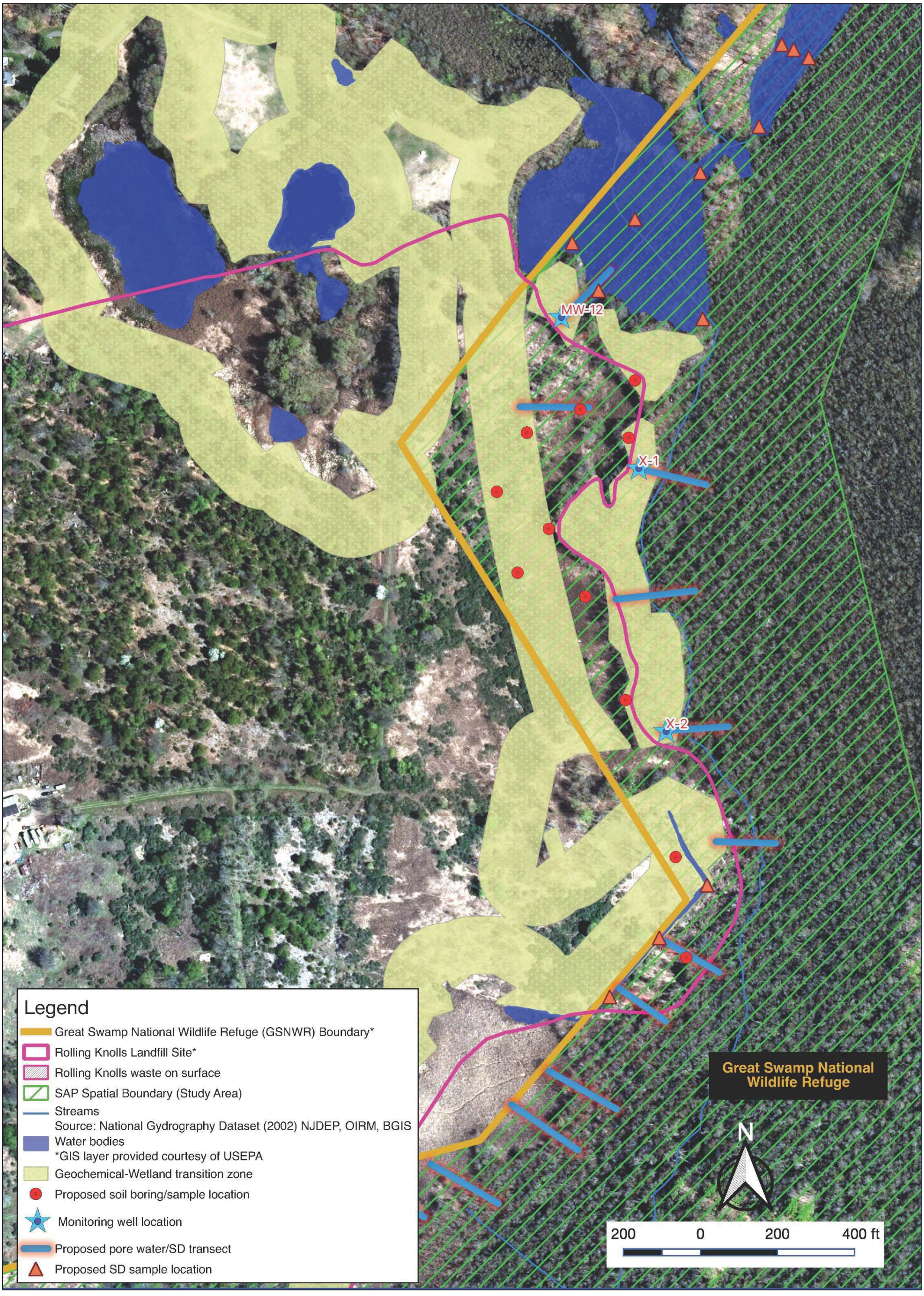

PROJECT NAME:
ROLLING KNOLLS DATA GAP INVESTIGATION

SECTION:
PROPOSED DATA GAP SAMPLING LOCATIONS - NORTH*

REVISION DATE:
9/28/2020

FINAL

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FIG 8A

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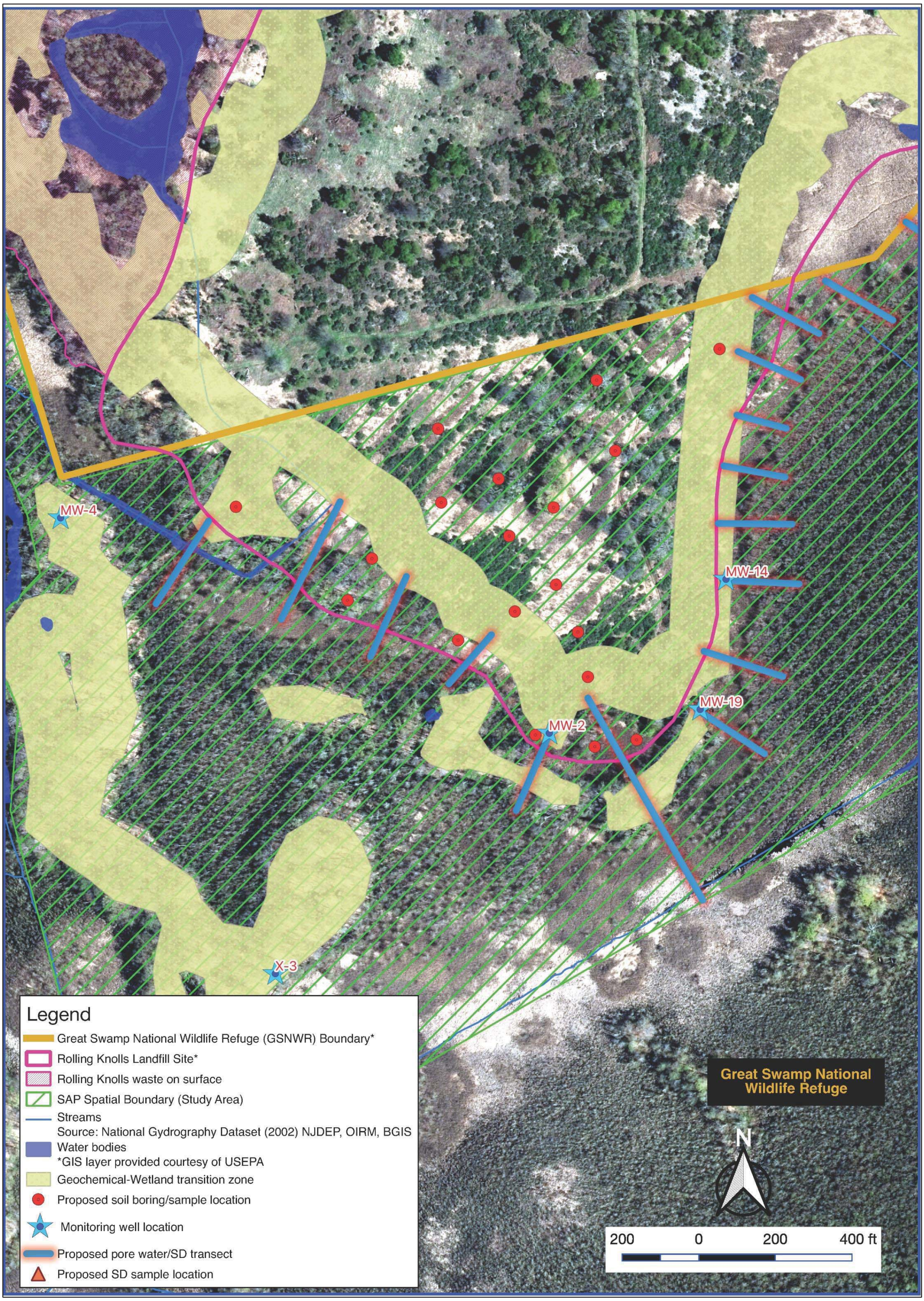

PROJECT NAME:
ROLLING KNOLLS DATA GAP INVESTIGATION

SECTION:
PROPOSED DATA GAP SAMPLING LOCATIONS - SOUTH*

REVISION DATE:
9/28/2020

FINAL

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FIG 8B

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Appendix A – Scoping Meeting Minutes

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W: www.ap-in.com

First DQO Call Summary
Thursday, August 13, 2020
Rolling Knolls Supplemental RI/FS

Project Title:	Rolling Knolls Supplemental RI/FS
Project Site:	Great Swamp National Wildlife Refuge, Chatham County, New Jersey
Contract Number:	GS-10F-026BA
Delivery Order Number:	140F0520F0195
Amendment Number:	
Applied Intellect Project Manager:	Jeff Hart
FWS Contracting Officer:	Christine Beauregard, FWS
FWS Contracting Officer's Representative:	George Molnar
Contract Period of Performance:	August 1, 2020– April 30, 2021
Reporting Period:	August 13, 2020

SUMMARY OF WORK ACCOMPLISHED

The meeting was conducted as a teleconference using Microsoft (MS) Teams and began at 13:30EDT/11:30MDT/10:30PT.

Attendees:
George Molnar, US Fish and Wildlife Service (FWS) Contracting Officer's Representative (COR);
Jeff Hart, Applied Intellect, LLC (AI) Project Manager (PM)
Preston Sowell, AI Senior Environmental Scientist
David Back, AI Senior Hydrogeologist
Linda Ziccardi, AI Sr Ecological Risk Assessor
Bernard Kronschnabel, AI Sr Environmental Engineer

Mr. Molnar, FWS COR introduced the meeting. In response to questions. Mr. Molnar indicated that the objectives of the FWS was to fill data gaps associated with soil, sediment and groundwater contaminant onto the Refuge from the Rolling Knolls Landfill and to use standards developed in the Responsible Party (RP) CERCLA documents rather than to conduct additional biota samples and conduct a detailed risk assessment.

Following Mr. Molnar's introduction, Mr. Hart, AI PM presented the AI Agenda:

1. Present and discuss the AI's understanding of FWS preliminary DQOs based on the SOW and AI's proposal in Uniform Federal Policy for Quality Assurance Program (UFP-QAPP), Worksheet 11 format;
2. Present considerations for laboratory analytical methods to be utilized for the site characterization.

Mr. Sowell presented AI' preliminary UFP-QAPP worksheet 11 discussions while the text of WS 11 was provided onscreen for the teleconference attendees. The objective of the meeting was to begin developing the project quality objectives (PQOs) using a systematic planning process based on EPA's 7-step DQO process.

1. State the Problem.
2. Identify the Goal of the Investigation.
3. Identify the Information Inputs.
4. Define the Boundaries of the Investigation.
5. Develop the Analytic Approach.
6. Specify Performance or Acceptance Criteria.
7. Develop the Plan for Obtaining Data.

Mr. Sowell defined the "Refuge portion of the Site" as the portion Great Swamp National Wildlife Refuge (Refuge) that is located on the Rolling Knolls Superfund Site as defined by the Responsible Party (RP) in previous CERCLA documents. Mr. Sowell defined "adjacent portions of the Refuge" as the area of the Refuge that has significant potential for contaminants of concern migrating from the Site onto the non-Site portions of the Refuge.

Goals of the Study include determining the distribution and concentration of contaminants located on the Refuge portion of the Ste and adjacent portions of the Refuge in:

- Sediment;
- Surface water
- Pore water
- Groundwater and
- Soil including uncharacterized surface soil and subsurface soil.

They also include:
Determining the physical, chemical, and geotechnical properties of the soil and wastes within the landfill on the Refuge portion of the site, and the clay layer lying beneath the landfill;

Data to be collected were presented in the following table.

Data Type	Data Purpose
Sediment contaminant concentrations collected from open surface water bodies and/or surface water drainage swales within the Refuge portion of the Site and adjacent Refuge property.	Evaluation of potential migration pathways, and the spatial extent and relative concentrations of Site-related contaminants .
Sediment contaminant concentrations from upgradient of the Refuge portion of the Site and adjacent Refuge property.	Evaluation of potential upgradient sediment contaminant contributions.
Survey to identify potential groundwater discharge points within the Refuge portion of the Site and adjacent Refuge property.	Evaluation of potential groundwater discharge points to surface water and to optimize pore water/sediment/surface water sampling locations.
Sediment pore water and collocated sediment contaminant concentrations from areas adjacent to the Site where contaminated groundwater is expected to discharge.	Evaluation of contaminated groundwater potentially discharging into Refuge wetlands.
Surface water contaminant concentrations from within the Refuge portion of the Site and adjacent Refuge property.	Evaluation of potential migration pathways, and the spatial extent and relative concentrations of Site-related contaminants .
Surface water contaminant concentrations from upgradient of the Refuge portion of the Site.	Evaluation of potential upgradient surface water contaminant contributions.
Surface water flow measurements from upgradient, within the Refuge portion of the Site and adjacent Refuge property.	Evaluation of the relative contributions of groundwater to surface water flow.

Data Type	Data Purpose
Groundwater water-level elevation measurements from within the Refuge portion of the Site and adjacent Refuge property.	Evaluation of groundwater flow directions.
Aquifer hydraulic conductivity measurements from within the Refuge portion of the Site and adjacent Refuge property.	Evaluation of groundwater flow velocities and contaminant mass-flux.
Groundwater contaminant concentrations from within the Refuge portion of the Site and adjacent Refuge property.	Evaluation of potential migration pathways, and the spatial extent and relative concentrations of Site-related contaminants in groundwater and where contaminated groundwater might be discharging into surface water. This information will also enable pre-Remedial implementation baseline conditions to be established.
Groundwater quality parameters from within the Refuge portion of the Site and adjacent Refuge property.	Evaluation of overall groundwater quality and provides an indication of the presence of, and types of contaminants potentially present in groundwater. This information will also enable pre-Remedial implementation baseline conditions to be established.
Speciation of metals (specifically Fe and Mn) in groundwater from within the Refuge portion of the Site and adjacent Refuge property.	Provides an indication of the oxidation/reduction potential (ORP) of groundwater flowing through the landfill wastes and of the implications of changing that environment through implementation of a remedial alternative.
Surface soil contaminant concentrations from within the Refuge portion of the Site.	Evaluation of potential hotspots/source areas, migration pathways, and the spatial extent and relative concentrations of Site-related contaminants.
Subsurface soil and waste contaminant concentrations and fraction of organic carbon from within the Refuge portion of the Site (i.e., the landfill on the Refuge portion of the Site).	Determine physical, chemical, and geotechnical properties of the soil and wastes to evaluate fate and transport of contaminants and the leaching potential of the landfill wastes, and identify potential hotspots/source areas, and provide information necessary for remedial design and implementation.

Data Type	Data Purpose
Subsurface soil contaminant concentrations below the landfill in the Refuge portion of the Site to the upper bound of the underlying clay layer.	Determine whether the soils beneath the landfill wastes have been contaminated and whether they would be classified as a hazardous waste.
Subsurface soil and waste, physical, chemical, and geotechnical properties from within the Refuge portion of the Site.	Provides information necessary for remedial design and implementation.
GPS coordinates of sample locations	Documentation of sample locations and evaluation of the spatial distribution of contamination across the site.
Survey coordinates for monitoring wells sampled.	Data necessary for groundwater gradient determination.
Visual descriptions of soils at soil boring locations	Provides a potential visual indication of waste material and mineral speciation (i.e., color associated with oxidation/reduction) and for saturated intervals indicating groundwater contact with material. Also provides information for the estimation of potential volumes of contaminated material.
Topography/geomorphology	Evaluation of potential migration pathways from on-Site source areas. (e.g., drainage patterns, drainage areas, pooling areas, etc.)
Aerial and satellite imagery	Allows remote identification of possible surface water pooling areas and flow regimes, vegetation changes, and optimization of sample placement planning and access to sample locations.

Study Boundaries include the following temporal boundaries:

- Based on FWS schedule to complete on April 30, 2020, field work is anticipated for October 2020 to avoid difficulties associated with winter sampling

Spatial boundaries include:

- Both onsite and adjacent portions of the Refuge
- FWS will work with the RPs to determine if private portions of the Site can be accessed and sampled, which will be important in evaluating groundwater gradients and potential flow patterns onto the Refuge from the private portion of the Site

- Upgradient portion of surface water and sediment inflow areas directly adjacent to the site to better define the relevant background concentrations of contaminants for the study area.

For the analytical approach, Mr. Hart identified that AI is working with Eurofins/TestAmerica (ETA) to conduct the laboratory analysis for the project. ETA laboratories conducted Contract Laboratory Program (CLP) as part of the EPA Superfund requirements for the RPs to identify the following contaminant distribution and concentrations in soil/sediment and surface water, pore water, and groundwater, as shown in Table 2-6 of the RP's remedial investigation (RI)

Table 2-6
Target Analytes and Analytical Methods
Remedial Investigation Report
Rolling Knolls Landfill Superfund Site
Chatham, New Jersey

Geosyntec Consultants

Matrix Type	Parameter	Analytical Method[1]	Laboratory
Soil	TCL VOCs[2]	USEPA SOM01.1/SOM01.2	Shealy Environmental Services, TestAmerica Laboratories, Inc.
	TCL SVOCs[2]	USEPA SOM01.1/SOM01.2	
	TCL SVOCs (SIM)	USEPA SOM01.1/SOM01.2	
	TCL PCBs	USEPA SOM01.1/SOM01.2	
	TCL Pesticides[3]	USEPA SOM01.1/SOM01.2	
	TAL Inorganics	USEPA ILM05.3/ILM05.4	
	pH	USEPA 9045C	
	TOC	Lloyd Kahn	
	Grain Size	ASTM D-422	
	PCB Congeners	USEPA 1668A/1668C	TestAmerica Laboratories, Inc.
	Dioxins and Furans	USEPA 1613/1613B	
	TAL Inorganics	USEPA ISM01.3	
	Moisture	Moisture	
Sediment, and Potential Industrial Waste	TCL VOCs[2]	USEPA SOM01.1/SOM01.2	Shealy Environmental Services, TestAmerica Laboratories, Inc.
	TCL SVOCs[2]	USEPA SOM01.1/SOM01.2	
	TCL SVOCs (SIM)	USEPA SOM01.1/SOM01.2	
	TCL PCBs	USEPA SOM01.1/SOM01.2	
	TCL Pesticides[3]	USEPA SOM01.1/SOM01.2	
	TAL Inorganics	USEPA ILM05.3/ISM01.3	
	pH	USEPA 9045C/9045D	
	TOC	Lloyd Kahn	
	Grain Size	ASTM D-422	
	PCB Congeners	US EPA 1668C	TestAmerica Laboratories, Inc.
	Dioxins and Furans	US EPA 1613B	
	Moisture	Moisture	
Groundwater and Surface Water	TCL VOCs[2]	USEPA SOM01.1/SOM01.2	Shealy Environmental Services, TestAmerica Laboratories, Inc.
	TCL VOCs (SIM)	USEPA SOM01.1	
	TCL SVOCs[2]	USEPA SOM01.1/SOM01.2	
	TCL SVOCs (SIM)	USEPA SOM01.1/SOM01.2	
	TCL PCBs	USEPA SOM01.1/SOM01.2	
	TCL Pesticides[3]	USEPA SOM01.1/SOM01.2	
	TAL Inorganics	USEPA ILM05.3/ISM01.3	
	Hardness	USEPA 130.1	
Surface Water	Low-Level Mercury	USEPA 1631E	Shealy Environmental Services
	Low Level Mercury	USEPA 1631E	TestAmerica Laboratories, Inc.
	Hardness as $CaCO_3$	SM 2340C-1997	
Air	VOCs	USEPA TO-15	Test America Laboratories, Inc.

Based on discussion with the FWS, this RI will not be conducted under CLP protocol. Rather, the same contaminans will be analyzed using SW846 methods with similar or lower method detection limits and rteporting limits. These include:

Method Description	Method Code	Prep Method
Volatile Organic Compounds by GC/MS	8260C	5035A_FW
Semivolatile Organic Compounds (GC/MS)	8270D	3546
Semivolatile Organic Compounds by GC/MS - Low Level	8270D_LL	3541_LL
Organochlorine Pesticides (GC)	8081B	3546
Metals (ICP/MS)	6020B	3050B
Mercury (CVAA)	7471B	7471B_Prep
Cyanide, Total andor Amenable	9012B	9012B_Prep
Dioxins and Furans (HRGC/HRMS)	8290A	8290_P_Sox
Chlorinated Biphenyl Congeners (HRGC/HRMS)	1668A	HRMS_Sox_P

In addition to these SW846 methods, AI is proposing the following analyses to support sediment contaminant fate and transport assessment.

- Simultaneously Extracted Metals (SEM): Simultaneously Extracted Metals (SEM) by Acid Volatile Sulfide (AVS)/SEM and EPA Method 6010C; and
- Acid Volatile Sulfide (AVS): by AVS/SEM and EPA Method 9034.

AI is scheduled to conduct a site visit to the Refuge on August 18, 2020. Mr. Molnar will meet after the meeting to discuss the specifics with Mr. David Back, who will be attending the site walk as a representative for AI on August 18, 2020.

The meeting was adjourned at 1500EDT/1300MDT/1200PDT.

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Second DQO Call Summary
Friday, August 21, 2020
Rolling Knolls Supplemental RI/FS

Project Title:	Rolling Knolls Supplemental RI/FS
Project Site:	Great Swamp National Wildlife Refuge, Chatham County, New Jersey
Contract Number:	GS-10F-026BA
Delivery Order Number:	140F0520F0195
Amendment Number:	
Applied Intellect Project Manager:	Jeff Hart
FWS Contracting Officer:	Christine Beauregard, FWS
FWS Contracting Officer's Representative:	George Molnar
Contract Period of Performance:	August 1, 2020– April 30, 2021
Reporting Period:	August 21, 2020

SUMMARY OF WORK ACCOMPLISHED

The meeting was conducted as a teleconference using Microsoft (MS) Teams and began at 10:00EDT/08:000MDT.

Attendees:
George Molnar, US Fish and Wildlife Service (FWS) Contracting Officer's Representative (COR)
Graham Taylor, FWS, Refuge Supervisor-North Zone, North Atlantic-Appalachian Region
Michael Horne, FWS, Refuge Manager, Lenape National Wildlife Refuge Complex
Jeff Hart, Applied Intellect, LLC (AI) Project Manager (PM)
Preston Sowell, AI Senior Environmental Scientist
David Back, AI Senior Hydrogeologist

This call was focused on access issues associated with getting brush cleared along access pathways to sampling locations for field work in late autumn. It is a follow-up to Applied Intellect, LLC's site visit on Tuesday August 18, 2020. Mr. David Back, Sr Hydrogeologist accompanied Mr. George Molnar into the Refuge utilizing the FWS MarshMaster and collected photos and video imagery of the overgrowth. AI has had discussions internally about potential solutions to providing bush clearance including Skid-steer-mounted Bush Hog (ST-BH) for dryer land, and MarshMaster-mounted mowers (MM-M) for wet areas.

Dave Back discussed possible subcontractors for both SM-BH work and MM-M. He is in communication with several potential subcontractors and hopes to have mobilization and daily rates by midweek next week for these activities.

Mr. Horne indicated that the SM-BH is the most efficient tool for relatively dry firm ground, but the MM-m will be likely necessary for wetter and saturated ground. The FWS indicated that they would strongly consider conducting the clearance work internally and have access to both tools, but requested a clear map of the sampling locations, including GPS coordinates of important features, including onsite monitoring wells (MWs), and other desired sampling locations.

- Extra attention must be paid to locating the MWs prior to clearing and putting up clearly visible barriers around each MW to ensure they are not destroyed during the clearing activity.

- AI will move forward with identifying the best subcontractors and unit costs and providing thus information to the FWS for cost-benefits decision-making.

- New MM-M have front mowers, which may be more efficient. FWS system have back mowers that require forward and backward mowing.

- Direct Push technology (DPT)Subcontractors will utilize track-mounted systems that must be hauled to the Site on truck-mounted trailer, therefore access to north western portions of the Refuge would benefit from access through the northern ball-park/shooting range. The FWS will request access from the land manager (Greenfield Fire Department?).

- AI is developing sampling location maps and will provide these to the FWS by mid to late next week (August 26-27, 2020), along with unit rate costs for land clearance subcontractors. The land clearing subcontracting will likely move forward to ensure the project has a backup for FWS clearing.

- FWS will consider internal strategy of mowing/land clearing soon after getting the sampling maps in order to get the clearing done by the time the sampling program begins in late October 2020: get bulk of the work done early and final clearing can be done internally or by AI subcontractor directly prior to fieldwork.

The team briefly discussed whether Project Quality Objectives include analysis of emerging contaminants that were not evaluated by the RP, including PFOS/PFOA perfluorinated compounds, and PCB congeners. The FWS chose to have initial discussions internally before these discussions and it was determined that this issue would be addressed next week.

Action Items:

- AI will develop list of clearing contractors and unit rates to ensure FWS has a backup and comparison of costs for decision-making;
- AI will develop sampling location map to identify bush clearing access pathways to sample locations for FWS and subcontractor review;
- FWS will contact Greenfield Fire Dept about getting access to the northwestern Refuge through the ball park and shooting range;
- FWS will discuss whether additional emerging contaminants should be analyzed in multimedia samples for this site characterization; and
- AI will set up a DQO call for Wednesday or Thursday next week with the FWS to further develop DQOs based on discussions today and discussions last week at the initial DQO call on August 13, 2020.

The meeting was adjourned at 10:30ET/8:30MT.

Appendix B – Standard Operating Procedures
Applied Intellect, LLC

ACRONYMS AND ABBREBVIATIONS

ASTM	American Society for Testing and Materials
bgs	below ground surface
°C	degrees Celsius
CFR	Code of Federal Regulation
COC	chain of custody
COR	contracting officer representative
DNAPL	dense non-aqueous phase liquid
DOT	Department of Transportation
DRO	diesel range organics
DO	dissolved oxygen
DQO	data quality objective
EAL	Environmental Action Level
EC	electrical conductivity
EPA	Environmental Protection Agency
°F	degrees Fahrenheit
ft	feet
FID	flame ionization detector
FTL	Field Team Leader
gal/ft3	gallons per cubic foot
GPR	ground penetrating radar
GPS	global positioning system
GRO	gasoline range organics
HASP	Health and Safety Plan
HSA	hollow-stem auger
IAW	in accordance with
ID	Inside Diameter
IDW	investigation-derived waste
LIMS	laboratory information management system
LNAPL	light non-aqueous phase liquid ml/L milliliters per liter
MS/MSD	matrix spike/matrix spike duplicate
NAPL	non-aqueous phase liquid
NTU	nephelometric turbidity unit
OD	outside diameter
ORP	oxidation/reduction potential
OSHA	Occupational Safety and Health Administration
OVA	organic vapor analyzer
PAH	polynuclear aromatic hydrocarbon
PID	photoionization detector
PM	Project Manager
POL	petroleum oils and lubricants
PVC	polyvinyl chloride

QA	quality assurance
QA/QC	quality assurance/quality control
QAPP	quality assurance project plan
QC	quality control
RCRA	Resource Conservation and Recovery Act
RG	Registered Geologist
RI/FS	Remedial Investigation/Feasibility Study
RLS	registered land surveyor
RPM	Remedial Project Manager
SOP	Standard Operating Procedure
SOW	statement of work
SP	spontaneous potential
SU	standard unit
SVOC	semivolatile organic compound
TCLP	toxicity characteristic leaching procedure
TOC	total organic carbon
TPH	total petroleum hydrocarbon
TVH	total volatile hydrocarbons
UFP-QAPP	Uniform Federal Policy Quality Assurance Project Plan
µg/L	micrograms per liter
USCS	Unified Soil Classification System
UTM	Universal Transverse Mercator
VOC	volatile organic compound
WMMP	Waste Management and Minimization Plan

1. FIELD OPERATIONS

1.1 Geologic Standards SOP (AI-W-FO-01)

The lithologic descriptions for consolidated materials (igneous, metamorphic, and sedimentary rocks) shall follow the standard professional nomenclature (Tennissen, 1983), with special attention given to describing fractures, vugs, solution cavities and their fillings or coatings, and any other characteristics affecting permeability. Colors shall be designated by the Munsell Color System.

The lithologic descriptions for unconsolidated materials (soils [engineering usage] or deposits) shall use the name of the predominant particle size (e.g., silt, fine sand, etc.). The dimensions of the predominant and secondary sizes shall be recorded using the metric system. The grain size and name of the deposit shall be accompanied by the predominant mineral content, accessory minerals, color, particle angularity, and any other characteristics. The clastic deposit descriptions shall include, as a supplement, symbols of the Unified Soil Classification System (USCS). The color descriptions shall be designated by the Munsell Color System.

The scales for maps, cross sections, or 3-D diagrams shall be selected in accordance with the geologic and hydrologic complexity of the area and the purposes of the illustrations. Geophysical logs shall be run at a constant vertical scale of 1 inch equals 20 feet. When geophysical logs are superimposed on geologic logs, cross sections, or 3-D diagrams, the scales shall be the same. If defining geological conditions requires other scales, additional logs at those scales shall be provided.

For orientation, the cross sections shall show the Northern end on the viewer's right. If the line of cross section is predominantly East-West, the Eastern end is on the right. Maps shall be oriented with North toward the top unless the shape of the area dictates otherwise. Indicate orientation with a North arrow.

The USCS is a soil classification system used engineering and geology to describe the texture and grain size of a soil. The classification system can be applied to most unconsolidated materials, and is represented by a two-letter symbol. Each letter is described below (with the exception of Pt [peat]).

Table 1. Classification System Letters

Letter	Definition
G	gravel
S	sand
M	silt
O	organic

If the soil has 5–12% by weight of fines passing a #200 sieve (5% < P#200 < 12%), both grain size distribution and plasticity have a significant effect on the engineering properties of the soil, and dual notation may be used for the group symbol. For example, GW-GM corresponds to "well-graded gravel with silt."

If the soil has more than 15% by weight retained on a #4 sieve (R#4 > 15%), there is a significant amount of gravel, and the suffix "with gravel" may be added to the group name, but the group symbol does not change. For example, SP-SM could refer to "poorly graded SAND with silt" or "poorly graded SAND with silt and gravel."

Table 2. Symbol Chart

Major Divisions			Group Symbol	Group Name
Course grained soils more than 50% retained on or above No. 200 (0.075 mm) sieve	gravel > 50% of coarse fraction retained on No. 4 (4.75 mm) sieve	clean gravel < 5% smaller than No. 200 sieve	GW	Well-graded gravel, fine to coarse gravel
			GP	Poorly graded gravel
		gravel with > 12% fines	GM	Silty gravel
			GC	Clayey gravel
	gravel > 50% of coarse fraction passes No. 4 (4.75 mm) sieve	clean sand	SW	Well-graded sand, fine to coarse sand
			SP	Poorly graded sand
		sand with > 12% fines	SM	Silty sand
			SC	Clayey sand
Fine grained soils 50% or more passing the No. 200 sieve	silt and clay liquid limit < 50	inorganic	ML	Silt
			CL	Clay of low plasticity, lean clay
		organic	OL	Organic silt, organic clay
	silt and clay liquid limit > 50	inorganic	MH	Silt of high plasticity, elastic silt
			CH	Clay of high plasticity, fat clay
		organic	OH	Organic clay, organic silt
Highly organic soils			Pt	Peat

1.2 Site Reconnaissance, Preparation, and Restoration SOP (AI-W-FO-02)

The Contractor shall conduct a site reconnaissance of the field sites prior to the start of the field investigations. The primary objectives of conducting a site reconnaissance are to recommend possible changes in the technical approach, and to allow for adequate review of any such changes. During the site reconnaissance, the Contractor shall perform the following tasks:

- Review pertinent documents;
- Interview personnel who may be knowledgeable about environmental conditions in the project area;
- Conduct multiple site walks to evaluate monitoring and sampling locations;
- Verify and mark proposed sampling locations;
- Evaluate site accessibility and security;

- Designate field office sites;
- Identify potentially contaminated areas (i.e., discolored soils, stressed vegetation), particularly those that may require emergency response; and
- Document and evaluate site reconnaissance observations and update site maps.

A geophysical survey shall be conducted for the presence of underground utilities using electromagnetic methods in areas designated for intrusive sampling. Utility locations are determined using existing utility maps, and in the field, are verified using a hand-held magnetometer or utility probe. Vehicle access routes to sampling locations shall be determined prior to any field activity. Prior to any intrusive fieldwork, the Contractor Project Manager (PM) shall coordinate with the base Facilities Remedial Project Manager (RPM) to insure that the most current base procedures for obtaining dig permits and utility clearances are followed. In addition, any fieldwork activities along active flight line corridors or other controlled access areas will require close coordination with the RPM to obtain access approval before fieldwork is initiated.

A centralized decontamination area shall be provided for drilling rigs and equipment. The decontamination area shall be large enough to allow storage of cleaned equipment and materials prior to use, as well as to stage drums of decontamination waste. Smaller decontamination areas for personnel and portable equipment shall be provided, as necessary. A designated location shall be provided for the field office if necessary.

Each work site or sampling location shall be returned to its original condition when possible. Efforts shall be made to minimize impacts to work sites and sampling locations, particularly those in or near sensitive environments such as wetlands. Following the completion of work at a site, all drums, trash, and other waste shall be removed and disposed of properly. Decontamination and/or purge water and soil cuttings shall be transported to the designated locations, as described in Section 1.17 and the site-specific Work Plan.

Site restoration shall be coordinated with the Facilities RPM and the field team leader (FTL) to ensure that site restoration is conducted according to facility requirements. A site restoration contractor shall be retained by the Contractor where necessary to restore areas following heavy equipment usage or drilling activity.

1.3 Utilities Location SOP (AI-W-FO-03)

Prior to any intrusive work, the Contractor shall work with the Facilities RPM to follow project – specific requirements and current Facility requirements for Dig Permits. Careful planning plays an important role and is the first step in utility damage prevention efforts. HASPs for projects that include subsurface drilling or excavation shall include a Site-Specific Hazard Analysis for, but not limited to, safety and provisions for utility line damage prevention. The Drilling Subcontractor and the Contractor shall determine the actual location of all known utilities through careful review of the civil/utility drawings before drilling in areas where the safety of existing utilities may be compromised, and shall evaluate aboveground features such as, but not limited to,

manhole investigation (lifting manhole covers), valve boxes and pipe and cable risers which indicate the location of underground utilities prior to excavation/drilling. In general, a qualified utilities location subcontractor shall be contracted to conduct this work.

It is mandatory that the pertinent utility Location Service Center be notified and that each location of intrusive work is cleared by the proper authority prior to conducting subsurface work. The service center must be called at least three days before excavation and clearance is good for 15 days following clearance. All utilities in a work area shall be marked prior to subsurface work, using of stakes, flags, paint or other clearly identifiable materials to show the field location of underground utilities in accordance with current color code standards such as the American Public Works Association. This process is initiated with a call to the Local Utility Locate Service Center, requesting locates be made and providing the exact location of the drill site.

Following the location and marking of known or suspected utilities and excavation locations are located away from known underground utilities; initial digging shall be conducted using a shovel or backhoe at a careful rate with observers evaluating the first 4 feet of excavation depth closely for signs of man-made material such as conduit, metal, or concrete. This is done to uncover any potential utilities not identified by other means, especially where drilling or excavating is required within 3 feet of known or suspected utilities, or if exact locations are not known.

If there is any indication that the drill may have encountered a man-made obstruction within the first 10 feet below ground surface (bgs), operations shall stop immediately, and the excavation shall be investigated prior to proceeding with subsurface work to determine whether that an unknown or improperly located utility may have been encountered.

If an existing utility is damaged, the Contractor Project Manager shall be notified immediately. The onsite Field Manager shall document the damage and pertinent circumstances surrounding the incident as soon as possible (in writing and with photographs or video), and a detailed incident/accident report shall be prepared.

1.4 Geophysical Survey SOP (AI -W- FO-04)

1.4.1 General Requirements for Geophysical Surveys

General requirements for all geophysical surveys are: (1) the prime contractor shall have a state licensed geologist or engineer to supervise geophysical survey work, (2) the locations of boreholes logged with geophysical instruments shall be shown on a site map, (3) the locations of surface geophysical grid system layouts shall be shown on a site map, (4) the location of areas analyzed with subsurface geophysical techniques shall be shown on a site map (5) final results shall be presented in plain views and cross sections. Contours shall be used where appropriate, (6) the interpretation of results shall discuss positive and negative results as well as limitations of the method and data and, (7) the interpretation of the data shall be incorporated into the conceptual site model.

1.4.2 Surface Geophysical Surveys

Surface geophysical techniques include, but are not limited to, ground penetrating radar (GPR), magnetometry, and electromagnetic techniques. Use of any of these techniques is dictated by the project data quality objectives, and the objectives of these techniques is to locate the boundaries of suspected or known underground metallic objects or volumes of disturbed soil. The areas to be surveyed are described and shown on site maps presented in the site specific UFP QAPP.

Surface geophysical surveys are conducted within predetermined grids defined by transect lines crossing each site or area of interest. The spacing of the grids is determined from the approximate dimensions of the features to be located. Qualified individuals shall conduct the surveys and shall be supervised by a state licensed geologist or state licensed engineer. Location and elevation information sufficient to map and assess the survey results shall be recorded. Depending on the level of accuracy and detail required, northing and easting from a surveyed reference point, measurements in a third order survey, depth bgs, and/or professionally surveyed points and transects may be included. Location data, instrument numbers, calibration information, geophysical interpretation, and maps for all geophysical surveys shall be stored in project files.

General requirements for surface geophysical surveys are: (1) the prime contractor shall correlate surface survey data (profiles and soundings) with at least one soil boring, well bore, or outcrop at the same site as the survey and, (2) the location and elevation of at least two points of the geophysical survey grid shall be surveyed according to the specifications of the site-specific Work Plan.

1.5 Lithologic Logging SOP (AI-W-FO-07)

The lithology in all boreholes shall be logged. The Drill Log form, (Appendix A), shall be used for recording the lithologic logging information. Information on the boring log sheet includes the borehole location; drilling information; sampling information such as sample intervals, recovery, and blow counts; and sample description information.

Unconsolidated samples for lithologic description shall be obtained at each change in lithology or every five (5) foot interval, whichever is less or as specifically stated in the SOW. Lithologic descriptions of unconsolidated materials encountered in the boreholes shall generally be described in accordance with American Society for Testing and Materials (ASTM) D-2488-90 Standard Practice for Description and Identification of Soils (Visual-Manual Procedure) (ASTM, 1990). Descriptive information to be recorded in the field shall include: (1) identification of the predominant particles size and range of particle sizes, (2) percent of gravel, sand, fines, or all three, (3) description of grading and sorting of coarse particles, (4) particle angularity and shape, and (5) maximum particle size or dimension.

Plasticity of fines description include: (1) color using Munsell Color System, (2) moisture (dry, wet, or moist), (3) consistency of fine grained soils, (4) structure of consolidated materials, and (4) cementation (weak, moderate, or strong).

Identification of the Unified Soil Classification System (USCS) group symbol shall be used. Additional information to be recorded includes the depth to the water table, caving or sloughing of the borehole, changes in drilling rate, depths of laboratory samples, presence of organic materials, presence of fractures or voids in consolidated materials, and other noteworthy observations or conditions, such as the locations of geologic boundaries.

Lithologic descriptions of consolidated materials encountered in the boreholes shall generally be described in accordance with Section 1.1. Consolidated samples for lithologic description shall be obtained at each change in lithology or at five-foot intervals, whichever is less, or as specified in the SOW. All samples shall be monitored with an organic vapor monitor (e.g., PID, organic vapor analyzer [OVA]). The samples shall be handled in such a way as to minimize the loss of volatiles, as described in Section 2.4. Cuttings shall be examined for their hazardous characteristics. Materials suspected to be hazardous because of abnormal color, odor, or organic vapor monitor readings shall be containerized in conformance with the Resource Conservation and Recovery Act (RCRA) and the state and local requirements. Rock cores shall be stored in standard core boxes, and missing sections of core shall be replaced with spacers.

Lithologic descriptions of consolidated materials shall follow the specifications in Section 1.1.

1.6 Borehole Abandonment SOP (AI-W-FO-08)

Boreholes that are not converted to monitor wells shall be abandoned with neat cement grout as regulated by the appropriate state regulatory department. The following bullets provide the state requirements.

EXAMPLE

1. All analytical data obtained from each environmental soil sampling boring shall be submitted to NJ DEP within 30 days of receipt of laboratory results unless another schedule has been approved by SCDHEC.
2. The boring shall be abandoned within five days of borehole completion.
3. Borings 25 feet in depth or shallower may be abandoned by backfilling with native fill material. Borings greater than 25 feet in depth shall be completely filled from the bottom of the borehole to the land surface with bentonite-cement, neat cement, or 20% high solids sodium bentonite grout.
    a. The boring shall be abandoned by forced injection of grout or pouring through a tremie pipe starting at the bottom of the borehole and proceeding to the surface in one continuous operation.

All abandoned boreholes shall be checked 24 to 48 hours after mud/solid bentonite emplacement to determine whether curing is occurring properly. More specific curing specifications may be recommended by the manufacturer and shall be followed. If settling has occurred, a sufficient amount of mud/solid bentonite shall be added to fill the hole to the ground surface. These curing checks and any addition of mud/solid bentonite shall be recorded in the field log book.

1.7 Hollow-Stem Auger Drilling and Sampling SOP (AI-W-FO-09)

Hollow-stem auger drilling techniques use large diameter (up to 14-inch outside diameter [OD]) continuous-flight augers, which mechanically excavate drilled materials from the hole. These augers are built with a large (up to 10.25-inch inside diameter [ID]) axial opening to allow access to the bottom of the hole without withdrawing the auger string. The augers act as temporary casing during and at the completion of drilling to facilitate the sampling of sediment and water and the installation of monitoring wells. Some advantages of hollow-stem auger drilling often make it the preferred method of installing monitoring wells. Hollow-stem auger drilling is relatively rapid, especially in shallow applications in poorly lithified to unlithified sediments. Little or no outside fluid is required in the drilling process. Though a relatively large volume of cuttings are generated, they are normally easily contained. The volume of effluent, resulting from well-development efforts and requiring disposal, is normally lower than with some methods, notably mud rotary. Hollow-stem auger drilling readily supports thin-wall and split-tube sampling in poorly lithified sediments. Most hollow-stem rigs operating in the state also support other drilling methods such as mud rotary, solid-stem auger, and coring. Hollow-stem rigs are relatively simple, with few lubricated parts at positions likely to contaminate the test hole or monitoring well. Rigs capable of supporting hollow-stem drilling are available Statewide.

There are some disadvantages and limitations to the use of the hollow-stem auger method in the construction of monitoring wells. It is limited to drilling in poorly lithified to unlithified sediments and to a maximum depth of about 150 feet. Shallow bedrock or other hard-to-drill materials may reduce this depth significantly. Hollow-stem augers are prone to cross contamination of fluids within the bore hole along the large annular space around the auger tubing. High hydrostatic pressures in the bore hole can cause problems with sand heaving up into the augers during sampling and well-installation procedures. Wide variations in bore-hole size, common to auger drilling in poorly cohesive sediments, may complicate effective sealing of the annular space during monitoring-well installation. The design of hollow-stem augers produces an approximately 1-inch thick rind of smeared cuttings which may effectively seal the bore-hole walls in clayey sediments. This rind may interfere with the flow of fluids to the monitoring well.

Drilling and soil sampling can be accomplished with a variety of hollow-stem auger systems. Types of systems can be chosen depending on the advantages of handling, sampling requirements, and subsurface conditions. There are two basic types of systems. One type of system uses inner drill rods or hex rods connecting the sampler or pilot bit assembly to the surface for advancing and retrieving the sampler barrel or pilot bit assembly. Another system

uses a wireline latching system in the HSA column to lower, latch, and retrieves a core barrel or pilot bit assembly.

Double tube hollow-stem auger sampling systems can be particularly advantageous for sampling water-sensitive soils, such as collapsible soils since fluid is not used in the drilling process. Since no pressurized circulation medium is used during the drilling process, the possibility for hydraulic fracturing of formation materials and core contamination from drill fluids is reduced.

Difficulties in drilling may occur if cohesionless soils are drilled below the water table. Possibilities for sand lock or wedging of cuttings may occur (2). In cases where sands enter the HSA, water or drilling fluid may be added to the HSA column to provide hydrostatic balance or special pilot bit assemblies can be used (see Section 1.6). Problems may occur in getting the soil core barrel or pilot bit assembly back to the bottom of the HSA column. Highly saturated sands or liquefiable material may be drawn into the HSA by vacuum created when the sampler barrel or pilot bit assembly is initially pulled back through the cutter head of the lead auger assembly from the bottom of the borehole.

Consideration should be given to proper decontamination and cleaning of drilling equipment, hollow-stem augers, samplers, and soil coring components. Decontamination procedures are described in the site-specific Work Plan and Section 1.16 of this document.

In most geotechnical explorations, hollow-stem auger drilling is combined with other sampling methods. Split barrel penetration tests (Test Method D 1586) are often performed to provide estimates of engineering properties of soils. Thin-wall tube (Practice D 1587) and ring-lined barrel samples (Practice D 3550) are also frequently taken. This document discusses bore-hole preparation for these sampling events. For information on the sampling process, consult the related standards. Other in situ tests, such as the vane shear Test Method D 2573, can be performed below the base of the boring by access through the drill string.

1.8 Direct Push Drilling and Sampling SOP (AI-W-FO-10)

A direct push sampling device is used to collect soil, soil-gas and groundwater samples at specific depths bgs. Direct push systems are hydraulically powered and mounted in a customized four-wheel or track driven vehicle. The base of the sampling device is positioned on the ground over the sampling location and the vehicle is hydraulically raised on the base. As the weight of the vehicle is transferred to the probe, the probe is pushed into the ground. A built-in hammer mechanism allows the probe to be driven through dense materials. Maximum depth penetration under favorable circumstances is about 50 feet.

Soil samples are collected with a specially-designed sample tube. The sample tube is pushed and/or vibrated to a specified depth (approximately one foot above the intended sample interval). The interior plug of the sample tube is removed by inserting small-diameter threaded

rods. The sample tube is then driven an additional foot to collect the samples. The probe sections and sample tube are then withdrawn and the sample is extruded from the tube into sample jars.

Slotted lengths of probe can be used to collect groundwater samples if the probe rods can be driven to the water table. Groundwater samples are collected using either a peristaltic pump or a small bailer.

1.8.1 Preparation

Determine extent of the sampling effort, sample matrices to be collected, and types and amounts of equipment and supplies required to complete the sampling effort. Obtain and organize necessary sampling and monitoring equipment. Decontaminate or pre-clean equipment, and ensure that it is in working order. Perform a general site survey prior to site entry in accordance with the site-specific HASP. Use stakes or flagging to identify and mark all sampling locations. All sample locations should be cleared for utilities prior to commencing intrusive activities.

1.8.2 Setup of Direct Push Rig

Back carrier vehicle to probing location, set vehicle to park, shut off ignition and set parking brake and block rear tires. Attach exhaust hoses so exhaust blows downwind of the sampling location (this is particularly important during soil gas sampling). Start engine using the remote ignition at the Geoprobe operator position. When positioning the probe, always use the SLOW speed. Check for clearance on vehicle roof before folding hydraulics out of the carrier vehicle. Extend the hydraulics and lower the derrick to the ground surface. When the probe axis is vertical and the weight of the vehicle is on the probe unit, probing is ready to begin. If ground-cover is concrete or asphalt, Carbide drill tip shall be inserted into hammer and ground-cover shall be cored via rotation.

1.8.3 Sample Collection

Soil samples can be collected continuously via 4 ft long acetate sleeves, or at specific intervals by driving the sampler to the correct depth with a drive point in place, then removing the drive point through the outer barrel, and inserting the soil core sampler and driving to the required depth. The soil core sampler is then removed and split length-wise for lithologic description, organic vapor screening, head space sampling, or sampling for laboratory analysis.

Groundwater samples can be collected at depth by driving a slotted rod with a drive point threaded to the end to below the potentiometric water table. A water level measuring tape should be used to ensure water is encountered and at static water level. Some time may be required for the water table to reach static levels, depending on the permeability of the aquifer material. Static depth to water should be measured and recorded prior and following groundwater collection. Groundwater samples shall be collected either with a mini-bailer or

pumping device. Volatile organic compounds (VOCs) shall be sampled first with a bailer. Non–VOCs can be sampled with a pump.

1.9 Monitor Well Construction SOP (AI-W-FO-11)

Monitor well construction is not part of this project.

1.10 Monitor Well Development SOP (AI-W-FO-12)

Monitor well development is not part of this project.

1.11 Monitor Well Abandonment SOP (AI-W-FO-13)

Monitor well abandonment is not part of this project.

1.12 Water Level Measurement SOP (AI-W-FO-14)

Water level measurements shall be performed to estimate principal groundwater flow direction(s) and hydraulic gradient. Water levels shall be measured from the notch located at the top of the well casing. If well casings are not notched, measurements shall be taken from the north edge of the top of the well casing, and a notch shall be made using a decontaminated metal file. The following procedures shall be used to measure water levels.

- A PID shall be calibrated to ambient air conditions. After uncapping the well, background and casing headspace organic vapor measurements shall be recorded;
- An interface probe shall be lowered into the groundwater monitoring well if a nonconductive floating product layer is suspected. The interface probe shall be lowered prior to measuring groundwater levels in the well;
- Date, well number, field instrument identification number, casing diameter, and other pertinent observations (e.g., availability of sounder port, well condition) shall be recorded;
- Depth to groundwater from the top of casing shall be measured to the nearest 0.01 foot and recorded on a Static Groundwater Measurement Form (Appendix A). Water level results shall be compared with previous measurements to check for discrepancies;
- The water level sounder tape and probe shall be thoroughly rinsed before being lowered into each groundwater monitoring well installation; and
- An inspection of the surrounding area shall be made to assure that all equipment and materials have been retrieved and that the appropriate well cap has been replaced and secured.

Following water level measurement, the total depth of the well from the top of the casing shall be determined using a weighted tape or electronic sounder and shall be recorded on the Static

Groundwater Measurement Form (Appendix A). The water level depth shall then be subtracted from the total depth of the monitoring well to determine the height of the water column present in the well casing. All water level and total depth measuring devices shall be routinely checked non

1.13 Test Pit Excavation SOP (AI-W-FO-15)

Test pit excavation is not part of this project.

1.14 Equipment Decontamination SOP (AI-W-FO-16)

All field equipment that may directly or indirectly contact samples shall be decontaminated in a designated decontamination station. A decontamination station shall be established at each field site prior to initiating intrusive field activities. It shall consist of a plastic-lined pad where drilling equipment, such as auger flights, drill rods, and sampling devices, can be steam cleaned. In addition, the portion of the drill rig which stands above the boreholes shall be decontaminated by steam cleaning at the decontamination station. Drilling rigs and associated drilling equipment shall be steam-cleaned between borings to minimize the potential for cross-contamination.

1.14.1 Heavy Equipment Decontamination

General decontamination procedures for large pieces of equipment include the following:

- Drill rigs shall be decontaminated upon entering the site, before leaving the site, or when being moved from one boring location to another;
- HSA flights, drill bits, and rods shall be decontaminated with high-pressure hot water and laboratory grade phosphate-free detergent, scrubbed if necessary, rinsed thoroughly with potable water, and allowed to air dry; and
- All casings, screws, and other downhole equipment shall be steam-cleaned prior to installation.

1.14.2 Instrument and Reusable Sampling Equipment Decontamination

Instruments that contact water, such as an electronic water level indicator, shall be decontaminated following the procedures for sampling equipment described below. Instruments that are sensitive to soap and solvents, such as the pH meter, shall be rinsed with potable and deionized water. The probes shall be cleaned daily and stored overnight according to the manufacturer's recommended procedures.

All reusable field equipment used to collect, handle, or measure samples shall be decontaminated before coming into contact with any soil or groundwater samples. The decontamination procedure must match the degree of contamination of the sampling tool. For example, steam cleaning shall be necessary to remove dirt from auger flights and to prepare well

screens and riser pipe for installation into the borehole. Brushes and soap may be required to remove dirt from split-spoon samplers. Sampling equipment that comes into contact with oil and grease shall be cleaned with methanol and hexane to remove any oily material. Clean, disposable gloves shall be worn during and after decontamination so that equipment shall not be re-contaminated.

General decontamination procedures for sampling and drilling devices such as split-spoon samplers, bailers, and other equipment that can be hand manipulated are as follows.

- Steam clean if practical;
- Scrub equipment with a solution of potable water and a laboratory grade phosphate-free detergent (e.g., Alconox) to remove all dirt from sampling or drilling item;
- Rinse sampling item thoroughly with potable water to remove residual dirt and rewash, if necessary;
- If glass sampling equipment is used and metals are part of the analysis program, rinse the interior/exterior surfaces of the glass equipment that will come in contact with the sample matrix with 10 percent nitric acid (HNO3) solution.;
- Rinse item with analyte-free water, and
- Rinse a final time with analyte free water. This last rinse water can be captured and containerized, labeled wrapped and cooled to 4 C in o cooler as the Equipment Blank for the day.

Each lot of reagent-free water obtained shall be tested by collecting and submitting one sample to the laboratory for analysis of target analyte types.

1.15 WASTE HANDLING SOP (AI – W FO – 17)

The Contractor shall prepare an updated Waste Management and Minimization Plan (WMMP) for the required activity. The WMMP shall be included as part of the site-specific Work Plan and details waste handling procedures that shall be implemented during the field investigation. A Waste Inventory Tracking Log (Appendix A) shall be kept during sampling events. Non-investigative waste, such as litter and household garbage, shall be collected on an as-needed basis to maintain each site in a clean and orderly manner. This waste shall be containerized and transported to the designated sanitary landfill or collection bin. Acceptable containers shall be sealed boxes or plastic garbage bags.

Investigative-derived waste (IDW) shall be properly containerized and temporarily stored at each site, prior to transportation for off-site disposal. Depending on the constituents of concern, fencing or other special marking may be required. The number of containers shall be estimated on an as-needed basis. Acceptable containers shall be sealed, U.S. Department of Transportation (DOT)-approved steel 55-gallon drums or small dumping bins with lids. Other containers and procedures may be presented in site-specific WMMP. Containers shall be transported in such a manner to prevent spillage or particulate loss to the atmosphere.

The investigative derived waste shall be segregated at the site according to matrix (solid or liquid) and as to how it was derived (drill cuttings, drilling fluid, decontamination fluids, and purged groundwater). Each container shall be properly labeled. Label information shall be placed directly on the drum with indelible pain pen and on a plastic-coated label with indelible marker (Sharpie tm), which shall be placed in a zip lock bag and taped to the drum with duct tape. All drums shall be labeled with the following information:

1. Drum number
2. Contract and TO number;
3. Contents (development water and well identifiers);
4. Dates of development activities; and
5. Facilities RPM name and phone number.

Information for each drum shall be recorded on a Waste Inventory Tracking Log (Appendix A).

1.15.1 IDW Sampling

Soil cuttings, groundwater, and rinse water generated during soil and groundwater sampling will be sampled and analyzed in accordance with the program stated in the task-specific work plans. For soils placed in drums, each drum will be opened and scanned using a PID. Samples from a specific location will be taken from the drum that produces the highest PID readings. Samples collected for VOC analysis will be collected by Encore, Method 5035, otherwise samples will be grab samples and handled as any other soil. Additional samples may be required if the results from these samples do not meet the disposal requirements for soils. Additional grab samples would be collected for toxicity characteristic leaching procedure (TCLP) analyses.

For soil placed in roll-off containers during excavation work, the soils will be “sectioned” and scanned using a PID. The sections of the soil within the roll-off found to have the highest reading on the PID will be collected via Encore for VOCs or mixed as described in the surface soil and subsurface soil sampling sections (2.6 and 2.7) of this SOP. The analyses to be performed will be determined in the site-specific Work Plan. Results for these IDW samples will then be evaluated against the disposal criteria following the protocols outlined in the site-specific Work Plan.

Typically, groundwater results for the individual locations should generate data that can be used for the disposal of water samples. However, should additional sampling be required, grab samples will be collected using a bailer for drums or directly from the valve of a baker tank. These water samples will then be treated as any other water sample being sent to the laboratory.

1.16 Surveying SOP (AI-W-FO-18)

Surveying shall be conducted at two levels of precision. General site features, such as property boundaries, fence lines, utilities locations, and (petroleum oil and lubricant (POL) pipeline locations shall be surveyed at sub-meter accuracy and precision in Northing, Easting and Elevation. These measurements shall be collected using Global Positioning System (GPS) surveying equipment operated by the Contractor staff. Soil gas survey locations and soil boring locations shall be surveyed using GPS, as well. Features that require precise elevation controls, such as monitoring wells, stream or ditch bottoms, or reference points, shall be surveyed by a licensed surveyor in the state. Monitoring wells and reference points for stream gauging shall be measured at ground level and at the reference point. Monitoring well reference points shall be at the notch located at this northern fencing point on the PVC casing stickup. Stream gauges shall include a semi-permanent measuring point, depth with top above ground surface. The horizon and the vertical precision for these survey data shall be ±1 foot and ±0.01 foot, respectively.

Site features and sampling locations shall be surveyed and the results shall be presented in a table containing the name of the feature/Locid, date, reference datum, survey method, Northing, Easting, Elevation, coordinate system, and measurement units. The final survey data shall be submitted in Universal Transverse Mercator (UTM) Projection Zone 17, WGS 84 Meters, NAD 83.

All surveying locations measured by a certified land surveyor in the previously described coordinate system shall be third order (Urquhart, 1962). An XY-coordinate system shall be used to identify locations.

1.17 AQUIFER TESTING FOR HYDRAULIC PARAMETERS SOP (AI – W FO – 19)

Aquifer testing is not part of this project.

2. ENVIRONMENTAL SAMPLING

In general, all sampling activities should include a pre-mobilization phase to ensure sampling equipment and supplies are available onsite during the sampling activity; the subcontracted laboratory project manager is prepared for samples to arrive and be analyzed within site-specific holding times, and data required for specialized data deliverables are available to be input into the Laboratory Information Management System (LIMS). The contract laboratory will provide certified-clean sampling containers properly preserved, shipping containers, packing supplies, de-ionized water for field blanks, chain of custody forms and custody seals. All sampling activities shall include the Site-specific and Base-wide Work Plans and the site-specific HASP as reference documents.

Prior to sample collection, Sample Field Forms should be completed to the extent possible, and finalized at each sampling location to track associated quality assurance/quality control (QA/QC) samples, sample times and dates, sample analytical method requirements, and samplers. Each sampling location shall be screened for potential exposure based on the expected target compounds, with general use of the project-specific organic vapor analyzer readings at all

sampling locations to be recorded on sampling forms and/or in the field log book. Each sample activity shall require a field log book to be completed, a camera and pictures to be taken to document the activities (noted in the log book), decontamination supplies, containers for IDW, sufficient supplies of new clean, non-powdered disposable nitrile gloves or similar, and other personal protective equipment required in the site-specific HASP. To the extent possible, sampling activities should be conducted with disposable or dedicated sampling equipment to limit the potential for cross-contamination and the need for rinsate blanks.

In addition, material of the sampling devices (e.g., plastic, PVC, metal) discussed below shall be appropriate for the contaminant of concern and shall not interfere with the chemical analyses being performed. These will be described in the site-specific Work Plan addenda. All purging and sampling equipment that is not certified clean and disposable shall be decontaminated according to the specifications in Section 1.16 prior to any sampling activities and shall be protected from contamination until ready for use.

2.1 General Water Sampling SOP (AI-W-ES-01)

VOCs and Metals: In most cases, samples collected for organic compounds and metals must be collected prior to other samples, with VOC samples being collected first. The VOC samples must be collected so that no air bubbles remain in the sample container. These samples must be collected by slowly pouring the sample contents into the vial until a convex meniscus is seen on the surface of the vial. A Teflon lined septum cap must carefully be placed on the vial until finger tight. The sample bottle should then be inverted to verify that no air bubbles have been trapped inside.

Filtering: As a general rule, groundwater samples should not be filtered. However, filtration may be needed to correct for chronically turbid wells. Filtered samples must not be collected from usable water supply wells. Filtering is also not recommended when the sample turbidity appears to be chemically-induced or colloidal. When samples are filtered, such as under conditions of excessive turbidity, both filtered and unfiltered samples must be submitted for analyses. Samples for organic compounds analysis must not be filtered. It is recommended that efforts be undertaken to minimize any persistent sample turbidity problems. These efforts may consist of the following:

1. Implementation of low flow/low stress purging and sampling techniques, or
2. Redevelopment of permanent ground water monitoring wells.

Containers, Preservation Methods, Volumes, and Holding Times: Required sample containers, preservation methods, volumes, and holding times are given in Work Sheet 19 of the Base-wide Work Plan or site-specific Work Plan addenda. Sampling equipment shall be decontaminated in accordance with Section 1.16 upon completion of sampling activities.

2.2 Monitor Well Sampling SOP (AI-W-ES-02)

Monitoring wells sampling is not part of this project.

2.3 Surface Water Sampling SOP (AI-W-ES-03)

Surface water samples may be collected from standing water in the ditches, creeks, ponds, and seeps located at the Facility. The equipment required for surface water sampling may include:

- Nylon rope;
- pH, temperature, turbidity, and specific conductance meters;
- Appropriate sample bottles and ice chest with ice;
- Plastic sheeting and
- 5-gallon buckets with lids.

Special attention should be paid to sampling locations, with locations surveyed using a site map and GPS device. If no site map is available, a hand drawn map should be developed with the location of site features with respect to the surface water sampling locations presented to scale. Locations should be staked and photographed for photo documentation and the time and date of the picture and location ID should be documented in the field notebook. The Surface Water Field Sampling Report form should be completed as required (Appendix A). In addition, the following information should be documented at each sampling location:

- Location of the sample in the body of water sampled.

The presence or absence of possible interfering surface water features such as:

- Tributaries
- Outfalls Disturbances (eddies, rapids, falls)
- Subsurface stratification (in still water or slow flow bodies)
- Sample collection method;
- Physical description of the collected sample;
- Water depth; and
- Measured field parameters.

To collect a representative sample of a stream or canal, the technician must also take into account the approximate depth and width of the body at the anticipated location. EPA provides guidance on the selection of sampling points and compositing techniques in the EPA SOP/QAM (EPA, 1996) document. For most locations where the depth and width of the stream is not great, a single sample collected in midstream at mid-depth is usually sufficiently representative. A subsurface water grab-sampling device (such as the peristaltic pump) allows the technician to submerge a sample collection vessel beneath the surface and then open to collect a single point, depth integrated sample. In cases where surface water and sediment samples are to be collected

in the same location, the surface water will be collected first, followed by the sediment sample to avoid undue disturbance of the sediment and possible contaminant release into the surrounding surface water.

Compositing samples in response to observed subsurface stratification poses an additional problem to the sampling technician. To maintain sample representativeness in a body of deep, still water or a low-flow water body where stratification is obvious, the technician should prepare a composite sample of the major layers using depth-integrated sampling devices, or submit a sample of the largest, most representative layer. Overall, adequate documentation of the sampling approach used by the technician in the field is the key to understanding how to use the associated analytical data.

General sampling requirements should be followed, including site-specific health and safety concerns, use of new nitrile sampling gloves with every sample collected, sample collection at the anticipated lowest concentration point first, grading to the highest anticipated concentration point last to minimize the effects of cross contamination. If possible, work should be segregated, so that one member of the team takes all notes, pictures, fills out labels, Chain of custodies, etc., while the other members collect the samples (SESDPROC -201-R1, 2007). If multiple samples are to be collected along a stream or water body, sampling should begin downstream and work upstream to reduce the potential for sampling cross contamination from disturbances due to the sample collection activity.

Surface Water Sampling Procedure

Surface water samples will be collected from drainages and the seeping adit using a peristaltic pump. The equipment required for surface water sampling may include:

- Field water quality meter(s) and flow measuring device;
- Peristaltic pump;
- Disposable 0.45 micron filters for collection of dissolved metals samples; and
- Appropriate sample bottles and ice chest with ice.

Surface water samples will be collected using laboratory provided, certified pre-cleaned containers. . All samples requiring filtering and preservation will be filtered and preserved as soon as practically possible, ideally immediately at the time of sample collection. Proper labeling, preservation, and shipping procedures will be followed as specified in the SOPs.

Special attention will be paid to sampling locations, with locations surveyed using a site map and GPS device. Locations will be staked and photographed for photo documentation, and the time and date of the picture and location ID will be documented in the field notebook. In addition, the following information will be documented at each sampling location:

- Location of the sample in the body of water sampled; and

- The presence or absence of possible interfering surface water features such as:
  - Tributaries;
  - Outfall disturbances (eddies, rapids, falls);
  - Subsurface stratification (in still water or slow flow bodies);
  - Sample collection method;
  - Physical description of the collected sample;
  - Water depth; and
  - Measured field parameters.

USEPA provides guidance on the selection of sampling points and compositing techniques in the USEPA SOP/QAM document (USEPA, 1996). Decontaminated between each sample location and an Equipment Blank must be analyzed for each constituent that may be cross contaminated at the end of each day.

1. Before laboratory samples are collected, an aliquot of the sample will be analyzed for geochemical characteristics using the field water quality instrumentation for temperature, pH, ORP, conductivity, turbidity, DO, and results will be recorded on the Field Sampling Report form provided in Appendix A.
2. During sample collection, if transferring the sample from a collection device, make sure that the device does not come in contact with the sample containers.
3. Place the sample into appropriate, labeled containers. Samples collected for VOC analysis must not have any headspace. All other sample containers must be filled with an allowance for headspace.
4. All samples requiring preservation must be preserved as soon as practically possible, ideally immediately at the time of sample collection.
5. Proper labeling, preservation, COC, and shipping procedures shall be followed as specified in Section 2.11 of this document.

2.4 Method 5035 Requirements for VOCS in Soil and Sediments SOP (AI-W-ES-04)

Soil and sediment VOC aliquots shall be collected in accordance with Method 5035. Commercially available kits should be used, (such as En Novative Technologies, Inc.) to account for both low (less than 200 µg/kg) and high level (greater than 200 µg/kg) media. Sample kits should contain the following.

- One disposable calibrated core sampler (5 g or 10 g);
- One 2-ounce container for percent moisture with lid;
- Two 40-ml VOA vials with stir bar, 5 mls of Sodium Bisulfate solution and tare weigh for low level analyses; and
- One 40-ml VOA vial with 5 mls of Methanol and tare weight for high level analyses.

The disposable calibrated core sampler shall be used to collect an undisturbed (if possible) sample aliquot for each of the three preserved VOA vials. Vials are immediately sealed with the caps and placed in a plastic bag. No labels or seals are placed on the individual containers as they

are pre-weighed and doing so will change the container weight. Labels and seals are instead placed on the plastic bag which holds the VOAs. In addition, the 2-ounce container is filled with the remaining sample to be used to determine the moisture content of the sample so that the result can be provided in dry-weight.

2.5 Soil Sampling SOP (AI-W-ES-05)

This discussion of soil sampling methodology reflects both the equipment used (required/needed) to collect the sample, as well as how the sample is handled and processed after retrieval. Selection of equipment is primarily based on the depth of sampling, but it is also controlled, to a certain extent, by the characteristics of the material. Simple, manual techniques and equipment, such as hand augers, are usually selected for surface or shallow, subsurface soil sampling. As the depth of the sampling interval increases, some type of powered sampling equipment is usually needed to overcome torque induced by soil resistance and depth. The following is an overview of the various sample collection methods employed over three general depth classifications: surface, shallow subsurface, and deep subsurface. Any of the deep collection methods described may be used to collect samples from the shallower intervals.

2.5.1 Manual Collection Techniques and Equipment

These methods are used primarily to collect surface and shallow subsurface soil samples. Surface soils are generally classified as soils between the ground surface and 12 inches below ground surface. The shallow subsurface interval may be considered to extend from approximately 12 inches below ground surface to 24 inches or to a site-specific depth at which sample collection using manual methods becomes impractical. The sample must be obtained from an area that is not in contact with metal sampler surface.

2.6 Surface Soil Sampling SOP (AI-W-ES-06)

Collection of samples from near-surface soil can be accomplished with decontaminated tools such as spades, shovels, trowels, and scoops. Surface material is removed to the required depth and a stainless steel or plastic scoop is then used to collect the sample. In general, certified clean disposable sampling tools shall be used to collect samples to reduce the potential for cross-contamination and requirement for an Equipment Blank.
A flat, pointed disposable trowel or similar shall be used to cut a block of the desired soil when undisturbed profiles are required. Tools plated with chrome or other materials shall not be used.

The following procedure is used to collect surface soil samples.

1. Carefully remove the top layer of soil or debris to the desired sample depth with a pre-cleaned spade.
2. Using a pre-cleaned, disposable scoop, spoon, or trowel, remove and discard a thin layer of soil from the area which came in contact with the spade.

3. If volatile organic analysis is to be performed, sample should be collected according to method 5035 (Section 2.4).
4. For non-VOC analyses and un-composited samples, place the remainder of the sample into a pre-cleaned disposable homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly.
5. For non-VOC analyses and composited samples, place a sample from another sampling interval or location into a pre-cleaned disposable homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.

Gravel, rock, and vegetation shall be excluded from surface soil samples. Samples shall be collected as quickly as possible to minimize loss of organics. This method generates a less disturbed soil sample, which is more suitable for volatile analysis. During field sampling activities, the Contractor shall record surface conditions that may affect chemical analyses on a Soil Field Sampling Report (Appendix A). These conditions may include:

- Approximate distance to roadways and access roads;
- Observations of seepage;
- Adjacent water level in ditch/stream;
- Distance of sample above surface water surface;
- Soil type (clay, silt, sand, gravel);
- PID reading;
- Site features, such as nearby drainage outfalls, activities occurring on plateau to west and east;
- Obvious deposition of contaminated or clean soil at the site;
- Evidence of dumping or spillage of contaminants; and
- Soil discoloration, unusual condition of growing plants, and stressed vegetation.

Surface soils may be collected with a wide variety of equipment. Spoons, shovels, hand-augers, push tubes, and post-hole diggers (made of the appropriate material) may be used to collect surface soil samples. As discussed in the section on powered equipment, surface soil samples may also be collected in conjunction with the use of heavy equipment.

2.7 Subsurface Soil Sampling SOP (AI-W-ES-07)

Hand-auger sampling is the most common manual method used to collect near-surface soil samples. Typically, auger-buckets with cutting heads are pushed and twisted into the ground and removed as the buckets are filled. The auger holes are advanced one bucket at a time.

The practical depth of investigation using a hand-auger is related to the material being sampled. In sands, hand-augering is usually easily accomplished, but the depth of investigation is controlled by the depth at which sands begin to cave. At this point, auger holes usually begin to collapse and cannot practically be advanced to lower depths, and further samples, if required, must be collected using some type of pushed or driven device.

Hand-augering may also become difficult in tight clays or cemented sands. At depths approaching 20 feet, torqueing of hand-auger extensions becomes so severe that in resistant materials, powered methods must be used if deeper samples are required. Some powered methods, discussed later, are not acceptable for actual sample collection, but are used solely to gain easier access to the required sample depth, where hand-augers or push tubes are generally used to collect the sample.

When a vertical sampling interval has been established, one auger-bucket is used to advance the auger hole to the first desired sampling depth. If the sample at this location is to be a vertical composite of all intervals, the same bucket may be used to advance the hole, as well as to collect subsequent samples in the same hole. However, if discrete grab samples are to be collected to characterize each depth, a new bucket must be placed on the end of the auger extension immediately prior to collecting the next sample. The top several inches of soil should be removed from the bucket to minimize the chances of cross-contamination of the sample from fall-in of material from the upper portions of the hole.

Another piece of soil sampling equipment commonly used to collect shallow subsurface soil samples is the Shelby or "push tube". This is a thin-walled tube, generally of stainless steel construction and having a beveled leading edge, which is twisted and pushed directly into the soil. This type of sampling device is particularly useful if an undisturbed sample is required. The sampling device is removed from the push-head, then the sample is extruded from the tube into the pan with a spoon or special extruder. Even though the push-head is equipped with a check valve to help retain samples, the Shelby tube will generally not retain loose and watery soils, particularly if collected at lower depths.

2.7.1 Powered Sampling Devices

Powered sampling devices and sampling aids may be used to acquire samples from any depth but are generally limited to depths of 20 feet or less. Among the common types of powered equipment used to collect or aid in the collection of subsurface soil samples are power augers; split-spoon samplers driven with a drill rig drive-weight assembly or hydraulically pushed using drill rig hydraulics; continuous split-spoon samplers; specialized hydraulic cone penetrometer rigs; and back-hoes. The use of each of these is described below.

Power Augers: Not used.

Drill Rigs: Drill rigs offer the capability of collecting soil samples from greater depths. For all practical purposes, the depth of investigation achievable by this method is controlled only by the depth of soil overlying bedrock, which may be in excess of 100 feet. When used in conjunction with drilling, split-spoon samplers are usually driven either inside a hollow-stem auger or inside an open borehole after rotary drilling equipment has been temporarily removed. The spoon is driven with a 140-pound hammer through a distance of up to 24 inches and removed. If geotechnical data are also required, the number of blows with the hammer for each six-inch interval should be recorded.

Back-Hoes: Not used.

2.7.1.1 Split-Spoon Samples

When subsurface soil samples are collected using hollow-stem auger drilling wire line methods, soil samples are to be submitted for laboratory analysis, they shall be collected using stainless steel, continuous drive, California modified split-spoon samplers, or equivalent. These samplers are 18 to 24 inches in length with a 3-inch OD to accommodate three to four 2-inch diameter brass/stainless steel rings, each of which is six inches in length.

Each time a split-spoon sample is taken, a standard penetration test shall be performed in accordance with ASTM D-1586 "Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils." (ASTM, 1999). The sample is obtained by driving the sampler a distance of 1 foot into undisturbed soil with a 140-pound hammer, free falling a distance of 30 inches. The sampler is first driven six inches to seat it in undisturbed soil, then the test is performed. The number of hammer blows for seating the spoon and making the test are then recorded for each six inches of penetration on the drill log (i.e., 5/7/8). The standard penetration test result (N) is obtained by adding the last two figures (i.e., 7+8=15 blows per foot). The sampler is then driven an additional six inches to fill the remainder of the split-spoon prior to retrieval.

As soon as the split-spoon is opened, the open ends of the brass/stainless steel rings shall be monitored for organic vapors using the PID or FID. Air monitor results shall be recorded on the Drill Log (Appendix A) and in the field log book.

Method 5035 shall be used to collect VOC samples directly from the sampling rings (Section 2.4). Additional sample material shall be collected for analysis of other required analytical types (PAHs, inorganics, pesticides, for example) from each sample interval where a VOC sample is taken.

2.7.2 Backhoe Soil Sampling

Not used.

2.8 Field Headspace Screening SOP (AI-W-ES-08)

If initial screening results indicate the presence of organic vapors, a headspace analysis shall be conducted on remaining portions of the sample.

Following collection of split-spoon soil samples, headspace screening shall be performed in the field using a portable PID on the remaining portions of samples selected on the basis of initial screening. Soil samples collected from the borings shall be field screened by filling a self-closing polyethylene plastic bag with approximately 250 grams of soil. The soil samples shall then be vigorously shaken for approximately 30 seconds and allowed to equilibrate a minimum of 15 minutes. The bag headspace shall then be screened for organic vapors by puncturing the bag exterior with the PID probe, inserting the tip to a distance approximately one-half the headspace depth, and recording the highest reading displayed on the instrument meter. The results of field headspace screening shall be recorded on the Drill Log (Appendix A) and used to select samples from each boring for laboratory analysis of selected analytical constituents. The field headspace sample shall be saved for future reference and stored on site.

All information regarding field headspace screening results, soil texture, density, consistency, and color shall be recorded on the Drill Log (Appendix A).

2.9 Geotechnical Sampling SOP (AI-W-ES-09)

During HSA drilling in the relatively soft, moist soil that lies above the saturated zone, geotechnical samples shall be collected, if necessary, in 1 foot long by 3 inches in diameter Shelby tube samplers. Once the FTL has determined that the sampling depth has been reached, the driller shall remove all equipment from the HSA internal casing. The Shelby tube sampler shall then be connected to the end of the drilling rod and lowered into the bottom of the boring, with successive lengths of rod. The full length of the sample shall be pressed into the undisturbed material at the base of the borehole using the hydraulics associated with the Kelly bar. The sampling rod shall then be removed from the borehole and the filled sampler shall be removed from the rod by the drill crew and handed to the sampling technician. The sampling technician shall take a small portion of the material and place it in an evacuated Ziploc TM bag for lithologic logging and headspace assessment. The sampling technician shall cut the Shelby tube sampler to remove unfilled portions of tube, then cover both ends of the sampler with a TeflonTM swatch and plastic cap, label the sampler with information pertaining to the location where the sample was collected, mark the distance bgs associated with each end on the pertinent plastic cap, and store it at the sample station until all geotechnical samples haven collected. The FTL shall then review sampling locations, lithology, and sample integrity and determine which samples shall be sent to the geotechnical laboratory for analysis. Samples shall be stored vertically in the same orientation withdrawn from the borehole. Each sample shall be packed as tightly as possible to minimize impact to the sample integrity. The samples shall be analyzed for geotechnical parameters in accordance with the site-specific Work Plan following the appropriate ASTM

methods. Geotechnical samples shall be sent to a geotechnical laboratory that has been approved by Facilities and SCDHEC.

2.10 Sediment Sampling SOP (AI-W-ES-10)

A potentially more reliable indicator of surface water contamination is the underlying stream sediments. Unlike surface water contamination, which may become diluted or chemically transformed downstream, contaminants have a tendency to accumulate in sediments. Stream sediment samples shall be obtained following collection of surface water samples. The order of stream sediment sampling shall begin with the farthest downgradient sample and move progressively upgradient to minimize potential cross-contamination between locations and media. Where appropriate, stream sediment samples shall be collected from the active streambed on the stream side nearest the contamination source. During surface water and stream sediment sampling, the Contractor field personnel shall sketch the approximate sampling locations and record the following field observations on the Surface Water/Sediment Field Sampling Report (Appendix A):

- Sediment type (e.g., sand, silt, gravel, clay, organic, other);
- The percent recovery if sediment cores are collected;
- Geomorphology (channel shape, stream bank description, erosional/depositional characteristics);
- Vegetation type;
- Color and/or discoloration;
- Sample depth;
- Odor; and
- The sampling device.

Stream sediment samples shall be collected using a disposable scoop, with VOCs collected directly from the scoop according to Section 2.4 for Method 5035. The remaining non-VOC aliquot will be transferred to a clean disposable homogenization container where excess water will be decanted and the remaining material will be thoroughly homogenized, then transferred to the proper container, sealed, labeled and placed in a cooler on ice to be chilled to 4̊C until shipment.

If low concentration samples effervesce from contact with the acid preservative, then either a test for effervescence must be performed prior to sampling, or the investigators must be prepared to collect each sample both preserved or un- preserved as needed, or all samples must be collected unpreserved. To check for effervescence, collect a test sample and add to a pre-preserved vial. If preservation (acidification) of the sample results in effervescence (rapid formation of bubbles) then preservation by acidification is not acceptable, and the sample must be collected un-preserved.

If effervescence occurs and only pre-preserved sample vials are available, the preservative solution may be placed into an appropriate hazardous waste container and the vials triple rinsed with deionized water. An appropriate amount of deionized water, equal to the amount of preservative solution, should be placed into the vial. The sample may then be collected as an un-preserved sample. Note that the amount of organic free water placed into the vials will have to be accurately measured and recorded to allow for the change in the tare weight of each vial (SESDPROC-200-R2, 2010).

2.11 Sample Handling SOP (AI-W-ES-11)

2.11.1 Sample Containers

Sample containers are purchased pre-cleaned and treated according to EPA specifications for the methods. Sampling containers that are reused are decontaminated between uses by the EPA-recommended procedures (i.e., EPA 540/R-93/051). Containers are stored in clean areas to prevent exposure to fuels, solvents, and other contaminants. Amber glass bottles are used routinely where glass containers are specified in the sampling protocol.

2.11.2 Sample Volumes, Container Types, and Preservation Requirements

Sample volumes, container types, and preservation requirements for the analytical methods performed on samples are listed in the Facility Work Plan, Worksheet # 19.

Sample holding time tracking begins with the collection of samples and continues until the analysis is complete. Holding times for methods required routinely for work are specified in Worksheet #19.

2.11.3 Sample Identification

A formal sample identification system shall be used to designate each field investigation. The identification system shall provide a tracking procedure to allow retrieval of information about a particular sample location and to ensure that each sample is assigned a unique identification that describes where it was collected.

This is described in detail in Worksheet 27, Section 27.1, Sample Identification.

2.11.4 Sample Custody

Procedures to ensure the custody and integrity of the samples begin at the time of sampling and continue through transport, sample receipt, preparation, analysis and storage, data generation and reporting, and sample disposal. Records concerning the custody and condition of the samples are maintained in field and laboratory records.

The contractor shall maintain COC records for all field and field quality control (QC) samples. A sample is defined as being under a person's custody if any of the following conditions exist: (1) it is in their possession, (2) it is in their view, after being in their possession, (3) it was in their possession and they locked it up or, (4) it is in a designated secure area.

All sample containers shall be sealed in a manner that shall prevent or detect tampering if it occurs. In no case shall tape be used to seal sample containers. The following minimum information concerning the sample shall be documented on the COC form.

- Unique sample identification;
- Date and time of sample collection;
- Source of sample (including name, location, and sample type);
- Designation of MS/MSD;
- Preservative used;
- Analyses required;
- Name of collector(s);
- Pertinent field data (pH, temperature, etc.);
- Serial numbers of custody seals and transportation cases (if used);
- Custody transfer signatures and dates and times of sample transfer from the field to transporters and to the laboratory or laboratories; and
- Bill of lading or transporter tracking number (if applicable).

All samples shall be uniquely identified, labeled, and documented in the field at the time of collection as discussed in Section 2.11.

Samples collected in the field shall be transported to the laboratory or field testing site as expeditiously as possible. When a 4°C requirement for preserving the sample is indicated, the samples shall be packed in ice or chemical refrigerant to keep them cool during collection and transportation. During transit, it is not always possible to rigorously control the temperature of the samples. As a general rule, storage at low temperature is the best way to preserve most samples. A temperature blank (a 40 ml vial filled with water or similar) shall be included in every cooler and used to determine the internal temperature of the cooler upon receipt of the cooler at the laboratory.

When transferring samples, the individuals relinquishing and receiving shall sign, date, and note the time on the COC record. The analytical laboratory shall maintain a file copy, and the completed original shall be returned to the Contractor PM as part of the final analytical report. This record shall serve to document sample custody transfer from the sampler to the laboratory.

Samples collected in the field shall be transported to the laboratory or field testing site as expeditiously as possible via overnight courier. Air bills shall be retained as part of the permanent

documentation and all sample shipments shall be regulated by the DOT, as described in 49 CFR Parts 171 to 177.

Once the samples have been received by the laboratory, a file of the original documents (e.g., COC forms, special analytical services request form, etc.) pertinent to sample custody and sample analytical protocol shall be maintained, and a copy of the COC file will be sent to the prime Contractor Project Manager.

2.12 FIELD QUALITY CONTROL SAMPLES

2.12.1 Material Blank

No Material Blanks will be collected for this Project.

All distilled decontamination water shall be procured in large batches and each batch shall be evaluated using a material blank. A material blank is a sample of the distilled decontamination water that shall be analyzed for all laboratory analyses requested for the environmental samples collected at the site. Environmental samples shall be assessed based on the results of the material blanks if similar compounds are detected in the material blanks, equipment blanks, and environmental sample. If material blanks contain analytes or compounds of concern, the source of water shall be changed immediately.

Material blanks shall be designated by “Site Identifier-MB” and numbered sequentially.

2.12.2 Equipment Blank

An Equipment Blank is a sample of distilled water of known quality poured into or over or pumped through a previously used and decontaminated sampling device, collected in a sample container, and transported to the laboratory for analysis. Equipment Blanks shall not be used if sampling equipment clean and dedicated or disposable for single use. Equipment Blanks are used to assess the effectiveness of equipment decontamination procedures. The frequency of collection for Equipment Blanks is specified in the site-specific Work Plan. Equipment Blanks shall be collected immediately after the equipment has been decontaminated. The blank shall be analyzed for all laboratory analyses requested for the environmental samples collected at the site.

Equipment blanks shall be designated by “Site identifier -EB” and numbered sequentially.

2.12.3 Trip Blank

The trip blank consists of a VOC sample vial filled in the laboratory with ASTM Type II reagent grade water, transported to the sampling site, handled like an environmental sample and returned to the laboratory for analysis. Trip blanks are not opened in the field. Trip blanks are prepared only when VOC water samples are taken and are analyzed only for VOC analytes. Trip

blanks are used to assess the potential introduction of contaminants from sample containers or during the transportation and storage procedures. One trip blank shall accompany each cooler of samples sent to the laboratory for analysis of VOCs. Trip blanks shall only be used for non-frozen samples.

Trip blanks shall be designated by Site Identifier-TB", and numbered sequentially.

2.12.4 Field Duplicates

A field duplicate sample is a second sample collected at the same location as the original sample. Duplicate samples are collected simultaneously or in immediate succession, using identical recovery techniques, and treated in an identical manner during storage, transportation, and analysis. The sample containers are assigned an identification number in the field such that they cannot be identified (blind duplicate) as duplicate samples by laboratory personnel performing the analysis.

Duplicate sample results are used to assess precision of the sample collection process. Precision of soil samples to be analyzed for VOCs is assessed from collocated samples because the compositing process required to obtain uniform samples could result in loss of the compounds of interest. The frequency of collection for field duplicates in the site-specific Work Plan.

3. FIELD MEASUREMENTS

3.1 General Field Measurement Parameters SOP (AI-W-FM-01)

The following parameters shall be measured in the field:

- Organic and explosive vapors, using a Photovac 2020 PID or equivalent;
- Water level, using a cable electronic water level indicator or equivalent;
- Immiscible layer, using the Solinst Model 121 Interface Meter or equivalent;
- EC, pH, temperature, ORP, and DO, using an ORION hand held meter or equivalent;
- Groundwater turbidity, using a portable LaMotte Model 2008 turbidimeter or equivalent;
- Site features and certain sampling locations (Soil Gas Survey locations, boreholes, surface soil samples, sediment samples, surface water samples), elevation, and coordinates shall be surveyed using GPS; and
- Monitoring well, ditch bed, and water reference elevation and coordinates using a state registered land surveyor (RLS).

3.2 Field Equipment Calibration and Quality Control SOP (AI – W FM-02)

3.2.1 Calibration Frequencies

All field instruments shall be calibrated on a daily basis, if they are used that day. This does not apply to the PID, which shall be calibrated at least twice per day. In some instances, calibration shall be performed more frequently. Calibration shall provide quality assurance (QA) checks on all field equipment used during implementation of the field investigations. Each instrument shall have an individual identification number. This number shall be transcribed on field data records when using a particular instrument for a sampling event. All calibration, repair, and service records shall be kept in individual equipment log books maintained for each type of instrument. Field equipment that consistently fails to meet calibration standards or exceeds manufacturer's critical limits shall be promptly repaired or replaced. The Contractor shall record equipment calibration on a Field Instrument Calibration Check Form (Appendix C).

3.2.2 Calibration Procedures

The following are examples of calibration procedures that may be performed during field investigations.

3.2.2.1 Photo Ionization Detector

The PID shall be calibrated each day prior to the start of field activities. If the PID is in continuous operation, it shall be calibrated at 4-hour intervals. Instrument calibration shall be performed using isobutylene calibration gas of known concentration (100 or 250 ppm). All adjustments to instrument settings shall be recorded in a field log book.

3.2.2.2 Interface Meter

Calibration of the Interface Meters is performed by checking the infrared and conductivity circuits. The infrared circuit detects the presence of a liquid, while a conductivity circuit differentiates between conductive liquid (water) and nonconductive liquid (NAPL).

To check the infrared circuit, follow these procedures: with the main and probe switches on, insert the cleaning brush into the base of the probe until it reaches the zero measuring point. The zero measuring point is the juncture between the stainless steel body of the probe and the base plug. This cuts the infrared beam and activates a steady tone and two lights.
To check the conductivity circuit, follow these procedures: with both the main and probe switches on, insert the probe into normal tap water as far as the zero measuring point. This causes a single light and intermittent tone to activate.

The tape is calibrated annually by using a surveyor's steel tape to adjust for stretching of the calibrated line.

3.2.2.3 Electrical Conductivity, pH, Temperature, Dissolved Oxygen, and Turbidity

Each of these water quality parameters shall be calibrated at each groundwater sampling location and during well development and sample purging.

The pH function shall be calibrated immediately before well development and purging using at least two buffer solutions that bracket the expected pH.

The EC function shall be calibrated using two solutions of known value that bracket the expected ranges of conductivities.

The DO function shall be calibrated against temperature-compensated, air-saturated water.

The calibration of the portable turbidimeter shall be evaluated by using two supplied standards within the range of anticipated sample turbidities. These standards have been carefully manufactured and are guaranteed to be accurate within one percent.

3.2.2.4 Field Quality Assurance / Quality Control Program

To ensure that sampling and monitoring activities shall meet DQOs, QC checks shall be implemented for parameters measured in the field. All QC control check information shall be recorded in project-specific field log books and/or forms. The following sections discuss control parameters, control units, and corrective actions for the RI field investigation.

3.2.2.5 Control Parameters

Several parameters shall be controlled during the field sampling and measurement activities. As previously described, calibration of field instruments and operational checks shall be conducted periodically. The frequency of field control check duplicates shall be a minimum of 10 percent of all field measurements. Temperature, pH, EC, DO, and turbidity shall be checked at the same frequency. As applicable, the materials used to verify control parameter measurements shall be from certified sources. Instrument use, maintenance, and calibration shall follow manufacturer guidelines.

3.2.2.6 Control Limits

Field Equipment control limits are specified the site-specific Work Plan. Field instrument calibration accuracy and duplicate precision for field measurements must meet acceptance criteria, or instrument readings shall be considered suspect. Appropriate corrective actions shall be taken whenever field instruments fail to meet acceptance for accuracy and precision.

3.2.2.7 Corrective Action

The corrective action required for field instruments that are used to measure water quality parameters shall include recalibrating and re-measuring the parameter. Corrective action for all

field instruments shall involve a review of the operator's manual. If necessary, instrument maintenance and repairs shall be performed as corrective actions in addition to normally scheduled maintenance operations. Any maintenance shall be recorded on a Field Instrument Calibration Check Form (Appendix A).

3.3 Equipment Maintenance and Decontamination SOP (AI-W-FM-03)

All field measurement equipment shall be decontaminated according to the specifications in Section 1.16 prior to any measurement activities and shall be protected from contamination until ready for use.

3.4 Organic and Explosive Vapor Measurement SOP (AI-W-FM-04)

During borehole advancement, the air in the breathing zone of on-site personnel shall be evaluated for the presence of organic (e.g., VOCs, SVOCs, petroleum hydrocarbons) and explosive vapors (lower explosive limit and oxygen content) using a Photovac 2020 PID and explosimeter (Industrial Scientific Model MX 251), respectively, or equivalents. Air monitoring data shall be tabulated on a Health and Safety Exposure Monitoring Form from the site-specific HASP. Procedures provided in the site-specific HASP shall be followed. In addition to monitoring the breathing zone around the borehole, the PID shall be used to screen for organic vapors in the well bore each time a well casing cap is removed for developing and purging groundwater monitoring wells and when performing field headspace screening of split-spoon soil samples.

3.5 Groundwater Level Measurement SOP (AI-W-FM-05)

Water levels will not be collected for this project.

3.6 Immiscible Layer Measurement SOP (AI-W-FM-06)

Immiscible Layer measurements are not part of this project.

The Solinst Model 121 Interface Meter or its equivalent shall also be used to monitor the overburden groundwater table for the presence of an immiscible layer such as petroleum product. Depth and thickness shall be measured to the nearest 0.01 foot.

3.7 Electrical Conductivity, pH, Temperature, Oxidation/Reduction Potential, and Dissolved Oxygen Measurement SOP (AI-W-FM-07)

During monitoring well development and sample purging, the above-referenced water quality parameters shall be monitored using portable field equipment. The Contractor shall monitor EC, pH, groundwater temperature, ORP, and DO using a hand-held multi-function water quality

meter. A multiprobe sampling chamber (flow cell) may be used for measuring parameters simultaneously. The probes of the meters are attached to the individual sample ports of the flow cell. When groundwater is pumped in-line into the flow cell from the monitoring well, continuous readings of these water quality indicator parameters shall be recorded until stabilization is reached for each parameter.

3.8 Groundwater Turbidity SOP (AI-W-FM-08)

A portable turbidimeter with an accuracy of two percent of reading or 0.05 NTU, shall be used to facilitate well development.

4. RECORD KEEPING

4.1 GENERAL RECORD KEEPING SOP (AI-W RK-01)

The Contractor shall maintain field records sufficient to recreate all sampling and measurement activities and to meet all Project data loading requirements. The requirements listed in this section apply to all measuring and sampling activities. Requirements specific to individual activities are listed in the section that addresses each activity. These records shall be archived in an easily accessible form and made available to the Facility RPM upon request.

The following information shall be recorded with indelible ink in a permanently bound notebook with sequentially numbered pages for all field activities:

- Location,
- Date and time,
- Identity of people performing activity, and
- Weather conditions.

For field measurements, the numerical value and units of each measurement, and the identity of and calibration results for each field instrument shall also be recorded on field logs.

The following additional information shall be recorded for all sampling activities:

- Sample type and sampling method;
- The identity of each sample and depth(s), where applicable, from which it was collected;
- The amount of each sample;
- Sample description (e.g., color, odor, clarity);
- Identification of sampling devices; and
- Identification of conditions that might affect the representativeness of a sample (e.g., refueling operations, damaged casing).

Records shall be kept for all activities associated with the field activities, as a means to maintain full documentation of project QA/QC procedures and compliance. In general, all documents shall be completed in permanent black ink. Errors shall be corrected by crossing them out with a single line and then dating and initialing. The use of correction fluids shall not be allowed. The documents used during the field investigation shall remain on site (if possible) during the entire effort so that they can be reviewed by interested parties. Forms shall be kept organized and in a central file also located on site, if applicable. Records shall be kept in the form of field log books and standardized forms which have been included as Appendix A.

4.2 Log Book SOP (AI-W-RK-02)

The Contractor field personnel shall maintain a field log book during the investigation. The field log book is the master field investigation document that is a bound book with a hard cover and sequentially numbered pages. The primary objective of the field log book is to maintain, within one document, the actual field data or references to other field documents that contain a specific description of every activity that has occurred in the field on any given day. Any administrative occurrences, conditions, or activities that have affected the field work shall be recorded in the field log book.

All field activities entered into the field log book shall be signed and dated by the responsible party. The following is a list of the type of information that shall be recorded in the field log book:

- Name and title of author, date and time of entry, and physical/environmental conditions during the field activity;
- Name and address of field contact;
- Name and titles of field crew;
- Name and titles of all site visitors;
- Documentation of Health and Safety activities;
- Type of sampled media (e.g., soil, groundwater);
- Number and volume of samples taken;
- Description of sampling points;
- Date and time of overall sample collection;
- Sample identification numbers;
- References, maps and descriptions of photographs;
- General decontamination procedures;
- Instrument calibration;
- Records of telephone conversations; and
- Weather conditions

4.3 Field Data Forms SOP (AI-W-RK-03)

In addition to the above-referenced field log book, the Contractor shall complete and maintain standardized field data forms for specified field activities. Field data forms are included in Appendix A. They consist of the following:

- Employee/Visitor Daily Roster;
- Tailgate Safety Meeting Report;
- Field Instrument Calibration Check Form;
- Water Quality Analyzer Check Form;
- Field Sampling Report (Soil/Sediment);
- Field Sampling Report (Groundwater/Surface Water);
- HTRW Drill Log (Eng. Form 5056-R);
- Borehole Abandonment Log;
- Waste Inventory Tracking Log
- Quality Assurance Report Daily Log (Eng. Form 2538-1);

5. REFERENCES

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Appendix C – Field Forms

Rolling Knolls Data Gap Investigation

EMPLOYEE/VISITOR DAILY ROSTER

This roster is required for emergency response planning. All personnel arriving to and from the site must sign this roster. This log does not replace the H&S Orientation

Site Name:	**Contract No.:**
Date:	**Delivery Order No.**
Project Manager:	

DATE	NAME	COMPANY	TIME ONSITE	TIME OFF-SITE

TAILGATE SAFETY MEETING ATTENDEES		DATE	
NAME	**COMPANY**		**SIGNATURE**

Meeting conducted by _____ Title _____

Signature _____ Date/Time _____

Tailgate Safety Briefing

Project:

Project Number:

Location:

Officer Conducting Briefing:

Date:

HEALTH & SAFETY REVIEW

- ☐ H & S Objectives
- ☐ Site History
- ☐ Chain of Command
- ☐ Visitor Policy
- ☐ Site Control
- ☐ General Site Hazards
- ☐ Chemical Hazards
- ☐ Physical Hazards
- ☐ Exposure Pathways
- ☐ Site Emergency Procedures
- ☐ Hospital Location
- ☐ Decontamination Procedures
- ☐ Noise Monitoring

FIELD ACTIVITY

ATTENDEES

Daily Field Summary

Project:
Project Number:
Location:
Field Personnel:
Date:

FIELD CONDITIONS

Weather: Temp:
Precipitation: Wind:

FIELD WORK SUMMARY

COMMENTS

HTRW DRILLING LOG

	DISTRICT	HOLE NUMBER
1. COMPANY NAME	2. DRILLING CONTRACTOR	SHEET SHEETS OF
3. PROJECT	4. LOCATION F	
5. NAME OF DRILLER	6. MANUFACTURER'S DESIGNATION OF DRILL	
7. SIZES AND TYPES OF DRILLING AND SAMPLING EQUIPMENT	8. HOLE LOCATION	
	9. SURFACE ELEVATION	
	10. DATE STARTED / 11. DATE COMPLETED	
12. OVERBURDEN THICKNESS	15. DEPTH GROUNDWATER ENCOUNTERED	
13. DEPTH DRILLED INTO ROCK	16. DEPTH TO WATER AND ELAPSED TIME AFTER DRILLING COMPLETED	
14. TOTAL DEPTH OF HOLE	17. OTHER WATER LEVEL MEASUREMENTS (SPECIFY)	

18. GEOTECHNICAL SAMPLES	DISTURBED	UNDISTURBED	19. TOTAL NUMBER OF CORE BOXES

20. SAMPLES FOR CHEMICAL ANALYSIS	VOC	METALS	OTHER (SPECIFY)	OTHER (SPECIFY)	OTHER (SPECIFY)	21. TOTAL CORE RECOVERY %

22. DISPOSITION OF HOLE	BACKFILLED	MONITORING WELL	OTHER (SPECIFY)	23. SIGNATURE OF INSPECTOR
Cuttings Drummed	see next			

LOCATION SKETCH/COMMENTS SCALE: 1" =

PROJECT	HOLE NO

ENG FORM 5056-R, AUG 94 (Proponent:

Client/Project Name: _____
Project Number: _____
Geologist: _____
Drilling Contractor: _____
Drilling Method: _____

Boring Number: _____
Date Begun: _____
Date Finished: _____
Total Depth: _____
Page _____ of _____

Photoionization Dectector	Well Installation Details	Sample Number	Blow Count	Direct Push Interval	Sampled Interval	Depth in Feet	Recovery	Soil Group Symbol (USCS)	Schematic of Boring/Probe Location
	Ground Surface	Sample Information							Lithologic Description
						1			
						2			
						3			
						4			
						5			
						6			
						7			
						8			
						9			
						10			
						11			
						12			
						13			
						14			
						15			
						16			
						17			
						18			
						19			
						20			

Notes/Observations/Comments:

Depth to Water ATD: _____

Client/Project Name: _____
Project Number: _____
Geologist: _____
Drilling Contractor: _____
Drilling Method: _____

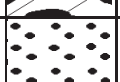
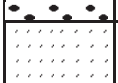
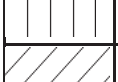
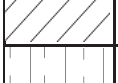
Boring Number: _____
Date Begun: _____
Date Finished: _____
Total Depth: _____
Page _____ of _____

Photoionization Dectector	Well Installation Details	Sample Number	Blow Count	Direct Push Interval	Sampled Interval	Depth in Feet	Recovery	Soil Group Symbol (USCS)	Schematic of Boring/Probe Location
	Ground Surface	Sample Information							Lithologic Description
						1			
						2			
						3			
						4			
						5			
						6			
						7			
						8			
						9			
						10			
						11			
						12			
						13			
						14			
						15			
						16			
						17			
						18			
						19			
						20			

Notes/Observations/Comments:

Depth to Water ATD: _____

SOIL CLASSIFICATION CHART

MAJOR DIVISIONS			USCS SYMBOLS GRAPH	USCS SYMBOLS LETTERS	TYPICAL DESCRTIPTIONS
COARSE-GRAINED SOILS	GRAVEL AND GRAVELLY SOILS	CLEAN GRAVELS (LITTLE OR NO FINES)		GW	WELL-GRADED GRAVELS, AND GRAVEL-SAND MIXTURES, WITH LITTLE OR NO FINES CONTENT.
				GP	POORLY-GRADED GRAVELS, AND GRAVEL-SAND MIXTURES, WITH LITTLE OR NO FINES CONTENT.
	MORE THAN 50% OR COARSE FRACTION RETAINED ON NO. 4 SIEVE	GRAVELS WITH FINES (APPRECIABLE AMOUNT OF FINES)		GM	SILTY GRAVELS, AND GRAVEL-SAND-SILT MIXTURES.
				GC	CLAYEY GRAVELS, AND GRAVEL-SAND-CLAY MIXTURES.
MORE THAN 50% OF MATERIAL IS LARGER THAN NO.200 SIEVE	SAND AND SANDY SOILS	CLEAN SANDS (LITTLE OR NO FINES)		SW	WELL-GRADED SANDS, AND GRAVELLY SANDS, WITH LITTLE OR NO FINES CONTENT.
				SP	POORLY-GRADED SANDS, AND GRAVEL-SAND MIXTURES, WITH LITTLE OR NO FINES CONTENT.
	MORE THAN 50% OR COARSE FRACTION PASSING ON NO. 4 SIEVE	SANDS WITH FINES (APPRECIABLE AMOUNT OF FINES)		SM	SILTY SANDS, AND SILTY SAND -SILT MIXTURES.
				SC	CLAYEY SANDS, AND SAND-CLAY MIXTURES.
FINE-GRAINED SOILS	SILTS AND CLAYS	LIQUID LIMIT LESS THAN 50		ML	INORGANIC SILTS AND VERY FINE SANDS, ROCK FLOUR, SILTY OR CLAYEY FINE SANDS, OR CLAYEY SILTS WITH SLIGHT PLASTICITY.
				CL	INORGANIC CLAYS OF LOW TO MEDIUM PLASTICITY, GRAVELLY CLAYS, SANDY CLAYS, SILTY CLAYS, AND LEAN CLAYS.
				OL	ORGANIC SILTS AND ORGANIC SILTY CLAYS OF LOW PLASTICITY.
MORE THAN 50% OF MATERIAL IS SMALLER THAN NO.200 SIEVE	SILTS AND CLAYS	LIQUID LIMIT GREATER THAN 50		MH	INORGANIC SILTS, MICACEOUS OR DIATOMACEOUS FINE SANDS OR SILTY SOILS.
				CH	INORGANIC CLAYS OF HIGH PLASTICITY.
				OH	ORGANIC CLAYS OF MEDIUM TO HIGH PLASTICITY AND ORGANIC SILTS.
HIGHLY ORGANIC SOILS				PT	PEAT, HUMUS, SWAMP SOILS WITH HIGH NATURAL ORGANIC MATTER CONTENTS.

NOTE: DUAL SYMBOLS (GP/GM) ARE USED TO INDICATE BORDERLINE SOIL CLASSIFICATIONS.

MOISTURE CONTENT

DRY - ABSENCE OF MOISTURE, DUSTY, DRY TO THE TOUCH

DAMP - PERCEPTIBLE MOISTURE, BUT BELOW OPTIMUM MOISTURE CONTENT FOR COMPACTION. NO FREE WATER.

MOIST - PERCEPTIBLE MOISTURE, AT ABOUT OPTIMUM MOISTURE CONTENT FOR COMPACTION. NO FREE WATER.

WET - VISIBLE FREE WATER IN SAMPLE. PROBABLY ABOVE OPTIMUM MOISTURE CONTENT FOR COMPACTION.

ESTIMATED SOIL PERCENTAGES

TRACE -	O TO 5 %
SLIGHTLY -	5 TO 12 %
NO MODIFIER - (SILTY, SANDY)	12 TO 30 %
VERY -	30 TO 50 %

RELATIVE SOIL DENSITY AND CONSISTENCY

COARSE-GRAINED SOILS
(PARETHESIS INDICATE ESTIMATED VALUE)

DENSITY	SPT BLOWS PER FOOT
VERY LOOSE	0 TO 4
LOOSE	4 TO 10
MEDIUM DENSE	10 TO 30
DENSE	30 TO 50
VERY DENSE	GREATER THAN 50

FINE-GRAINED SOILS (PARENTHESIS INDICATE ESTIMATED VALUE)

CONSISTENCY	SPT BLOWS PER FOOT
VERY SOFT	0 TO 2
SOFT	2 TO 4
MEDIUM STIFF	4 TO 8
STIFF	8 TO 15
VERY STIFF	15 TO 30
HARD	GREATER THAN 30

FIELD MEASUREMENTS

- WATER LEVEL OBSERVED DURING DRILLING.
- STATIC WATER LEVEL MEASURED AFTER DRILLING.
- WATER SEEPAGE

LABORATORY TESTS

M=	MOISURE CONTENT (%)
D=	DRY DENSITY (PSF)
Tv=	TORVANE
Pp=	POCKET PENETROMETER
GS=	GRAIN SIZE
G2=	% PASSIING NO. 200 SIEVE
A=	ATTERBERG LIMITS

WELL CONSTRUCTION

- CONCRETE
- SOLID SCHEDULE 40 PVC WELL CASING AND BENTONITE SEAL
- SOLID SCHEDULE 40 PVC WELL CASING AND SAND FILTER PACK
- SLOTTED SCHEDULE 40 PVC WELL SCREEN AND SAND FILTER PACK
- BENTONITE

SOIL SAMPLES

- DIRECT-PUSH SAMPLE, USING ACRYLIC LINERS, LABORATORY ANALYSIS PERFORMED.
- SPLIT-SPOON SAMPLE, (3-INCH O.D.) 140 LB. HAMMER (NOT STANDARD SPT).
- DIRECT-PUSH SAMPLE, USING ACRYLIC LINERS, no LABORATORY ANALYSIS PERFORMED.
- GRAB SAMPLE FROM AUGER FLIGHTS OR HAND AUGER.
- NO SAMPLE RECOVERY

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ENVIRONMENTAL CONSULTING SERVICES
809 156TH STREET NE
ARLINGTON, WA 98223

FIGURE A-1

BORING LOG AND WELL SYMBOL LEGEND
TACO TIME SUBSURFACE INVESTIGATION
1420 EAST MADISON

Soil / Sediment Sample Collection Record

SAMPLE ID:

Sample Location:	Photo:
Description:	
Date:	
Field Personnel:	
Conditions:	Weather:

METHOD

Grab: Depth:

Composite:

Habitat	Depth	Comments

SAMPLE COLLECTION

Equipment: Trowel Push Sampler Other:

Sample Collection Time:

Sample ID	# Sample Containers	Size			Analysis								Other		
		8 oz glass jar	4 oz glass jar	zip-lock bag											

DESCRIPTION/COMMENTS

Water Sample Collection Record

SAMPLE ID:

Sample Location:

Description:

Date:

Field Personnel:

Conditions: | Weather:

FIELD PARAMETER MEASUREMENT

Field Meter:	Field Parameters:	Observation 1	Observation 2	Observation 3
	Temperature (Celcius)			
	pH (pH units)			
	SpCond (microS/cm)			
	Dissolved O_2 (mg/L)			
	ORP (mV)			

Method:

Measurement START Time: | Measurement END Time:

FLOW MEASUREMENT

Method: Flow Meter: Flume: Timed Volumetric:

Description:

Flow:

SAMPLE COLLECTION

Sample Method: Bottle Pump Other:

Sample Collection Time:

Sample ID	Time	# of Sample Containers	Size		Filtration		Analysis						Preservative			
			500-mL plastic bottle	250-mL plastic bottle	Filtered	Unfiltered							HCl	HNO_3	NaOH	NONE

COMMENTS

FIELD INSTRUMENT CALIBRATION CHECK FORM									
Instrument Model No:						Serial No.:			
Analyte:						Stock Solution Conc. (mg/L): $\mathbf{C_S}$			
Date	Volume of Stock Solution (L) V_S	Volume of Distilled Water (L) V_W	Standard Conc. (mg/L)	Lot No.	Exp. Date	Indicated Conc. (mg/L)	Diff. (%)	Comments	Technicians Initials

WATER QUALITY ANALYZER ACCURACY CHECK FORM									
Analyzer Model No.:						Serial No.:			
Date	Parameter	Units	Standard Value	Lot No.	Exp. Date	Instrument Reading		Comments	Initials
						Before Adjustment	After Adjustment		

Borehole Abandonment Log

Location Identifier	Direct Push (DP) or Auger (A)	Date	Total Depth (ft)	Diameter (in)	Backfill Material	Surface Completion

Notes:

HBP - Hydrated bentonite pellets

BHS- Bore hole soil

AS- Asphalt

CEM- Neat Cement

SO- Soil

Waste Inventory Tracking Log

Drum Identifier	Contents (Site, Matrix, Sample Locations, Level of contamination)	Date Filled	Storage Location	Analytical Results (Y/N)	Properly Disposed (Y/N and Subcontractor)	Manifest and Custody completed and returned (Y/N)

Notes:

QUALITY ASSURANCE REPORT *(QAR)* DAILY LOG OF ACTIVITIES For use of this form see ER 1180-1-6;.	1. REPORT NUMBER	
9	3. DATE (YYYYMMDD)	4. CONTRACT NUMBER
	5. CONTRACTOR *(or hired labor)*	
	6. WEATHER	
7. CQC CONTROL PHASES ATTENDED AND INSTRUCTION GIVEN		
8. RESULTS OF QA ACTIVITIES AND TESTS, DEFICIENCIES OBSERVED, ACTIONS TAKEN AND CORRECTIVE ACTION OF CONTRACTOR. INCLUDE COMMENT PERTAINING TO CONTRACTORS CQC ACTIVITIES.		
9. VERBAL INSTRUCTION GIVEN TO CONTRACTOR (*Include names, reactions and remarks)*		
10. HAS ANYTHING DEVELOPED ON THE WORK, WHICH MIGHT LEAD TO A CHANGE ORDER OR FINDING OF FACT? YES☐ ☐ NO		

ENG FORM 2538-1, MAY 1994 PREVIOUS EDITIONS ARE OBSOLETE. Page 1 of 2

11. INFORMATION ON PROGRESS OF WORK, CAUSES FOR DELAYS AND EXTENT OF DELAYS, WEATHER, PLANT, MATERIAL, ETC.,
12. INFORMATION, INSTRUCTIONS OR ACTIONS TAKEN NOT COVERED ON QCR REPORT OR DISAGREEMENTS.
13. SAFETY: *(Include any infractions of approved safety plan, safety manual or instructions from Government personnel. Specify corrective action taken.)*
14. REMARKS: *(Include visitors to project and miscellaneous remarks pertinent to work.)*

15a. NAME AND TITLE *(Last, First MI)*	b. DATE *(YYYYMMDD)*	*c.* QA REPRESENTATIVE'S SIGNATURE
16a. NAME AND TITLE *(Last, First MI)*	b. DATE *(YYYYMMDD)*	*c.* SUPERVISOR'S SIGNATURE

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Appendix D – Performance Criteria
Rolling Knolls Data Gap Investigation

Appendix D -Measurement Perfomance Criteria
Table D-1 **Soil Samples - SW846, Volatile Organic Compounds by GC/MS**

Matrix	**Soil Samples - SW846**
Method Description	**Volatile Organic Compounds by GC/MS**
Method Code	**8260C**
Prep Method	**5035A_FW**
Units	**ug/Kg**

Analyte Description	CAS Number	RL	MDL	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
1,1,1,2-Tetrachloroethane	630-20-6	1	0.19	ug/Kg	73	124	30	73	124	30		
1,1,1-Trichloroethane	71-55-6	1	0.233	ug/Kg	78	132	30	78	132	30		
1,1,2,2-Tetrachloroethane	79-34-5	1	0.214	ug/Kg	69	123	30	69	123	30		
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	1	0.301	ug/Kg	78	136	30	78	136	30		
1,1,2-Trichloroethane	79-00-5	1	0.178	ug/Kg	75	120	30	75	120	30		
1,1-Dichloroethane	75-34-3	1	0.206	ug/Kg	76	129	30	76	129	30		
1,1-Dichloroethene	75-35-4	1	0.225	ug/Kg	77	132	30	77	132	30		
1,2,3-Trichlorobenzene	87-61-6	1	0.181	ug/Kg	77	120	30	77	120	30		
1,2,4-Trichlorobenzene	120-82-1	1	0.358	ug/Kg	75	120	30	75	120	30		
1,2-Dibromo-3-Chloropropane	96-12-8	1	0.46	ug/Kg	60	126	30	60	126	30		
1,2-Dichlorobenzene	95-50-1	1	0.144	ug/Kg	80	120	30	80	120	30		
1,2-Dichloroethane	107-06-2	1	0.296	ug/Kg	70	132	30	70	132	30		
1,2-Dichloropropane	78-87-5	1	0.423	ug/Kg	73	124	30	73	124	30		
1,3-Dichlorobenzene	541-73-1	1	0.159	ug/Kg	80	120	30	80	120	30		
1,4-Dichlorobenzene	106-46-7	1	0.225	ug/Kg	80	120	30	80	120	30		
1,4-Dioxane	123-91-1	20	9.18	ug/Kg								
2-Butanone (MEK)	78-93-3	5	2.71	ug/Kg	75	120	30	75	120	30		
2-Hexanone	591-78-6	5	1.71	ug/Kg	78	120	30	78	120	30		
2-Methyl-2-propanol	75-65-0	10	3.3	ug/Kg	72	120	30	72	120	30		
4-Methyl-2-pentanone (MIBK)	108-10-1	5	1.56	ug/Kg	80	122	30	80	122	30		
Acetone	67-64-1	6	5.72	ug/Kg	63	131	30	63	131	30		
Benzene	71-43-2	1	0.258	ug/Kg	80	123	30	80	123	30		
Bromoform	75-25-2	1	0.425	ug/Kg	48	142	30	48	142	30		
Bromomethane	74-83-9	1	0.474	ug/Kg	68	136	30	68	136	30		
Carbon disulfide	75-15-0	1	0.266	ug/Kg	67	136	30	67	136	30		
Carbon tetrachloride	56-23-5	1	0.387	ug/Kg	72	136	30	72	136	30		
Chlorobenzene	108-90-7	1	0.177	ug/Kg	80	120	30	80	120	30		
Chlorobromomethane	74-97-5	1	0.281	ug/Kg	76	127	30	76	127	30		
Chlorodibromomethane	124-48-1	1	0.194	ug/Kg	62	128	30	62	128	30		
Chloroethane	75-00-3	1	0.522	ug/Kg	65	134	30	65	134	30		
Chloroform	67-66-3	1	0.319	ug/Kg	79	126	30	79	126	30		
Chloromethane	74-87-3	1	0.435	ug/Kg	48	150	30	48	150	30		
cis-1,2-Dichloroethene	156-59-2	1	0.152	ug/Kg	80	123	30	80	123	30		
cis-1,3-Dichloropropene	10061-01-5	1	0.273	ug/Kg	72	120	30	72	120	30		
Cyclohexane	110-82-7	1	0.221	ug/Kg	80	132	30	80	132	30		
Dichlorobromomethane	75-27-4	1	0.257	ug/Kg	73	124	30	73	124	30		
Dichlorodifluoromethane	75-71-8	1	0.338	ug/Kg	40	146	30	40	146	30		
Ethylbenzene	100-41-4	1	0.199	ug/Kg	80	120	30	80	120	30		
Ethylene Dibromide	106-93-4	1	0.18	ug/Kg	79	120	30	79	120	30		
Isopropylbenzene	98-82-8	1	0.126	ug/Kg	80	120	30	80	120	30		
Methyl acetate	79-20-9	5	4.3	ug/Kg	58	143	30	58	143	30		
Methyl tert-butyl ether	1634-04-4	1	0.125	ug/Kg	80	125	30	80	125	30		
Methylcyclohexane	108-87-2	1	0.499	ug/Kg	79	133	30	79	133	30		
Methylene Chloride	75-09-2	1	0.464	ug/Kg	76	127	30	76	127	30		
m-Xylene & p-Xylene	179601-23-1	1	0.174	ug/Kg	80	120	30	80	120	30		

o-Xylene	95-47-6	1	0.194	ug/Kg	80	120	30	80	120	30		
Styrene	100-42-5	1	0.278	ug/Kg	80	120	30	80	120	30		
Tentatively Identified Compound	STL00231			ug/Kg			30			30		
Tetrachloroethene	127-18-4	1	0.143	ug/Kg	78	123	30	78	123	30		
Toluene	108-88-3	1	0.234	ug/Kg	80	120	30	80	120	30		
trans-1,2-Dichloroethene	156-60-5	1	0.246	ug/Kg	78	128	30	78	128	30		
trans-1,3-Dichloropropene	10061-02-6	1	0.266	ug/Kg	68	120	30	68	120	30		
Trichloroethene	79-01-6	1	0.144	ug/Kg	79	120	30	79	120	30		
Trichlorofluoromethane	75-69-4	1	0.406	ug/Kg	67	142	30	67	142	30		
Vinyl chloride	75-01-4	1	0.546	ug/Kg	56	147	30	56	147	30		
1,2-Dichloroethane-d4 (Surr)	17060-07-0			ug/Kg			30			30	77	145
4-Bromofluorobenzene	460-00-4			ug/Kg			30			30	79	125
Dibromofluoromethane (Surr)	1868-53-7			ug/Kg							48	150
Toluene-d8 (Surr)	2037-26-5			ug/Kg			30			30	80	120

Appendix D -Measurement Perfomance Criteria
Table D-2 **Soil Samples - SW846, Semivolatile Organic Compounds (GC/MS)**

Matrix	**Soil Samples - SW846**
Method Description	**Semivolatile Organic Compounds (GC/MS)**
Method Code	**8270D**
Prep Method	**3546**
Units	**ug/Kg**

Analyte Description	CAS Number	RL	MDL	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
1,1'-Biphenyl	92-52-4	330	4.39	ug/Kg	65	110	30	65	110	30		
1,2,4,5-Tetrachlorobenzene	95-94-3	330	10.3	ug/Kg	64	110	30	64	110	30		
1,4-Dioxane	123-91-1	100	9.12	ug/Kg	31	81	30	31	81	30		
2,2'-oxybis[1-chloropropane]	108-60-1	330	5.99	ug/Kg	49	109	30	49	109	30		
2,3,4,6-Tetrachlorophenol	58-90-2	330	22.4	ug/Kg	58	113	30	58	113	30		
2,4,5-Trichlorophenol	95-95-4	330	33.7	ug/Kg	64	112	30	64	112	30		
2,4,6-Trichlorophenol	88-06-2	133	42.5	ug/Kg	63	113	30	63	113	30		
2,4-Dichlorophenol	120-83-2	133	21.2	ug/Kg	66	113	30	66	113	30		
2,4-Dimethylphenol	105-67-9	330	14.5	ug/Kg	63	107	30	63	107	30		
2,4-Dinitrophenol	51-28-5	266	163	ug/Kg	37	125	30	37	125	30		
2,4-Dinitrotoluene	121-14-2	67	35.6	ug/Kg	65	124	30	65	124	30		
2,6-Dinitrotoluene	606-20-2	67	23.9	ug/Kg	67	121	30	67	121	30		
2-Chloronaphthalene	91-58-7	330	15.3	ug/Kg	65	109	30	65	109	30		
2-Chlorophenol	95-57-8	330	11.8	ug/Kg	63	106	30	63	106	30		
2-Methylnaphthalene	91-57-6	330	9.25	ug/Kg	64	108	30	64	108	30		
2-Methylphenol	95-48-7	330	12.4	ug/Kg	63	108	30	63	108	30		
2-Nitroaniline	88-74-4	330	12.4	ug/Kg	59	119	30	59	119	30		
2-Nitrophenol	88-75-5	330	33.1	ug/Kg	64	112	30	64	112	30		
3,3'-Dichlorobenzidine	91-94-1	133	50	ug/Kg	4	119	30	4	119	30		
3-Nitroaniline	99-09-2	330	37.3	ug/Kg	31	102	30	31	102	30		
4,6-Dinitro-2-methylphenol	534-52-1	266	53.7	ug/Kg	64	129	30	64	129	30		
4-Bromophenyl phenyl ether	101-55-3	330	13.1	ug/Kg	67	113	30	67	113	30		
4-Chloro-3-methylphenol	59-50-7	330	18.6	ug/Kg	66	114	30	66	114	30		
4-Chloroaniline	106-47-8	330	23.1	ug/Kg	20	98	30	20	98	30		
4-Chlorophenyl phenyl ether	7005-72-3	330	11.7	ug/Kg	66	110	30	66	110	30		
4-Methylphenol	106-44-5	330	20.7	ug/Kg	61	108	30	61	108	30		
4-Nitroaniline	100-01-6	330	38	ug/Kg	50	110	30	50	110	30		
4-Nitrophenol	100-02-7	670	53.9	ug/Kg	47	123	30	47	123	30		
Acenaphthene	83-32-9	330	24.1	ug/Kg	53	110	30	53	110	30		
Acenaphthylene	208-96-8	330	3.42	ug/Kg	64	108	30	64	108	30		
Acetophenone	98-86-2	330	16.2	ug/Kg	61	103	30	61	103	30		
Anthracene	120-12-7	330	10.1	ug/Kg	67	114	30	67	114	30		
Atrazine	1912-24-9	133	8.35	ug/Kg	44	145	30	44	145	30		
Benzaldehyde	100-52-7	330	14.4	ug/Kg	39	113	30	39	113	30		
Benzo[a]anthracene	56-55-3	33	11.5	ug/Kg	67	115	30	67	115	30		
Benzo[a]pyrene	50-32-8	33	8.81	ug/Kg	63	108	30	63	108	30		
Benzo[b]fluoranthene	205-99-2	33	8.56	ug/Kg	64	116	30	64	116	30		
Benzo[g,h,i]perylene	191-24-2	330	9.76	ug/Kg	61	113	30	61	113	30		
Benzo[k]fluoranthene	207-08-9	33	6.49	ug/Kg	67	115	30	67	115	30		
Bis(2-chloroethoxy)methane	111-91-1	330	25.8	ug/Kg	62	107	30	62	107	30		
Bis(2-chloroethyl)ether	111-44-4	33	11.5	ug/Kg	60	107	30	60	107	30		
Bis(2-ethylhexyl) phthalate	117-81-7	330	17.5	ug/Kg	69	124	30	69	124	30		
Butyl benzyl phthalate	85-68-7	330	15.5	ug/Kg	70	123	30	70	123	30		
Caprolactam	105-60-2	330	51.5	ug/Kg	59	140	30	59	140	30		

Carbazole	86-74-8	330	12.6	ug/Kg	64	113	30	64	113	30		
Chrysene	218-01-9	330	5.59	ug/Kg	71	122	30	71	122	30		
Dibenz(a,h)anthracene	53-70-3	33	14.3	ug/Kg	66	119	30	66	119	30		
Dibenzofuran	132-64-9	330	4.65	ug/Kg	65	108	30	65	108	30		
Diethyl phthalate	84-66-2	330	4.79	ug/Kg	63	109	30	63	109	30		
Dimethyl phthalate	131-11-3	330	75.2	ug/Kg	65	109	30	65	109	30		
Di-n-butyl phthalate	84-74-2	330	58.4	ug/Kg	66	114	30	66	114	30		
Di-n-octyl phthalate	117-84-0	330	17.5	ug/Kg	65	122	30	65	122	30		
Fluoranthene	206-44-0	330	11.6	ug/Kg	64	113	30	64	113	30		
Fluorene	86-73-7	330	4.49	ug/Kg	65	109	30	65	109	30		
Hexachlorobenzene	118-74-1	33	15.7	ug/Kg	70	119	30	70	119	30		
Hexachlorobutadiene	87-68-3	67	7.04	ug/Kg	62	109	30	62	109	30		
Hexachlorocyclopentadiene	77-47-4	330	29	ug/Kg	22	124	30	22	124	30		
Hexachloroethane	67-72-1	33	11.4	ug/Kg	61	102	30	61	102	30		
Indeno[1,2,3-cd]pyrene	193-39-5	33	12.9	ug/Kg	62	121	30	62	121	30		
Isophorone	78-59-1	133	95.6	ug/Kg	63	107	30	63	107	30		
Naphthalene	91-20-3	330	5.72	ug/Kg	63	106	30	63	106	30		
Nitrobenzene	98-95-3	33	7.94	ug/Kg	63	110	30	63	110	30		
N-Nitrosodi-n-propylamine	621-64-7	33	24	ug/Kg	61	108	30	61	108	30		
N-Nitrosodiphenylamine	86-30-6	330	6.33	ug/Kg	67	113	30	67	113	30		
Pentachlorophenol	87-86-5	266	67.8	ug/Kg	44	126	30	44	126	30		
Phenanthrene	85-01-8	330	5.81	ug/Kg	66	112	30	66	112	30		
Phenol	108-95-2	330	12.2	ug/Kg	63	110	30	63	110	30		
Pyrene	129-00-0	330	8.23	ug/Kg	71	122	30	71	122	30		
2,4,6-Tribromophenol (Surr)	118-79-6			ug/Kg			30			30	10	123
2-Fluorobiphenyl	321-60-8			ug/Kg			30			30	25	104
2-Fluorophenol (Surr)	367-12-4			ug/Kg			30			30	18	106
Nitrobenzene-d5 (Surr)	4165-60-0			ug/Kg			30			30	19	105
Phenol-d5 (Surr)	4165-62-2			ug/Kg			30			30	26	101
Terphenyl-d14 (Surr)	1718-51-0			ug/Kg			30			30	25	127

Appendix D -Measurement Perfomance Criteria
Table D-3 **Soil Samples - SW846, Semivolatile Organic Compounds by GC/MS - Low Level**

Matrix	**Soil Samples - SW846**
Method Description	**Semivolatile Organic Compounds by GC/MS - Low Level**
Method Code	**8270D_LL**
Prep Method	**3541_LL**
Units	**ug/Kg**

Analyte Description	CAS Number	RL	MDL	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
1,1'-Biphenyl	92-52-4	33	1.4	ug/Kg	43	102	25	43	102	25		
1,2,4,5-Tetrachlorobenzene	95-94-3	33	1.44	ug/Kg	37	100	24	37	100	24		
2,2'-oxybis[1-chloropropane]	108-60-1	6.7	2.47	ug/Kg	38	121	28	38	121	28		
2,3,4,6-Tetrachlorophenol	58-90-2	33	13.9	ug/Kg	42	100	19	42	100	19		
2,4,5-Trichlorophenol	95-95-4	33	2.38	ug/Kg	44	100	19	44	100	19		
2,4,6-Trichlorophenol	88-06-2	33	1.83	ug/Kg	44	100	21	44	100	21		
2,4-Dichlorophenol	120-83-2	6.7	2.57	ug/Kg	48	100	26	48	100	26		
2,4-Dimethylphenol	105-67-9	33	2.07	ug/Kg	47	100	26	47	100	26		
2,4-Dinitrophenol	51-28-5	330	185	ug/Kg	28	103	17	28	103	17		
2,4-Dinitrotoluene	121-14-2	33	5.03	ug/Kg	54	107	20	54	107	20		
2,6-Dinitrotoluene	606-20-2	33	2.06	ug/Kg	52	110	17	52	110	17		
2-Chloronaphthalene	91-58-7	6.7	1.53	ug/Kg	49	100	22	49	100	22		
2-Chlorophenol	95-57-8	33	1.55	ug/Kg	51	100	31	51	100	31		
2-Methylnaphthalene	91-57-6	6.7	1.6	ug/Kg	50	100	29	50	100	29		
2-Methylphenol	95-48-7	33	9.56	ug/Kg	50	100	27	50	100	27		
2-Nitroaniline	88-74-4	170	15.2	ug/Kg	53	101	20	53	101	20		
2-Nitrophenol	88-75-5	33	5.31	ug/Kg	52	107	31	52	107	31		
3,3'-Dichlorobenzidine	91-94-1	33	31.1	ug/Kg	34	100	23	34	100	23		
3-Nitroaniline	99-09-2	170	8.48	ug/Kg	49	108	21	49	108	21		
4,6-Dinitro-2-methylphenol	534-52-1	170	57.5	ug/Kg	44	107	29	44	107	29		
4-Bromophenyl phenyl ether	101-55-3	33	2.33	ug/Kg	48	100	26	48	100	26		
4-Chloro-3-methylphenol	59-50-7	33	1.57	ug/Kg	47	100	29	47	100	29		
4-Chloroaniline	106-47-8	33	2.27	ug/Kg	46	100	31	46	100	31		
4-Chlorophenyl phenyl ether	7005-72-3	33	2.02	ug/Kg	49	100	22	49	100	22		
4-Nitroaniline	100-01-6	170	1.62	ug/Kg	49	111	21	49	111	21		
4-Nitrophenol	100-02-7	170	23.4	ug/Kg	22	101	19	22	101	19		
Acenaphthene	83-32-9	6.7	1.92	ug/Kg	48	101	22	48	101	22		
Acenaphthylene	208-96-8	6.7	1.46	ug/Kg	45	104	23	45	104	23		
Acetophenone	98-86-2	67	1.81	ug/Kg	39	100	28	39	100	28		
Anthracene	120-12-7	6.7	1.73	ug/Kg	50	111	26	50	111	26		
Atrazine	1912-24-9	67	14.6	ug/Kg	10	110	23	10	110	23		
Benzaldehyde	100-52-7	67	4.14	ug/Kg	10	100	36	10	100	36		
Benzo[a]anthracene	56-55-3	6.7	3.01	ug/Kg	48	100	24	48	100	24		
Benzo[a]pyrene	50-32-8	6.7	2.89	ug/Kg	48	106	21	48	106	21		
Benzo[b]fluoranthene	205-99-2	6.7	1.64	ug/Kg	46	100	26	46	100	26		
Benzo[g,h,i]perylene	191-24-2	6.7	1.44	ug/Kg	47	105	22	47	105	22		
Benzo[k]fluoranthene	207-08-9	6.7	2	ug/Kg	46	107	18	46	107	18		
Bis(2-chloroethoxy)methane	111-91-1	33	1.59	ug/Kg	45	100	30	45	100	30		
Bis(2-chloroethyl)ether	111-44-4	6.7	1.21	ug/Kg	47	100	28	47	100	28		
Bis(2-ethylhexyl) phthalate	117-81-7	330	35.5	ug/Kg	46	109	29	46	109	29		
Butyl benzyl phthalate	85-68-7	33	22.9	ug/Kg	48	104	27	48	104	27		
Caprolactam	105-60-2	170	21.7	ug/Kg	44	106	33	44	106	33		
Carbazole	86-74-8	6.7	1.56	ug/Kg	49	111	27	49	111	27		
Chrysene	218-01-9	6.7	3.7	ug/Kg	48	100	26	48	100	26		

Dibenz(a,h)anthracene	53-70-3	6.7	4.27	ug/Kg	47	106	24	47	106	24		
Dibenzofuran	132-64-9	33	1.46	ug/Kg	49	100	19	49	100	19		
Diethyl phthalate	84-66-2	33	11.7	ug/Kg	52	100	23	52	100	23		
Dimethyl phthalate	131-11-3	33	2.47	ug/Kg	52	100	18	52	100	18		
Di-n-butyl phthalate	84-74-2	33	14.6	ug/Kg	53	113	22	53	113	22		
Di-n-octyl phthalate	117-84-0	33	19.4	ug/Kg	43	112	22	43	112	22		
Fluoranthene	206-44-0	6.7	1.76	ug/Kg	52	100	26	52	100	26		
Fluorene	86-73-7	6.7	1.31	ug/Kg	49	100	22	49	100	22		
Hexachlorobenzene	118-74-1	6.7	2.39	ug/Kg	47	100	27	47	100	27		
Hexachlorobutadiene	87-68-3	6.7	1.95	ug/Kg	30	100	27	30	100	27		
Hexachlorocyclopentadiene	77-47-4	33	3.41	ug/Kg	31	100	22	31	100	22		
Hexachloroethane	67-72-1	33	1.72	ug/Kg	48	100	27	48	100	27		
Indeno[1,2,3-cd]pyrene	193-39-5	6.7	3.32	ug/Kg	48	105	22	48	105	22		
Isophorone	78-59-1	33	1.7	ug/Kg	41	100	29	41	100	29		
Methylphenol, 3 & 4	106-44-5	33	9.79	ug/Kg	51	100	24	51	100	24		
Naphthalene	91-20-3	6.7	1.3	ug/Kg	49	100	28	49	100	28		
Nitrobenzene	98-95-3	66.7	12.2	ug/Kg	48	100	28	48	100	28		
N-Nitrosodi-n-propylamine	621-64-7	6.7	2.26	ug/Kg	49	106	31	49	106	31		
N-Nitrosodiphenylamine	86-30-6	33	11.1	ug/Kg	48	109	26	48	109	26		
Pentachlorophenol	87-86-5	170	53.6	ug/Kg	31	100	28	31	100	28		
Phenanthrene	85-01-8	6.7	1.79	ug/Kg	48	106	27	48	106	27		
Phenol	108-95-2	33	10.1	ug/Kg	49	100	30	49	100	30		
Pyrene	129-00-0	6.7	1.58	ug/Kg	47	100	27	47	100	27		
2,4,6-Tribromophenol (Surr)	118-79-6			ug/Kg							22	116
2-Fluorobiphenyl	321-60-8			ug/Kg							37	107
2-Fluorophenol (Surr)	367-12-4			ug/Kg							38	106
Nitrobenzene-d5 (Surr)	4165-60-0			ug/Kg							41	112
Phenol-d5 (Surr)	4165-62-2			ug/Kg							40	110
Terphenyl-d14 (Surr)	1718-51-0			ug/Kg							32	115

Appendix D -Measurement Perfomance Criteria
Table D-4 **Soil Samples - SW846, Organochlorine Pesticides (GC)**

Matrix	**Soil Samples - SW846**
Method Description	**Organochlorine Pesticides (GC)**
Method Code	**8081B**
Prep Method	**3546**
Units	**ug/Kg**

Analyte Description	CAS Number	RL	MDL	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
1-Bromo-2-nitrobenzene	577-19-5			ug/Kg								
4,4'-DDD	72-54-8	6.7	1.14	ug/Kg	70	140	30	70	140	30		
4,4'-DDE	72-55-9	6.7	0.79	ug/Kg	71	137	30	71	137	30		
4,4'-DDT	50-29-3	6.7	1.23	ug/Kg	63	131	30	63	131	30		
Aldrin	309-00-2	6.7	1.01	ug/Kg	74	140	30	74	140	30		
alpha-BHC	319-84-6	2	0.68	ug/Kg	72	142	30	72	142	30		
beta-BHC	319-85-7	2	0.75	ug/Kg	65	137	30	65	137	30		
Chlordane (technical)	12789-03-6	67	16.2	ug/Kg	65	133	30	65	133	30		
delta-BHC	319-86-8	2	0.41	ug/Kg	70	143	30	70	143	30		
Dieldrin	60-57-1	2	0.87	ug/Kg	70	135	30	70	135	30		
Endosulfan I	959-98-8	6.7	1.02	ug/Kg	68	135	30	68	135	30		
Endosulfan II	33213-65-9	6.7	1.72	ug/Kg	64	130	30	64	130	30		
Endosulfan sulfate	1031-07-8	6.7	0.84	ug/Kg	66	143	30	66	143	30		
Endrin	72-20-8	6.7	0.96	ug/Kg	68	136	30	68	136	30		
Endrin aldehyde	7421-93-4	6.7	1.58	ug/Kg	68	132	30	68	132	30		
Endrin ketone	53494-70-5	6.7	1.3	ug/Kg	60	150	30	60	150	30		
gamma-BHC (Lindane)	58-89-9	2	0.62	ug/Kg	70	134	30	70	134	30		
Heptachlor	76-44-8	6.7	0.79	ug/Kg	69	134	30	69	134	30		
Heptachlor epoxide	1024-57-3	6.7	1	ug/Kg	70	135	30	70	135	30		
Methoxychlor	72-43-5	6.7	1.53	ug/Kg	57	135	30	57	135	30		
Toxaphene	8001-35-2	67	24.2	ug/Kg	49	114	30	49	114	30		
DCB Decachlorobiphenyl	2051-24-3			ug/Kg						30	28	148
Tetrachloro-m-xylene	877-09-8			ug/Kg						30	34	118

Appendix D -Measurement Perfomance Criteria
Table D-5 **Soil Samples - SW846, Polychlorinated Biphenyls (PCBs) by Gas Chromatography**

Matrix	**Soil Samples - SW846**
Method Description	**Polychlorinated Biphenyls (PCBs) by Gas Chromatography**
Method Code	**8082A**
Prep Method	**3546**
Units	**ug/Kg**

Analyte Description	CAS Number	RL	MDL	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
Aroclor 1016	12674-11-2	67	8.9	ug/Kg	65	133	30	65	133	30		
Aroclor 1221	11104-28-2	67	8.9	ug/Kg								
Aroclor 1232	11141-16-5	67	8.9	ug/Kg								
Aroclor 1242	53469-21-9	67	8.9	ug/Kg								
Aroclor 1248	12672-29-6	67	8.9	ug/Kg								
Aroclor 1254	11097-69-1	67	9.2	ug/Kg								
Aroclor 1260	11096-82-5	67	9.2	ug/Kg	71	150	30	71	150	30		
Aroclor 1268	11100-14-4	67	9.2	ug/Kg								
Aroclor-1262	37324-23-5	67	9.2	ug/Kg								
Polychlorinated biphenyls, Total	1336-36-3	67	9.2	ug/Kg								
DCB Decachlorobiphenyl	2051-24-3			ug/Kg							10	150
Tetrachloro-m-xylene	877-09-8			ug/Kg							58	145

Appendix D -Measurement Perfomance Criteria
Table D-6 **Soil Samples - SW846, Metals (ICP/MS)**

Matrix	**Soil Samples - SW846**
Method Description	**Metals (ICP/MS)**
Method Code	**6020B**
Prep Method	**3050B**
Units	**mg/Kg**

Analyte Description	CAS Number	RL	MDL	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
Aluminum	7429-90-5	20	2.6	mg/Kg	80	120	20	75	125	20		
Antimony	7440-36-0	1	0.146	mg/Kg	80	120	20	75	125	20		
Arsenic	7440-38-2	1	0.1	mg/Kg	80	120	20	75	125	20		
Barium	7440-39-3	2	0.145	mg/Kg	80	120	20	75	125	20		
Beryllium	7440-41-7	0.4	0.057	mg/Kg	80	120	20	75	125	20		
Cadmium	7440-43-9	1	0.113	mg/Kg	80	120	20	75	125	20		
Calcium	7440-70-2	100	17	mg/Kg	80	120	20	75	125	20		
Chromium	7440-47-3	2	0.174	mg/Kg	80	120	20	75	125	20		
Cobalt	7440-48-4	2	0.148	mg/Kg	80	120	20	75	125	20		
Copper	7440-50-8	2	0.22	mg/Kg	80	120	20	75	125	20		
Iron	7439-89-6	60	20.2	mg/Kg	80	120	20	75	125	20		
Lead	7439-92-1	0.6	0.2	mg/Kg	80	120	20	75	125	20		
Magnesium	7439-95-4	100	10.2	mg/Kg	80	120	20	75	125	20		
Manganese	7439-96-5	4	0.403	mg/Kg	80	120	20	75	125	20		
Nickel	7440-02-0	2	0.194	mg/Kg	80	120	20	75	125	20		
Potassium	7440-09-7	100	11.2	mg/Kg	80	120	20	75	125	20		
Selenium	7782-49-2	1.25	0.118	mg/Kg	80	120	20	75	125	20		
Silver	7440-22-4	1	0.089	mg/Kg	80	120	20	75	125	20		
Sodium	7440-23-5	100	15.6	mg/Kg	80	120	20	75	125	20		
Thallium	7440-28-0	0.4	0.041	mg/Kg	80	120	20	75	125	20		
Vanadium	7440-62-2	2	0.206	mg/Kg	80	120	20	75	125	20		
Zinc	7440-66-6	8	2.29	mg/Kg	80	120	20	75	125	20		

Appendix D -Measurement Perfomance Criteria
Table D-7 **Soil Samples - SW846, Mercury (CVAA)**

Matrix	**Soil Samples - SW846**
Method Description	**Mercury (CVAA)**
Method Code	**7471B**
Prep Method	**7471B_Prep**
Units	**mg/Kg**

Analyte Description	CAS Number	RL	MDL	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
Mercury	7439-97-6	0.017	0.004	mg/Kg	80	120	20	80	120	20		

Appendix D -Measurement Perfomance Criteria
Table D-8 **Soil Samples - SW846, Cyanide, Total andor Amenable**

Matrix	**Soil Samples - SW846**
Method Description	**Cyanide, Total andor Amenable**
Method Code	**9012B**
Prep Method	**9012B_Prep**
Units	**mg/Kg**

Analyte Description	CAS Number	RL	MDL	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
Cyanide, Total	57-12-5	0.24	0.123	mg/Kg	85	115		11	108	40		

Appendix D -Measurement Perfomance Criteria
Table D-9 **Soil Samples - SW846, Dioxins and Furans (HRGC/HRMS)**

Matrix	**Soil Samples - SW846**
Method Description	**Dioxins and Furans (HRGC/HRMS)**
Method Code	**8290A**
Prep Method	**8290_P_Sox**
Units	**pg/g**

Analyte Description	CAS Number	RL	MDL	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
1,2,3,4,6,7,8-HpCDD	35822-46-9	5	0.46	pg/g	86	134	20	86	134	20		
1,2,3,4,6,7,8-HpCDF	67562-39-4	5	0.38	pg/g	81	137	20	81	137	20		
1,2,3,4,7,8,9-HpCDF	55673-89-7	5	0.65	pg/g	79	139	20	79	139	20		
1,2,3,4,7,8-HxCDD	39227-28-6	5	0.71	pg/g	65	144	20	65	144	20		
1,2,3,4,7,8-HxCDF	70648-26-9	5	0.3	pg/g	72	140	20	72	140	20		
1,2,3,6,7,8-HxCDD	57653-85-7	5	0.58	pg/g	73	147	20	73	147	20		
1,2,3,6,7,8-HxCDF	57117-44-9	5	0.38	pg/g	63	152	20	63	152	20		
1,2,3,7,8,9-HxCDD	19408-74-3	5	0.58	pg/g	80	143	20	80	143	20		
1,2,3,7,8,9-HxCDF	72918-21-9	5	0.43	pg/g	72	152	20	72	152	20		
1,2,3,7,8-PeCDD	40321-76-4	5	0.3	pg/g	79	134	20	79	134	20		
1,2,3,7,8-PeCDF	57117-41-6	5	0.27	pg/g	81	134	20	81	134	20		
13C-1,2,3,4,6,7,8-HpCDD	109719-83-7	200		pg/g	40	135						
13C-1,2,3,4,6,7,8-HpCDF	109719-84-8	200		pg/g	40	135						
13C-1,2,3,4,7,8-HxCDF	114423-98-2	200		pg/g	40	135						
13C-1,2,3,6,7,8-HxCDD	109719-81-5	200		pg/g	40	135						
13C-1,2,3,7,8-PeCDD	109719-79-1	200		pg/g	40	135						
13C-1,2,3,7,8-PeCDF	109719-77-9	200		pg/g	40	135						
13C-2,3,7,8-TCDD	76523-40-5	200		pg/g	40	135						
13C-2,3,7,8-TCDF	89059-46-1	200		pg/g	40	135						
13C-OCDD	114423-97-1	400		pg/g	40	135						
2,3,4,6,7,8-HxCDF	60851-34-5	5	0.3	pg/g	72	151	20	72	151	20		
2,3,4,7,8-PeCDF	57117-31-4	5	0.29	pg/g	76	132	20	76	132	20		
2,3,7,8-TCDD	1746-01-6	1	0.15	pg/g	77	130	20	77	130	20		
2,3,7,8-TCDF	51207-31-9	1	0.11	pg/g	79	137	20	79	137	20		
OCDD	3268-87-9	10	1.5	pg/g	80	137	20	80	137	20		
OCDF	39001-02-0	10	1.2	pg/g	75	141	20	75	141	20		
Total HpCDD	37871-00-4	5	0.46	pg/g								
Total HpCDF	38998-75-3	5	0.65	pg/g								
Total HxCDD	34465-46-8	5	0.71	pg/g								
Total HxCDF	55684-94-1	5	0.3	pg/g								
Total PeCDD	36088-22-9	5	0.3	pg/g								
Total PeCDF	30402-15-4	5	0.29	pg/g								
Total TCDD	41903-57-5	1	0.15	pg/g								
Total TCDF	30402-14-3	1	0.11	pg/g								

Appendix D -Measurement Perfomance Criteria
Table D-10 **Soil Samples - SW846, Chlorinated Biphenyl Congeners (HRGC/HRMS)**

Matrix	**Soil Samples - SW846**
Method Description	**Chlorinated Biphenyl Congeners (HRGC/HRMS)**
Method Code	**1668A**
Prep Method	**HRMS_Sox_P**
Units	**pg/g**

Analyte Description	CAS Number	RL	MDL	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
PCB-1	2051-60-7	20	2.65	pg/g	50	150	50	50	150	50		
PCB-10	33146-45-1	20	2.22	pg/g								
PCB-100	39485-83-1	40	0.93	pg/g								
PCB-101	37680-73-2	60	1.92	pg/g								
PCB-102	68194-06-9	40	0.94	pg/g								
PCB-103	60145-21-3	20	0.59	pg/g								
PCB-104	56558-16-8	20	0.7	pg/g	50	150	50	50	150	50		
PCB-104L	234432-89-4	200		pg/g	30	140						
PCB-105	32598-14-4	4	1.09	pg/g	50	150	50	50	150	50		
PCB-105L	208263-62-1	200		pg/g	30	140						
PCB-106	70424-69-0	20	0.43	pg/g								
PCB-107	70424-68-9	40	0.52	pg/g								
PCB-108	70362-41-3	120	2.46	pg/g								
PCB-109	74472-35-8	20	0.38	pg/g								
PCB-11	2050-67-1	20	5.91	pg/g								
PCB-110	38380-03-9	40	2.25	pg/g								
PCB-111	39635-32-0	20	0.49	pg/g								
PCB-112	74472-36-9	20	0.58	pg/g								
PCB-113	68194-10-5	60	1.92	pg/g								
PCB-114	74472-37-0	2	0.55	pg/g	50	150	50	50	150	50		
PCB-114L	208263-63-2	200		pg/g	30	140						
PCB-115	74472-38-1	40	2.25	pg/g								
PCB-116	18259-05-7	60	1.28	pg/g								
PCB-117	68194-11-6	60	1.28	pg/g								
PCB-118	31508-00-6	4	1.5	pg/g	50	150	50	50	150	50		
PCB-118L	104130-40-7	200		pg/g	30	140						
PCB-119	56558-17-9	120	2.46	pg/g								
PCB-12	2974-92-7	40	1.69	pg/g								
PCB-120	68194-12-7	20	0.36	pg/g								
PCB-121	56558-18-0	20	0.37	pg/g								
PCB-122	76842-07-4	20	0.39	pg/g								
PCB-123	65510-44-3	2	0.7	pg/g	50	150	50	50	150	50		
PCB-123L	208263-64-3	200		pg/g	30	140						
PCB-124	70424-70-3	40	0.52	pg/g								
PCB-125	74472-39-2	120	2.46	pg/g								
PCB-126	57465-28-8	2	0.5	pg/g	50	150	50	50	150	50		
PCB-126L	208263-65-4	200		pg/g	30	140						
PCB-127	39635-33-1	20	0.3	pg/g								
PCB-128	38380-07-3	40	0.8	pg/g								
PCB-129	55215-18-4	60	3.58	pg/g								
PCB-13	2974-90-5	40	1.69	pg/g								
PCB-130	52663-66-8	20	0.46	pg/g								
PCB-131	61798-70-7	20	0.53	pg/g								
PCB-132	38380-05-1	20	2.58	pg/g								

PCB-133	35694-04-3	20	0.43	pg/g								
PCB-134	52704-70-8	40	0.98	pg/g								
PCB-135	52744-13-5	40	1.09	pg/g								
PCB-136	38411-22-2	20	1	pg/g								
PCB-137	35694-06-5	20	0.35	pg/g								
PCB-138	35065-28-2	60	3.58	pg/g								
PCB-139	56030-56-9	40	0.67	pg/g								
PCB-14	34883-41-5	20	1.05	pg/g								
PCB-140	59291-64-4	40	0.67	pg/g								
PCB-141	52712-04-6	20	2.19	pg/g								
PCB-142	41411-61-4	20	0.42	pg/g								
PCB-143	68194-15-0	40	0.98	pg/g								
PCB-144	68194-14-9	20	0.45	pg/g								
PCB-145	74472-40-5	20	0.36	pg/g								
PCB-146	51908-16-8	20	1.32	pg/g								
PCB-147	68194-13-8	40	2.02	pg/g								
PCB-148	74472-41-6	20	0.43	pg/g								
PCB-149	38380-04-0	40	2.02	pg/g								
PCB-15	2050-68-2	20	0.99	pg/g	50	150	50	50	150	50		
PCB-150	68194-08-1	20	0.49	pg/g								
PCB-151	52663-63-5	40	1.09	pg/g								
PCB-152	68194-09-2	20	0.44	pg/g								
PCB-153	35065-27-1	40	1.82	pg/g								
PCB-154	60145-22-4	20	0.49	pg/g								
PCB-155	33979-03-2	20	0.51	pg/g	50	150	50	50	150	50		
PCB-155L	234432-90-7	200		pg/g	30	140						
PCB-156	38380-08-4	4	1.32	pg/g	50	150	50	50	150	50		
PCB-156L	208263-68-7	200		pg/g	30	140						
PCB-156L/157L	STL01793	200		pg/g	30	140						
PCB-157	69782-90-7	4	1.32	pg/g	50	150	50	50	150	50		
PCB-157L	235416-30-5	200		pg/g	30	140						
PCB-158	74472-42-7	20	0.81	pg/g								
PCB-159	39635-35-3	20	0.23	pg/g								
PCB-15L	208263-67-6	200		pg/g	30	140						
PCB-16	38444-78-9	20	0.61	pg/g								
PCB-160	41411-62-5	20	0.53	pg/g								
PCB-161	74472-43-8	20	0.38	pg/g								
PCB-162	39635-34-2	20	0.74	pg/g								
PCB-163	74472-44-9	60	3.58	pg/g								
PCB-164	74472-45-0	20	0.6	pg/g								
PCB-165	74472-46-1	20	0.4	pg/g								
PCB-166	41411-63-6	40	0.8	pg/g								
PCB-167	52663-72-6	2	0.52	pg/g	50	150	50	50	150	50		
PCB-167L	208263-69-8	200		pg/g	30	140						
PCB-168	59291-65-5	40	1.82	pg/g								
PCB-169	32774-16-6	2	0.73	pg/g	50	150	50	50	150	50		
PCB-169L	208263-70-1	200		pg/g	30	140						
PCB-17	37680-66-3	20	0.8	pg/g								
PCB-170	35065-30-6	20	1.32	pg/g								
PCB-171	52663-71-5	40	0.74	pg/g								
PCB-172	52663-74-8	20	0.47	pg/g								
PCB-173	68194-16-1	40	0.74	pg/g								
PCB-174	38411-25-5	20	2.77	pg/g								
PCB-175	40186-70-7	20	0.37	pg/g								
PCB-176	52663-65-7	20	0.49	pg/g								

PCB-177	52663-70-4	20	1.51	pg/g								
PCB-178	52663-67-9	20	0.7	pg/g								
PCB-179	52663-64-6	20	1.69	pg/g								
PCB-18	37680-65-2	40	1.84	pg/g								
PCB-180	35065-29-3	40	1.67	pg/g								
PCB-181	74472-47-2	20	0.34	pg/g								
PCB-182	60145-23-5	20	0.29	pg/g								
PCB-183	52663-69-1	20	1.41	pg/g								
PCB-184	74472-48-3	20	0.39	pg/g								
PCB-185	52712-05-7	20	0.72	pg/g								
PCB-186	74472-49-4	20	0.45	pg/g								
PCB-187	52663-68-0	20	3.2	pg/g								
PCB-188	74487-85-7	20	0.4	pg/g	50	150	50	50	150	50		
PCB-188L	234432-91-8	200		pg/g	30	140						
PCB-189	39635-31-9	2	0.44	pg/g	50	150	50	50	150	50		
PCB-189L	208263-73-4	200		pg/g	30	140						
PCB-19	38444-73-4	20	0.65	pg/g	50	150	50	50	150	50		
PCB-190	41411-64-7	20	0.58	pg/g								
PCB-191	74472-50-7	20	0.36	pg/g								
PCB-192	74472-51-8	20	0.43	pg/g								
PCB-193	69782-91-8	40	1.67	pg/g								
PCB-194	35694-08-7	20	0.88	pg/g								
PCB-195	52663-78-2	20	0.62	pg/g								
PCB-196	42740-50-1	20	0.52	pg/g								
PCB-197	33091-17-7	20	0.4	pg/g								
PCB-198	68194-17-2	40	0.93	pg/g								
PCB-199	52663-75-9	40	0.93	pg/g								
PCB-19L	234432-87-2	200		pg/g	30	140						
PCB-1L	234432-85-0	200		pg/g	15	140						
PCB-2	2051-61-8	20	0.63	pg/g								
PCB-20	38444-84-7	60	18	pg/g								
PCB-200	52663-73-7	20	0.33	pg/g								
PCB-201	40186-71-8	20	0.42	pg/g								
PCB-202	2136-99-4	20	0.33	pg/g	50	150	50	50	150	50		
PCB-202L	105600-26-8	200		pg/g	30	140						
PCB-203	52663-76-0	20	0.59	pg/g								
PCB-204	74472-52-9	20	0.31	pg/g								
PCB-205	74472-53-0	20	0.39	pg/g	50	150	50	50	150	50		
PCB-205L	234446-64-1	200		pg/g	30	140						
PCB-206	40186-72-9	20	0.79	pg/g	50	150	50	50	150	50		
PCB-206L	208263-75-6	200		pg/g	30	140						
PCB-207	52663-79-3	20	0.48	pg/g								
PCB-208	52663-77-1	20	0.37	pg/g	50	150	50	50	150	50		
PCB-208L	234432-92-9	200		pg/g	30	140						
PCB-209	2051-24-3	20	2.5	pg/g	50	150	50	50	150	50		
PCB-209L	105600-27-9	200		pg/g	30	140						
PCB-21	55702-46-0	40	1.17	pg/g								
PCB-22	38444-85-8	20	0.66	pg/g								
PCB-23	55720-44-0	20	0.46	pg/g								
PCB-24	55702-45-9	20	0.54	pg/g								
PCB-25	55712-37-3	20	0.28	pg/g								
PCB-26	38444-81-4	40	0.87	pg/g								
PCB-27	38444-76-7	20	0.46	pg/g								
PCB-28	7012-37-5	60	18	pg/g								
PCB-29	15862-07-4	40	0.87	pg/g								

PCB-3	2051-62-9	20	0.9	pg/g	50	150	50	50	150	50		
PCB-30	35693-92-6	40	1.84	pg/g								
PCB-31	16606-02-3	20	1.43	pg/g								
PCB-32	38444-77-8	20	0.72	pg/g								
PCB-33	38444-86-9	40	1.17	pg/g								
PCB-34	37680-68-5	20	0.31	pg/g								
PCB-35	37680-69-6	20	0.33	pg/g								
PCB-36	38444-87-0	20	0.34	pg/g								
PCB-37	38444-90-5	20	0.46	pg/g	50	150	50	50	150	50		
PCB-37L	208263-79-0	200		pg/g	30	140						
PCB-38	53555-66-1	20	0.31	pg/g								
PCB-39	38444-88-1	20	0.47	pg/g								
PCB-3L	208263-77-8	200		pg/g	15	140						
PCB-4	13029-08-8	20	1.98	pg/g	50	150	50	50	150	50		
PCB-40	38444-93-8	40	0.78	pg/g								
PCB-41	52663-59-9	20	0.56	pg/g								
PCB-42	36559-22-5	20	0.58	pg/g								
PCB-43	70362-46-8	20	0.43	pg/g								
PCB-44	41464-39-5	60	6.27	pg/g								
PCB-45	70362-45-7	20	1.13	pg/g								
PCB-46	41464-47-5	20	0.52	pg/g								
PCB-47	2437-79-8	60	6.27	pg/g								
PCB-48	70362-47-9	20	0.54	pg/g								
PCB-49	41464-40-8	40	1.05	pg/g								
PCB-4L	234432-86-1	200		pg/g	30	140						
PCB-5	16605-91-7	20	1.85	pg/g								
PCB-50	62796-65-0	40	0.73	pg/g								
PCB-51	68194-04-7	20	0.94	pg/g								
PCB-52	35693-99-3	20	2.25	pg/g								
PCB-53	41464-41-9	40	0.73	pg/g								
PCB-54	15968-05-5	20	0.49	pg/g	50	150	50	50	150	50		
PCB-54L	234432-88-3	200		pg/g	30	140						
PCB-55	74338-24-2	20	0.31	pg/g								
PCB-56	41464-43-1	20	0.37	pg/g								
PCB-57	70424-67-8	20	0.42	pg/g								
PCB-58	41464-49-7	20	0.35	pg/g								
PCB-59	74472-33-6	60	1.36	pg/g								
PCB-6	25569-80-6	20	1.19	pg/g								
PCB-60	33025-41-1	20	0.54	pg/g								
PCB-61	33284-53-6	80	1.84	pg/g								
PCB-62	54230-22-7	60	1.36	pg/g								
PCB-63	74472-34-7	20	0.44	pg/g								
PCB-64	52663-58-8	20	0.69	pg/g								
PCB-65	33284-54-7	60	6.27	pg/g								
PCB-66	32598-10-0	20	1.8	pg/g								
PCB-67	73575-53-8	20	0.27	pg/g								
PCB-68	73575-52-7	20	0.9	pg/g								
PCB-69	60233-24-1	40	1.05	pg/g								
PCB-7	33284-50-3	20	1.81	pg/g								
PCB-70	32598-11-1	80	1.84	pg/g								
PCB-71	41464-46-4	40	0.78	pg/g								
PCB-72	41464-42-0	20	0.34	pg/g								
PCB-73	74338-23-1	20	0.38	pg/g								
PCB-74	32690-93-0	80	1.84	pg/g								
PCB-75	32598-12-2	60	1.36	pg/g								

PCB-76	70362-48-0	80	1.84	pg/g								
PCB-77	32598-13-3	2	0.81	pg/g	50	150	50	50	150	50		
PCB-77L	105600-23-5	200		pg/g	30	140						
PCB-78	70362-49-1	20	0.4	pg/g								
PCB-79	41464-48-6	20	0.42	pg/g								
PCB-8	34883-43-7	20	3.88	pg/g								
PCB-80	33284-52-5	20	0.36	pg/g								
PCB-81	70362-50-4	2	0.37	pg/g	50	150	50	50	150	50		
PCB-81L	208461-24-9	200		pg/g	30	140						
PCB-82	52663-62-4	20	0.52	pg/g								
PCB-83	60145-20-2	20	0.4	pg/g								
PCB-84	52663-60-2	20	0.6	pg/g								
PCB-85	65510-45-4	60	1.28	pg/g								
PCB-86	55312-69-1	120	2.46	pg/g								
PCB-87	38380-02-8	120	2.46	pg/g								
PCB-88	55215-17-3	40	0.91	pg/g								
PCB-89	73575-57-2	20	0.56	pg/g								
PCB-9	34883-39-1	20	1.58	pg/g								
PCB-90	68194-07-0	60	1.92	pg/g								
PCB-91	68194-05-8	40	0.91	pg/g								
PCB-92	52663-61-3	20	0.53	pg/g								
PCB-93	73575-56-1	40	0.93	pg/g								
PCB-94	73575-55-0	20	0.64	pg/g								
PCB-95	38379-99-6	20	2.16	pg/g								
PCB-96	73575-54-9	20	0.43	pg/g								
PCB-97	41464-51-1	120	2.46	pg/g								
PCB-98	60233-25-2	40	0.94	pg/g								
PCB-99	38380-01-7	20	1	pg/g								
PCB-111L	235416-29-2	200		pg/g	40	125					30	135
PCB-178L	232919-67-4	200		pg/g	40	125					30	135
PCB-28L	208263-76-7	200		pg/g	40	125					30	135

Appendix D -Measurement Perfomance Criteria
Table D-11 **Soil Samples - SW846, Percent Moisture**

Matrix	**Soil Samples - SW846**
Method Description	**Percent Moisture**
Method Code	**Moisture**
Prep Method	
Units	**%**

Analyte Description	CAS Number	RL	MDL	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
Percent Moisture	STL00177	1		%								
Percent Solids	STL00234	1		%								

Appendix D -Measurement Perfomance Criteria
Table D-12 **Water Samples - SW846, Volatile Organic Compounds by GC/MS**

Matrix	**Water Samples - SW846**
Method Description	**Volatile Organic Compounds by GC/MS**
Method Code	**8260C**
Prep Method	**5030C**
Units	**ug/L**

Analyte Description	CAS Number	RL	MDL	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
1,1,1-Trichloroethane	71-55-6	1	0.238	68	128	30	68	128	30		
1,1,2,2-Tetrachloroethane	79-34-5	1	0.367	63	139	30	63	139	30		
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	1	0.311	59	142	30	59	142	30		
1,1,2-Trichloroethane	79-00-5	1	0.433	74	125	30	74	125	30		
1,1-Dichloroethane	75-34-3	1	0.264	73	130	30	73	130	30		
1,1-Dichloroethene	75-35-4	1	0.264	68	133	30	68	133	30		
1,2,3-Trichlorobenzene	87-61-6	1	0.357	53	144	30	53	144	30		
1,2,4-Trichlorobenzene	120-82-1	1	0.365	64	132	30	64	132	30		
1,2-Dibromo-3-Chloropropane	96-12-8	1	0.376	41	143	30	41	143	30		
1,2-Dichlorobenzene	95-50-1	1	0.431	79	122	30	79	122	30		
1,2-Dichloroethane	107-06-2	1	0.43	75	121	30	75	121	30		
1,2-Dichloropropane	78-87-5	1	0.353	76	126	30	76	126	30		
1,3-Dichlorobenzene	541-73-1	1	0.342	80	121	30	80	121	30		
1,4-Dichlorobenzene	106-46-7	1	0.334	80	118	30	80	118	30		
1,4-Dioxane	123-91-1	50	28.2	70	142	30	70	142	30		
2-Butanone (MEK)	78-93-3	5	1.85	69	128	30	69	128	30		
2-Hexanone	591-78-6	5	1.14	74	127	30	74	127	30		
4-Methyl-2-pentanone (MIBK)	108-10-1	5	1.3	78	125	30	78	125	30		
Acetone	67-64-1	5	4.42	61	134	30	61	134	30		
Benzene	71-43-2	1	0.203	78	126	30	78	126	30		
Bromoform	75-25-2	1	0.536	38	144	30	38	144	30		
Bromomethane	74-83-9	1	0.55	10	150	30	10	150	30		
Carbon disulfide	75-15-0	1	0.821	64	138	30	64	138	30		
Carbon tetrachloride	56-23-5	1	0.208	56	131	30	56	131	30		
Chlorobenzene	108-90-7	1	0.377	80	119	30	80	119	30		
Chlorobromomethane	74-97-5	1	0.412	73	126	30	73	126	30		
Chlorodibromomethane	124-48-1	1	0.281	58	130	30	58	130	30		
Chloroethane	75-00-3	1	0.32	29	150	30	29	150	30		
Chloroform	67-66-3	1	0.326	78	125	30	78	125	30		
Chloromethane	74-87-3	1	0.402	38	150	30	38	150	30		
cis-1,2-Dichloroethene	156-59-2	1	0.219	78	121	30	78	121	30		
cis-1,3-Dichloropropene	10061-01-5	1	0.222	74	125	30	74	125	30		
Cyclohexane	110-82-7	1	0.321	67	133	30	67	133	30		
Dichlorobromomethane	75-27-4	1	0.343	72	121	30	72	121	30		
Dichlorodifluoromethane	75-71-8	1	0.311	31	150	30	31	150	30		
Ethylbenzene	100-41-4	1	0.298	78	120	30	78	120	30		
Ethylene Dibromide	106-93-4	1	0.498	69	126	30	69	126	30		
Isopropylbenzene	98-82-8	1	0.336	79	125	30	79	125	30		
Methyl acetate	79-20-9	5	0.785	70	127	30	70	127	30		
Methyl tert-butyl ether	1634-04-4	1	0.465	65	131	30	65	131	30		
Methylcyclohexane	108-87-2	1	0.258	60	139	30	60	139	30		
Methylene Chloride	75-09-2	1	0.315	74	127	30	74	127	30		
m-Xylene & p-Xylene	179601-23-1	1	0.296	78	123	30	78	123	30		
o-Xylene	95-47-6	1	0.361	78	122	30	78	122	30		
Styrene	100-42-5	1	0.415	75	127	30	75	127	30		

Tentatively Identified Compound	STL00231					30			30		
Tetrachloroethene	127-18-4	1	0.249	70	127	30	70	127	30		
Toluene	108-88-3	1	0.379	78	119	30	78	119	30		
trans-1,2-Dichloroethene	156-60-5	1	0.235	74	126	30	74	126	30		
trans-1,3-Dichloropropene	10061-02-6	1	0.485	66	127	30	66	127	30		
Trichloroethene	79-01-6	1	0.314	71	121	30	71	121	30		
Trichlorofluoromethane	75-69-4	1	0.32	61	140	30	61	140	30		
Vinyl chloride	75-01-4	1	0.171	61	144	30	61	144	30		
1,2-Dichloroethane-d4 (Surr)	17060-07-0					30			30	75	123
4-Bromofluorobenzene	460-00-4					30			30	76	120
Dibromofluoromethane (Surr)	1868-53-7					30			30	77	124
Toluene-d8 (Surr)	2037-26-5					30			30	80	120

Appendix D -Measurement Perfomance Criteria
Table D-13 **Water Samples - SW846, Volatile Organic Compounds (GC/MS)**

Matrix	**Water Samples - SW846**
Method Description	**Volatile Organic Compounds (GC/MS)**
Method Code	**8260C_SIM**
Prep Method	**5030C**
Units	**ug/L**

Analyte Description	CAS Number	RL	MDL	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
1,2-Dibromo-3-Chloropropane	96-12-8	0.02	0.0104	17	150	30	17	150	30		
1,4-Dioxane	123-91-1	0.4	0.333	64	138	30	64	138	30		
Ethylene Dibromide	106-93-4	0.02	0.0079	61	137	30	61	137	30		
1,2-Dichloroethane-d4 (Surr)	17060-07-0									10	150
4-Bromofluorobenzene	460-00-4									52	137

Appendix D -Measurement Perfomance Criteria
Table D-14 **Water Samples - SW846, Semivolatile Organic Compounds (GC/MS)**

Matrix	**Water Samples - SW846**
Method Description	**Semivolatile Organic Compounds (GC/MS)**
Method Code	**8270D**
Prep Method	**3510C_LVI**
Units	**ug/L**

Analyte Description	CAS Number	RL	MDL	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
1,1'-Biphenyl	92-52-4	10	1.19	59	102	30	59	102	30		
1,2,4,5-Tetrachlorobenzene	95-94-3	10	1.24	48	109	30	48	109	30		
2,2'-oxybis[1-chloropropane]	108-60-1	10	0.629	38	124	30	38	124	30		
2,3,4,6-Tetrachlorophenol	58-90-2	10	0.746	64	123	30	64	123	30		
2,4,5-Trichlorophenol	95-95-4	10	0.88	64	110	30	64	110	30		
2,4,6-Trichlorophenol	88-06-2	10	0.857	64	115	30	64	115	30		
2,4-Dichlorophenol	120-83-2	10	1.07	65	107	30	65	107	30		
2,4-Dimethylphenol	105-67-9	10	0.619	59	101	30	59	101	30		
2,4-Dinitrophenol	51-28-5	20	14.4	36	150	30	36	150	30		
2,4-Dinitrotoluene	121-14-2	2	0.997	63	122	30	63	122	30		
2,6-Dinitrotoluene	606-20-2	2	0.826	71	118	30	71	118	30		
2-Chloronaphthalene	91-58-7	10	1.18	57	102	30	57	102	30		
2-Chlorophenol	95-57-8	10	0.377	57	93	30	57	93	30		
2-Methylnaphthalene	91-57-6	10	1.1	57	103	30	57	103	30		
2-Methylphenol	95-48-7	10	0.671	45	86	30	45	86	30		
2-Nitroaniline	88-74-4	10	0.474	54	123	30	54	123	30		
2-Nitrophenol	88-75-5	10	0.747	60	126	30	60	126	30		
3,3'-Dichlorobenzidine	91-94-1	10	1.43	59	125	30	59	125	30		
3-Nitroaniline	99-09-2	10	1.94	57	110	30	57	110	30		
4,6-Dinitro-2-methylphenol	534-52-1	20	13.3	69	149	30	69	149	30		
4-Bromophenyl phenyl ether	101-55-3	10	0.745	65	115	30	65	115	30		
4-Chloro-3-methylphenol	59-50-7	10	0.575	60	107	30	60	107	30		
4-Chloroaniline	106-47-8	10	1.88	43	105	30	43	105	30		
4-Chlorophenyl phenyl ether	7005-72-3	10	1.28	60	113	30	60	113	30		
4-Methylphenol	106-44-5	10	0.651	37	86	30	37	86	30		
4-Nitroaniline	100-01-6	10	1.22	52	122	30	52	122	30		
4-Nitrophenol	100-02-7	20	3.98	17	61	30	17	61	30		
Acenaphthene	83-32-9	10	1.08	54	108	30	54	108	30		
Acenaphthylene	208-96-8	10	0.823	64	102	30	64	102	30		
Acetophenone	98-86-2	10	2.33	65	109	30	65	109	30		
Anthracene	120-12-7	10	0.634	69	110	30	69	110	30		
Atrazine	1912-24-9	2	1.35	10	150	30	10	150	30		
Benzaldehyde	100-52-7	10	2.1	47	134	30	47	134	30		
Benzo[a]anthracene	56-55-3	1	0.592	71	114	30	71	114	30		
Benzo[a]pyrene	50-32-8	1	0.405	67	106	30	67	106	30		
Benzo[b]fluoranthene	205-99-2	2	0.676	65	113	30	65	113	30		
Benzo[g,h,i]perylene	191-24-2	10	1.43	48	145	30	48	145	30		
Benzo[k]fluoranthene	207-08-9	1	0.674	66	116	30	66	116	30		
Bis(2-chloroethoxy)methane	111-91-1	10	0.589	64	114	30	64	114	30		
Bis(2-chloroethyl)ether	111-44-4	1	0.633	57	112	30	57	112	30		
Bis(2-ethylhexyl) phthalate	117-81-7	2	1.7	60	135	30	60	135	30		
Butyl benzyl phthalate	85-68-7	10	0.854	63	126	30	63	126	30		
Caprolactam	105-60-2	10	0.684	10	60	30	10	60	30		
Carbazole	86-74-8	10	0.679	68	113	30	68	113	30		

Chrysene	218-01-9	2	0.907	74	122	30	74	122	30		
Dibenz(a,h)anthracene	53-70-3	1	0.72	57	144	30	57	144	30		
Dibenzofuran	132-64-9	10	1.1	65	104	30	65	104	30		
Diethyl phthalate	84-66-2	10	0.976	65	105	30	65	105	30		
Dimethyl phthalate	131-11-3	10	0.766	68	105	30	68	105	30		
Di-n-butyl phthalate	84-74-2	10	0.84	66	113	30	66	113	30		
Di-n-octyl phthalate	117-84-0	10	4.75	40	133	30	40	133	30		
Fluoranthene	206-44-0	10	0.842	66	116	30	66	116	30		
Fluorene	86-73-7	10	0.912	64	108	30	64	108	30		
Hexachlorobenzene	118-74-1	1	0.396	59	129	30	59	129	30		
Hexachlorobutadiene	87-68-3	1	0.78	33	98	30	33	98	30		
Hexachlorocyclopentadiene	77-47-4	10	3.64	14	97	30	14	97	30		
Hexachloroethane	67-72-1	2	0.803	27	94	30	27	94	30		
Indeno[1,2,3-cd]pyrene	193-39-5	2	0.939	55	139	30	55	139	30		
Isophorone	78-59-1	10	0.798	64	113	30	64	113	30		
Naphthalene	91-20-3	2	1.13	56	99	30	56	99	30		
Nitrobenzene	98-95-3	1	0.567	67	109	30	67	109	30		
N-Nitrosodi-n-propylamine	621-64-7	1	0.43	60	111	30	60	111	30		
N-Nitrosodiphenylamine	86-30-6	10	0.891	67	110	30	67	110	30		
Pentachlorophenol	87-86-5	20	1.45	57	135	30	57	135	30		
Phenanthrene	85-01-8	10	0.58	69	108	30	69	108	30		
Phenol	108-95-2	10	0.292	20	53	30	20	53	30		
Pyrene	129-00-0	10	1.64	66	121	30	66	121	30		
Tentatively Identified Compound	STL00231					30			30		
2,4,6-Tribromophenol (Surr)	118-79-6					30			30	36	159
2-Fluorobiphenyl	321-60-8					30			30	42	127
2-Fluorophenol (Surr)	367-12-4					30			30	18	72
Nitrobenzene-d5 (Surr)	4165-60-0					30			30	46	137
Phenol-d5 (Surr)	4165-62-2					30			30	10	50
Terphenyl-d14 (Surr)	1718-51-0					30			30	39	150

Appendix A -Measurement Perfomance Criteria
Table D-15 **Water Samples - SW846, Semivolatile Organic Compounds (GC/MS SIM)**

Matrix	**Water Samples - SW846**
Method Description	**Semivolatile Organic Compounds (GC/MS SIM)**
Method Code	**8270D_SIM**
Prep Method	**3510C_LVI**
Units	**ug/L**

Analyte Description	CAS Number	RL	MDL	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
Acenaphthene	83-32-9	0.05	0.0142	29	131	30	29	131	30		
Acenaphthene-d10	15067-26-2										
Acenaphthylene	208-96-8	0.05	0.0146	10	150	30	10	150	30		
Anthracene	120-12-7	0.05	0.0092	37	149	30	37	149	30		
Benzo(a)anthracene	56-55-3	0.05	0.0156	44	150	30	44	150	30		
Benzo(a)pyrene	50-32-8	0.05	0.0216	38	139	30	38	139	30		
Benzo(b)fluoranthene	205-99-2	0.05	0.024	32	148	30	32	148	30		
Benzo(g,h,i)perylene	191-24-2	0.05	0.0351	20	150	30	20	150	30		
Benzo(k)fluoranthene	207-08-9	0.05	0.0278	44	150	30	44	150	30		
Chrysene	218-01-9	0.05	0.0299	52	150	30	52	150	30		
Chrysene-d12	1719-03-5										
Dibenzo(a,h)anthracene	53-70-3	0.05	0.02	23	134	30	23	134	30		
Fluoranthene	206-44-0	0.05	0.039	35	150	30	35	150	30		
Fluorene	86-73-7	0.05	0.0118	23	150	30	23	150	30		
Indeno(1,2,3-cd)pyrene	193-39-5	0.05	0.0362	21	126	30	21	126	30		
Naphthalene	91-20-3	0.2	0.124	33	124	30	33	124	30		
Naphthalene-d8	1146-65-2										
Pentachlorophenol	87-86-5	0.2	0.154	10	150	30	10	150	30		
Perylene-d12	1520-96-3										
Phenanthrene	85-01-8	0.05	0.0219	18	150	30	18	150	30		
Phenanthrene-d10	1517-22-2										
Pyrene	129-00-0	0.05	0.0314	45	150	30	45	150	30		

Appendix D -Measurement Perfomance Criteria
Table D-16 **Water Samples - SW846, Organochlorine Pesticides (GC)**

Matrix	**Water Samples - SW846**
Method Description	**Organochlorine Pesticides (GC)**
Method Code	**8081B**
Prep Method	**3510C_LVI**
Units	**ug/L**

Analyte Description	CAS Number	RL	MDL	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
1-Bromo-2-nitrobenzene	577-19-5										
4,4'-DDD	72-54-8	0.02	0.006	55	150	30	55	150	30		
4,4'-DDE	72-55-9	0.02	0.002	52	150	30	52	150	30		
4,4'-DDT	50-29-3	0.02	0.004	51	141	30	51	141	30		
Aldrin	309-00-2	0.02	0.003	46	144	30	46	144	30		
alpha-BHC	319-84-6	0.02	0.007	53	143	30	53	143	30		
beta-BHC	319-85-7	0.02	0.004	54	143	30	54	143	30		
Chlordane (technical)	12789-03-6	0.5	0.055	45	119	30	45	119	30		
delta-BHC	319-86-8	0.02	0.005	23	147	30	23	147	30		
Dieldrin	60-57-1	0.02	0.003	53	149	30	53	149	30		
Endosulfan I	959-98-8	0.02	0.002	54	149	30	54	149	30		
Endosulfan II	33213-65-9	0.02	0.004	60	144	30	60	144	30		
Endosulfan sulfate	1031-07-8	0.02	0.006	50	150	30	50	150	30		
Endrin	72-20-8	0.02	0.004	49	150	30	49	150	30		
Endrin aldehyde	7421-93-4	0.02	0.008	53	140	30	53	140	30		
Endrin ketone	53494-70-5	0.02	0.008	53	150	30	53	150	30		
gamma-BHC (Lindane)	58-89-9	0.02	0.012	53	140	30	53	140	30		
Heptachlor	76-44-8	0.02	0.003	49	140	30	49	140	30		
Heptachlor epoxide	1024-57-3	0.02	0.005	55	146	30	55	146	30		
Methoxychlor	72-43-5	0.02	0.004	52	145	30	52	145	30		
Toxaphene	8001-35-2	0.5	0.11	41	140	30	41	140	30		
DCB Decachlorobiphenyl	2051-24-3					30			30	10	132
Tetrachloro-m-xylene	877-09-8					30			30	10	150

Appendix D -Measurement Perfomance Criteria

Table D-17 **Water Samples - SW846, Polychlorinated Biphenyls (PCBs) by Gas Chromatography**

Matrix	**Water Samples - SW846**
Method Description	**Polychlorinated Biphenyls (PCBs) by Gas Chroma**
Method Code	**8082A**
Prep Method	**3510C_LVI**
Units	**ug/L**

Analyte Description	CAS Number	RL	MDL	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
Aroclor 1016	12674-11-2	0.4	0.119	66	141	30	66	141	30		
Aroclor 1221	11104-28-2	0.4	0.119								
Aroclor 1232	11141-16-5	0.4	0.119								
Aroclor 1242	53469-21-9	0.4	0.119								
Aroclor 1248	12672-29-6	0.4	0.119								
Aroclor 1254	11097-69-1	0.4	0.107								
Aroclor 1260	11096-82-5	0.4	0.107	75	150	30	75	150	30		
Aroclor 1268	11100-14-4	0.4	0.107								
Aroclor-1262	37324-23-5	0.4	0.107								
Polychlorinated biphenyls, Total	1336-36-3	0.4	0.119								
DCB Decachlorobiphenyl	2051-24-3									10	150
Tetrachloro-m-xylene	877-09-8									48	125

Appendix D -Measurement Perfomance Criteria
Table D-18 Water Samples - SW846, Metals (ICP/MS)

Matrix	**Water Samples - SW846**
Method Description	**Metals (ICP/MS)**
Method Code	**6020B**
Prep Method	**3010A**
Units	**ug/L**

Analyte Description	CAS Number	RL	MDL	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
Aluminum	7429-90-5	40	8.01	80	120	20	75	125	20		
Antimony	7440-36-0	2	0.757	80	120	20	75	125	20		
Arsenic	7440-38-2	2	0.887	80	120	20	75	125	20		
Barium	7440-39-3	4	0.913	80	120	20	75	125	20		
Beryllium	7440-41-7	0.8	0.098	80	120	20	75	125	20		
Cadmium	7440-43-9	2	0.156	80	120	20	75	125	20		
Calcium	7440-70-2	200	22.7	80	120	20	75	125	20		
Chromium	7440-47-3	4	0.688	80	120	20	75	125	20		
Cobalt	7440-48-4	4	0.263	80	120	20	75	125	20		
Copper	7440-50-8	4	2.45	80	120	20	75	125	20		
Iron	7439-89-6	120	8.52	80	120	20	75	125	20		
Lead	7439-92-1	1.2	0.11	80	120	20	75	125	20		
Magnesium	7439-95-4	200	15.4	80	120	20	75	125	20		
Manganese	7439-96-5	8	1.11	80	120	20	75	125	20		
Nickel	7440-02-0	4	0.447	80	120	20	75	125	20		
Potassium	7440-09-7	200	112	80	120	20	75	125	20		
Selenium	7782-49-2	2.5	0.456	80	120	20	75	125	20		
Silver	7440-22-4	2	0.194	80	120	20	75	125	20		
Sodium	7440-23-5	200	58.2	80	120	20	75	125	20		
Thallium	7440-28-0	0.8	0.168	80	120	20	75	125	20		
Vanadium	7440-62-2	4	0.369	80	120	20	75	125	20		
Zinc	7440-66-6	16	5.14	80	120	20	75	125	20		

Appendix D -Measurement Perfomance Criteria
Table D-19 **Water Samples - SW846, Mercury (CVAA)**

Matrix	**Water Samples - SW846**
Method Description	**Mercury (CVAA)**
Method Code	**7470A**
Prep Method	**7470A_Prep**
Units	**ug/L**

Analyte Description	CAS Number	RL	MDL	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
Mercury	7439-97-6	0.2	0.091	80	120	20	75	125	20		

Appendix D -Measurement Perfomance Criteria
Table D-20 **Water Samples - SW846, Cyanide, Total andor Amenable**

Matrix	**Water Samples - SW846**
Method Description	**Cyanide, Total andor Amenable**
Method Code	**9012B**
Prep Method	**9012B_Prep**
Units	**mg/L**

Analyte Description	CAS Number	RL	MDL	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
Cyanide, Total	57-12-5	0.01	0.004	85	115		90	110	35		

Appendix D -Measurement Perfomance Criteria
Table D-21 **Water Samples - SW846, Dioxins and Furans (HRGC/HRMS)**

Matrix	**Water Samples - SW846**
Method Description	**Dioxins and Furans (HRGC/HRMS)**
Method Code	**8290A**
Prep Method	**8290_P_Sep**
Units	**pg/L**

Analyte Description	CAS Number	RL	MDL	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
1,2,3,4,6,7,8-HpCDD	35822-46-9	50	9.4	78	139	20	78	139	20		
1,2,3,4,6,7,8-HpCDF	67562-39-4	50	2.5	79	133	20	79	133	20		
1,2,3,4,7,8,9-HpCDF	55673-89-7	50	3.8	83	130	20	83	130	20		
1,2,3,4,7,8-HxCDD	39227-28-6	50	10	56	146	20	56	146	20		
1,2,3,4,7,8-HxCDF	70648-26-9	50	2.1	75	131	20	75	131	20		
1,2,3,6,7,8-HxCDD	57653-85-7	50	5.7	73	144	20	73	144	20		
1,2,3,6,7,8-HxCDF	57117-44-9	50	5.1	76	133	20	76	133	20		
1,2,3,7,8,9-HxCDD	19408-74-3	50	5.2	71	151	20	71	151	20		
1,2,3,7,8,9-HxCDF	72918-21-9	50	2.3	77	142	20	77	142	20		
1,2,3,7,8-PeCDD	40321-76-4	50	2.5	71	140	20	71	140	20		
1,2,3,7,8-PeCDF	57117-41-6	50	2.2	76	135	20	76	135	20		
13C-1,2,3,4,6,7,8-HpCDD	109719-83-7	2000		40	135						
13C-1,2,3,4,6,7,8-HpCDF	109719-84-8	2000		40	135						
13C-1,2,3,4,7,8-HxCDF	114423-98-2	2000		40	135						
13C-1,2,3,6,7,8-HxCDD	109719-81-5	2000		40	135						
13C-1,2,3,7,8-PeCDD	109719-79-1	2000		40	135						
13C-1,2,3,7,8-PeCDF	109719-77-9	2000		40	135						
13C-2,3,7,8-TCDD	76523-40-5	2000		40	135						
13C-2,3,7,8-TCDF	89059-46-1	2000		40	135						
13C-OCDD	114423-97-1	4000		40	135						
2,3,4,6,7,8-HxCDF	60851-34-5	50	2.2	80	137	20	80	137	20		
2,3,4,7,8-PeCDF	57117-31-4	50	4.3	74	137	20	74	137	20		
2,3,7,8-TCDD	1746-01-6	10	1.2	64	142	20	64	142	20		
2,3,7,8-TCDF	51207-31-9	10	2	71	142	20	71	142	20		
OCDD	3268-87-9	100	46	80	132	20	80	132	20		
OCDF	39001-02-0	100	8.6	72	140	20	72	140	20		
Total HpCDD	37871-00-4	50	9.4								
Total HpCDF	38998-75-3	50	3.8								
Total HxCDD	34465-46-8	50	10								
Total HxCDF	55684-94-1	50	5.1								
Total PeCDD	36088-22-9	50	2.5								
Total PeCDF	30402-15-4	50	4.3								
Total TCDD	41903-57-5	10	1.2								
Total TCDF	30402-14-3	10	2								

Appendix D -Measurement Perfomance Criteria
Table D-22 **Water Samples - SW846, Chlorinated Biphenyl Congeners (HRGC/HRMS)**

Matrix	**Water Samples - SW846**
Method Description	**Chlorinated Biphenyl Congeners (HRGC/HRMS)**
Method Code	**1668A**
Prep Method	**HRMS_Sep_P**
Units	**pg/L**

Analyte Description	CAS Number	RL	MDL	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
PCB-1	2051-60-7	200	7.54	50	150	50	50	150	50		
PCB-10	33146-45-1	200	15.2								
PCB-100	39485-83-1	400	16.5								
PCB-101	37680-73-2	600	24.7								
PCB-102	68194-06-9	400	13.8								
PCB-103	60145-21-3	200	8.69								
PCB-104	56558-16-8	200	13.1	50	150	50	50	150	50		
PCB-104L	234432-89-4	200		30	140						
PCB-105	32598-14-4	20	9.23	50	150	50	50	150	50		
PCB-105L	208263-62-1	200		30	140						
PCB-106	70424-69-0	200	8.25								
PCB-107	70424-68-9	400	13.7								
PCB-108	70362-41-3	1200	43.6								
PCB-109	74472-35-8	200	6.89								
PCB-11	2050-67-1	200	19.1								
PCB-110	38380-03-9	400	13.8								
PCB-111	39635-32-0	200	8.69								
PCB-112	74472-36-9	200	7.54								
PCB-113	68194-10-5	200	24.7								
PCB-114	74472-37-0	20	7.67	50	150	50	50	150	50		
PCB-114L	208263-63-2	200		30	140						
PCB-115	74472-38-1	400	13.8								
PCB-116	18259-05-7	600	22.8								
PCB-117	68194-11-6	600	22.8								
PCB-118	31508-00-6	20	8.89	50	150	50	50	150	50		
PCB-118L	104130-40-7	200		30	140						
PCB-119	56558-17-9	1200	43.6								
PCB-12	2974-92-7	400	30.9								
PCB-120	68194-12-7	200	6.68								
PCB-121	56558-18-0	200	7.47								
PCB-122	76842-07-4	200	8.11								
PCB-123	65510-44-3	20	9.83	50	150	50	50	150	50		
PCB-123L	208263-64-3	200		30	140						
PCB-124	70424-70-3	400	13.7								
PCB-125	74472-39-2	1200	43.6								
PCB-126	57465-28-8	20	7.22	50	150	50	50	150	50		
PCB-126L	208263-65-4	200		30	140						
PCB-127	39635-33-1	200	6.2								
PCB-128	38380-07-3	400	16.1								
PCB-129	55215-18-4	600	11.8								
PCB-13	2974-90-5	400	30.9								
PCB-130	52663-66-8	200	6.65								
PCB-131	61798-70-7	200	6.98								
PCB-132	38380-05-1	200	5.61								

PCB-133	35694-04-3	200	4.54								
PCB-134	52704-70-8	400	14.8								
PCB-135	52744-13-5	400	9.77								
PCB-136	38411-22-2	200	5.58								
PCB-137	35694-06-5	200	6.43								
PCB-138	35065-28-2	600	11.8								
PCB-139	56030-56-9	400	9.34								
PCB-14	34883-41-5	200	8.75								
PCB-140	59291-64-4	400	9.34								
PCB-141	52712-04-6	200	7.12								
PCB-142	41411-61-4	200	6.1								
PCB-143	68194-15-0	400	14.8								
PCB-144	68194-14-9	200	5.34								
PCB-145	74472-40-5	200	4.51								
PCB-146	51908-16-8	200	4.73								
PCB-147	68194-13-8	400	8.67								
PCB-148	74472-41-6	200	6.2								
PCB-149	38380-04-0	400	8.67								
PCB-15	2050-68-2	200	6.27	50	150	50	50	150	50		
PCB-150	68194-08-1	200	6.48								
PCB-151	52663-63-5	400	9.77								
PCB-152	68194-09-2	200	4.51								
PCB-153	35065-27-1	400	9.47								
PCB-154	60145-22-4	200	5.42								
PCB-155	33979-03-2	200	11.8	50	150	50	50	150	50		
PCB-155L	234432-90-7	200		30	140						
PCB-156	38380-08-4	40	13.3	50	150	50	50	150	50		
PCB-156L	208263-68-7	200		30	140						
PCB-156L/157L	STL01793	200		30	140						
PCB-157	69782-90-7	40	13.3	50	150	50	50	150	50		
PCB-157L	235416-30-5	200		30	140						
PCB-158	74472-42-7	200	5.32								
PCB-159	39635-35-3	200	5.51								
PCB-15L	208263-67-6	200		30	140						
PCB-16	38444-78-9	200	7.95								
PCB-160	41411-62-5	200	7.64								
PCB-161	74472-43-8	200	7.33								
PCB-162	39635-34-2	200	9.02								
PCB-163	74472-44-9	600	11.8								
PCB-164	74472-45-0	200	5.54								
PCB-165	74472-46-1	200	5.06								
PCB-166	41411-63-6	200	16.1								
PCB-167	52663-72-6	20	7.66	50	150	50	50	150	50		
PCB-167L	208263-69-8	200		30	140						
PCB-168	59291-65-5	400	9.47								
PCB-169	32774-16-6	20	7.12	50	150	50	50	150	50		
PCB-169L	208263-70-1	200		30	140						
PCB-17	37680-66-3	200	6.82								
PCB-170	35065-30-6	200	7.78								
PCB-171	52663-71-5	400	13.9								
PCB-172	52663-74-8	200	5.78								
PCB-173	68194-16-1	400	13.9								
PCB-174	38411-25-5	200	7.31								
PCB-175	40186-70-7	200	5.45								
PCB-176	52663-65-7	200	6.96								

PCB-177	52663-70-4	200	6.54								
PCB-178	52663-67-9	200	6.91								
PCB-179	52663-64-6	200	4.99								
PCB-18	37680-65-2	400	14.9								
PCB-180	35065-29-3	400	28.4								
PCB-181	74472-47-2	200	5.42								
PCB-182	60145-23-5	200	6.36								
PCB-183	52663-69-1	200	5.11								
PCB-184	74472-48-3	200	6								
PCB-185	52712-05-7	200	12.1								
PCB-186	74472-49-4	200	5.31								
PCB-187	52663-68-0	200	5.28								
PCB-188	74487-85-7	200	9.92	50	150	50	50	150	50		
PCB-188L	234432-91-8	200		30	140						
PCB-189	39635-31-9	20	6.94	50	150	50	50	150	50		
PCB-189L	208263-73-4	200		30	140						
PCB-19	38444-73-4	200	9.37	50	150	50	50	150	50		
PCB-190	41411-64-7	200	7.29								
PCB-191	74472-50-7	200	7.93								
PCB-192	74472-51-8	200	4.65								
PCB-193	69782-91-8	400	28.4								
PCB-194	35694-08-7	200	8.76								
PCB-195	52663-78-2	200	7.62								
PCB-196	42740-50-1	200	5.08								
PCB-197	33091-17-7	200	5.73								
PCB-198	68194-17-2	400	7.81								
PCB-199	52663-75-9	400	7.81								
PCB-19L	234432-87-2	200		30	140						
PCB-1L	234432-85-0	200		15	140						
PCB-2	2051-61-8	200	4.32								
PCB-20	38444-84-7	400	47.5								
PCB-200	52663-73-7	200	6.33								
PCB-201	40186-71-8	200	6.4								
PCB-202	2136-99-4	200	5.51	50	150	50	50	150	50		
PCB-202L	105600-26-8	200		30	140						
PCB-203	52663-76-0	200	17.5								
PCB-204	74472-52-9	200	5.02								
PCB-205	74472-53-0	200	6.31	50	150	50	50	150	50		
PCB-205L	234446-64-1	200		30	140						
PCB-206	40186-72-9	200	8.32	50	150	50	50	150	50		
PCB-206L	208263-75-6	200		30	140						
PCB-207	52663-79-3	200	7.02								
PCB-208	52663-77-1	200	6.1	50	150	50	50	150	50		
PCB-208L	234432-92-9	200		30	140						
PCB-209	2051-24-3	200	11.4	50	150	50	50	150	50		
PCB-209L	105600-27-9	200		30	140						
PCB-21	55702-46-0	400	14.5								
PCB-22	38444-85-8	200	6.49								
PCB-23	55720-44-0	200	8.04								
PCB-24	55702-45-9	200	6.22								
PCB-25	55712-37-3	200	6.21								
PCB-26	38444-81-4	400	16.5								
PCB-27	38444-76-7	200	5.67								
PCB-28	7012-37-5	400	47.5								
PCB-29	15862-07-4	400	16.5								

PCB-3	2051-62-9	200	4.95	50	150	50	50	150	50		
PCB-30	35693-92-6	400	14.9								
PCB-31	16606-02-3	200	9.11								
PCB-32	38444-77-8	200	4.85								
PCB-33	38444-86-9	400	14.5								
PCB-34	37680-68-5	200	7.56								
PCB-35	37680-69-6	200	8.06								
PCB-36	38444-87-0	200	6.32								
PCB-37	38444-90-5	200	5.73	50	150	50	50	150	50		
PCB-37L	208263-79-0	200		30	140						
PCB-38	53555-66-1	200	7.22								
PCB-39	38444-88-1	200	10.1								
PCB-3L	208263-77-8	200		15	140						
PCB-4	13029-08-8	200	17.6	50	150	50	50	150	50		
PCB-40	38444-93-8	400	11.7								
PCB-41	52663-59-9	200	8.35								
PCB-42	36559-22-5	200	8.19								
PCB-43	70362-46-8	200	5.37								
PCB-44	41464-39-5	600	139								
PCB-45	70362-45-7	200	7.7								
PCB-46	41464-47-5	200	7.02								
PCB-47	2437-79-8	600	139								
PCB-48	70362-47-9	200	7.03								
PCB-49	41464-40-8	400	13.4								
PCB-4L	234432-86-1	200		30	140						
PCB-5	16605-91-7	200	6.77								
PCB-50	62796-65-0	400	10.9								
PCB-51	68194-04-7	200	7.82								
PCB-52	35693-99-3	200	10.8								
PCB-53	41464-41-9	400	10.9								
PCB-54	15968-05-5	200	8.34	50	150	50	50	150	50		
PCB-54L	234432-88-3	200		30	140						
PCB-55	74338-24-2	200	7.58								
PCB-56	41464-43-1	200	6.61								
PCB-57	70424-67-8	200	7.07								
PCB-58	41464-49-7	200	5.31								
PCB-59	74472-33-6	600	21.8								
PCB-6	25569-80-6	200	5.39								
PCB-60	33025-41-1	200	9.5								
PCB-61	33284-53-6	800	32.9								
PCB-62	54230-22-7	600	21.8								
PCB-63	74472-34-7	200	10								
PCB-64	52663-58-8	200	9.41								
PCB-65	33284-54-7	600	139								
PCB-66	32598-10-0	200	10.4								
PCB-67	73575-53-8	200	7.3								
PCB-68	73575-52-7	200	9.18								
PCB-69	60233-24-1	400	13.4								
PCB-7	33284-50-3	200	9.01								
PCB-70	32598-11-1	800	32.9								
PCB-71	41464-46-4	400	11.7								
PCB-72	41464-42-0	200	6.09								
PCB-73	74338-23-1	200	6.75								
PCB-74	32690-93-0	800	32.9								
PCB-75	32598-12-2	600	21.8								

PCB-76	70362-48-0	800	32.9								
PCB-77	32598-13-3	20	6.06	50	150	50	50	150	50		
PCB-77L	105600-23-5	200		30	140						
PCB-78	70362-49-1	200	6.98								
PCB-79	41464-48-6	200	8.14								
PCB-8	34883-43-7	200	8.24								
PCB-80	33284-52-5	200	8.14								
PCB-81	70362-50-4	20	7.12	50	150	50	50	150	50		
PCB-81L	208461-24-9	200		30	140						
PCB-82	52663-62-4	200	7.47								
PCB-83	60145-20-2	200	6.52								
PCB-84	52663-60-2	200	9.78								
PCB-85	65510-45-4	600	22.8								
PCB-86	55312-69-1	1200	43.6								
PCB-87	38380-02-8	1200	43.6								
PCB-88	55215-17-3	400	19.6								
PCB-89	73575-57-2	200	8.31								
PCB-9	34883-39-1	200	12.2								
PCB-90	68194-07-0	600	24.7								
PCB-91	68194-05-8	400	19.6								
PCB-92	52663-61-3	200	9.84								
PCB-93	73575-56-1	400	16.5								
PCB-94	73575-55-0	200	10.4								
PCB-95	38379-99-6	200	9.52								
PCB-96	73575-54-9	200	5.39								
PCB-97	41464-51-1	1200	43.6								
PCB-98	60233-25-2	400	13.8								
PCB-99	38380-01-7	200	7.99								
PCB-111L	235416-29-2	200		40	125					30	135
PCB-178L	232919-67-4	200		40	125					30	135
PCB-28L	208263-76-7	200		40	125					30	135

Appendix E – Laboratory Standard Operating Procedures
Eurofins/TestAmerica

Title: Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) by SW846 Methods 8260C and 8260D

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Approvals (Signature/Date):

Sylvanus Klusey Organics Operations Manager	7/16/20 Date	Dan Helfrich Health & Safety Manager / Coordinator	7/16/20 Date
Carl Armbruster Quality Assurance Manager	7/16/20 Date	Mark Acierno Laboratory Director	7/16/20 Date

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 USEPA SW846 Methods 8260C and 8260D are used for the determination of volatile organic compounds in a variety of aqueous and solid matrices by purge and trap gas chromatography (GC)/mass spectrometery (MS). The methods are applicable to the compounds listed in Table 1 (below). Actual target compound lists are determined through regulatory or project specifications. Method performance criteria for each target analyte will be determined prior to sample analysis.

1.1.2 This SOP also describes the optional procedure for analyses of compounds using 8260C/8260D Selected Ion Monitoring (SIM). SIM analyses is specific to target compounds: 1,2-dibromoethane, 1,2-dibromo-3-chloropropane, 1,2,3-Trichloropropane and 1,4-Dioxane. Benzene and Chloroform if needed.

Table 1: Method Analytes

Compound	CAS #	Compound	CAS #
1,1,1,2-Tetrachloroethane	630-20-6	cis-1,2-Dichloroethene	156-59-2
1,1,1-Trichloroethane	71-55-6	cis-1,3-Dichloropropene	10061-01-5
1,1,1-Trifluoro-2,2-dichloroethane	306-83-2	Cyclohexane	110-82-7
1,1,2,2-Tetrachloroethane	79-34-5	Cyclopentene	142-29-0
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	Dibromomethane	74-95-3
1,1,2-Trichloroethane	79-00-5	Dichlorobromomethane	75-27-4
1,1-Dichloroethane	75-34-3	Dichlorodifluoromethane	75-71-8
1,1-Dichloroethene	75-35-4	Dichlorofluoromethane	75-43-4
1,1-Dichloropropene	563-58-6	Dimethylnaphthalene (total)	28804-88-8
1,1-Difluoroethane	75-37-6	Epichlorohydrin	106-89-8
1,2,3-Trichlorobenzene	87-61-6	Ethanol	64-17-5
1,2,3-Trichloropropane **(1)**	96-18-4	Ethyl acetate	141-78-6
1,2,3-Trimethylbenzene	526-73-8	Ethyl acrylate	140-88-5
1,2,4,5-Tetramethylbenzene	95-93-2	Ethyl ether	60-29-7
1,2,4-Trichlorobenzene	120-82-1	Ethyl methacrylate	97-63-2
1,2,4-Trimethylbenzene	95-63-6	Ethylbenzene	100-41-4
1,2-Dibromo-3-Chloropropane **(1)**	96-12-8	Ethylene Dibromide **(1)**	106-93-4
1,2-Dichloro-1,1,2,2-tetrafluoroethane	76-14-2	Hexachlorobutadiene	87-68-3
1,2-Dichloro-1,1,2-trifluoroethane	354-23-4	Hexane	110-54-3
1,2-Dichlorobenzene	95-50-1	Indan	496-11-7
1,2-Dichloroethane	107-06-2	Iodomethane	74-88-4
1,2-Dichloroethene, Total	540-59-0	Isobutyl alcohol	78-83-1
1,2-Dichloropropane	78-87-5	Isopropyl acetate	108-21-4
1,3,5-Trichlorobenzene	108-70-3	Isopropyl alcohol	67-63-0
1,3,5-Trimethylbenzene	108-67-8	Isopropyl ether	108-20-3
1,3-Dichlorobenzene	541-73-1	Isopropylbenzene	98-82-8
1,3-Dichloropropane	142-28-9	Methacrylonitrile	126-98-7
1,3-Dichloropropene, Total	542-75-6	Methyl acetate	79-20-9

Compound	CAS #	Compound	CAS #
1,4-Dichlorobenzene	106-46-7	Methyl acrylate	96-33-3
1,4-Dioxane **(1)**	123-91-1	Methyl methacrylate	80-62-6
1-Chloropropane	540-54-5	Methyl tert-butyl ether	1634-04-4
2,2,4-Trimethylpentane	540-84-1	Methylcyclohexane	108-87-2
2,2-Dichloropropane	594-20-7	Methylene Chloride	75-09-2
2,4,4-Trimethyl-1-pentene	107-39-1	Methylnaphthalene (total)	1321-94-4
2-Butanone (MEK)	78-93-3	Monochloropentafluoroethane	76-15-3
2-Chloro-1,3-butadiene	126-99-8	m-Xylene & p-Xylene	179601-23-1
2-Chloroethyl vinyl ether	110-75-8	Naphthalene	91-20-3
2-Chloropropane	75-29-6	n-Butanol	71-36-3
2-Chlorotoluene	95-49-8	n-Butyl acetate	123-86-4
2-Hexanone	591-78-6	n-Butyl acrylate	141-32-2
2-Methyl-1,3-butadiene	78-79-5	n-Butylbenzene	104-51-8
2-Methyl-2-propanol	75-65-0	n-Heptane	142-82-5
2-Nitropropane	79-46-9	n-Propyl acetate	109-60-4
2-Octanol	123-96-6	N-Propylbenzene	103-65-1
2-Octanone	111-13-7	o-Xylene	95-47-6
4-Chlorotoluene	106-43-4	p-Diethylbenzene	105-05-5
4-Ethyltoluene	622-96-8	Pentane	109-66-0
4-Isopropyltoluene	99-87-6	Propene	115-07-1
4-Methyl-2-pentanone (MIBK)	108-10-1	Propionitrile	107-12-0
Acetaldehyde	75-07-0	sec-Butylbenzene	135-98-8
Acetone	67-64-1	Styrene	100-42-5
Acetonitrile	75-05-8	Tert-amyl methyl ether	994-05-8
Acrolein	107-02-8	Tert-butyl ethyl ether	637-92-3
Acrylonitrile	107-13-1	tert-Butylbenzene	98-06-6
Allyl chloride	107-05-1	Tetrachloroethene	127-18-4
Amyl acetate (mixed isomers)	628-63-7	Tetrahydrofuran	109-99-9
Benzene **(1)**	71-43-2	Toluene	108-88-3
Benzyl chloride	100-44-7	Total BTEX	STL00431
Bromobenzene	108-86-1	trans-1,2-Dichloroethene	156-60-5
Bromoform	75-25-2	trans-1,3-Dichloropropene	10061-02-6
Bromomethane	74-83-9	trans-1,4-Dichloro-2-butene	110-57-6
Butadiene	106-99-0	Trichloroethene	79-01-6
Butyl Methacrylate	97-88-1	Trichlorofluoromethane	75-69-4
Camphene	79-92-5	Vinyl acetate	108-05-4
Camphor	76-22-2	Vinyl chloride	75-01-4
Carbon disulfide	75-15-0	Xylenes, Total	1330-20-7
Carbon tetrachloride	56-23-5	1,4-Dichlorobenzene-d4 **(ISTD)**	3855-82-1
Chlorobenzene	108-90-7	1,4-Dioxane-d8 **(ISTD)**	17647-74-4
Chlorobromomethane	74-97-5	2-Butanone-d5 **(ISTD)**	24313-50-6
Chlorodibromomethane	124-48-1	Chlorobenzene-d5 **(ISTD)**	3114-55-4
Chlorodifluoromethane	75-45-6	Fluorobenzene **(ISTD)**	462-06-6
Chloroethane	75-00-3	TBA-d9 **(ISTD)**	25725-11-5
Chloroform **(1)**	67-66-3	1,2-Dichloroethane-d4 **(Surrogate)**	17060-07-0
Chloromethane	74-87-3	4-Bromofluorobenzene **(Surrogate)**	460-00-4

Compound	CAS #	Compound	CAS #
Chlorotrifluoroethene	79-38-9	Dibromofluoromethane **(Surrogate)**	1868-53-7
Chlorotrifluoromethane	75-72-9	Toluene-d8 **(Surrogate)**	2037-26-5

(1) Compound can be analyzed by full scan or Selected Ion Monitoring (SIM).

1.1.3 Methods 8260C and 8260D can be used to quantitate most volatile organic compounds that have boiling points below 200°C, and that are insoluble or slightly soluble in water. Water-soluble compounds can be included in this method, but quantitation limits will be higher due to poor purging efficiency.

1.1.4 The standard reporting limit (RL) is established at or above the low-level standard in the calibration curve (1 ug/l for most compounds). For a complete list of method detection limits (MDLs) and RLs, please see reference the current TALS (LIMS) active Method Limit Group database.

1.1.5 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (*Review of Work Request*) and 20 (*Test Methods and Method Validation*) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

1.1.6 Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in TestAmerica Edison SOP No. ED-GEN-003, *Standard Operating Procedure for Control of Non-Conformances and Corrective Action,* current revision NCM shall be filed in the project file and addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

2.0 Summary of Method

2.1 Methods 8260C and 8260D are used to determine volatile organic compounds in aqueous, non-aqueous and solid matrices. Sample preparation techniques vary, depending on the matrix and the level of contamination expected. Purge and trap techniques are used to introduce the sample to the GC/MS system. Refer to TestAmerica Edison SOP Nos. ED-MSV-001*, Purge and Trap for Aqueous Samples, SW846 Method 5030*, current revision and ED-MSV-002, *Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, SW846 Method 5035A,* current revision.

2.2 All samples extracts are screened by GC/FID static headspace analysis to provide the analyst with appropriate initial dilution factors. For additional details see

TestAmerica Edison SOP No. ED-GCV-001, *Screening for Volatile Organics, Static Headspace with GC FID, SW846 Method 5021*, current revision.

2.3 An aliquot of sample containing internal standard and surrogate spiking solution is purged with nitrogen in a closed sparging vessel. The volatile compounds are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatiles are trapped. After purging is complete, the sorbent column is heated and backflushed with helium to desorb the volatiles onto a gas chromatograph column.

2.4 Analytes eluted from the capillary chromatograhy column are introduced into the mass spectrometer via a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a minimum of a five-point calibration curve.

2.5 For aqueous VOA samples submitted for New Jersey Groundwater Quality Standard (NJ GWQS) evaluation, a full scan analysis is initially performed using the 8260 methodology No further analysis by SIM is required if all of the following compounds are present above the full scan RL: 1,2-dibromoethane, 1,2-dibromo-3-chloropropane, 1,2,3-Trichloropropane and 1,4-dioxane, chloroform, vinyl chloride and benzene. If any of these compounds are undetected in the undiluted, full scan analysis, the sample must be analyzed via 8260C SIM or 8260D SIM for those compounds.

2.6 In order to meet lower reporting limits of 0.5ug/L for most analytes, 2.5 ug/L for ketones and generally lower limits for other non-routine analytical compounds, samples must be analyzed against an initial calibration with a low point at those levels. The corresponding TALS login method for low level aqueous analysis is 8260_LL. See Table 3b for initial calibration levels and spike amounts.

3.0 Definitions

3.1 For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 This method is susceptible to contamination from a number of sources, including organic solvents used in other laboratory procedures, impurities in the purge gas, improper cleaning of syringes or purge vessels, and carryover from high level samples. Samples can be contaminated by the diffusion of volatile organics through the septum during shipment or storage. Steps have been taken to ensure that these potential problems are eliminated from the laboratory.

4.2 The volatiles analytical laboratory is housed in a separate building, away from the organic extraction lab area where large quantities of organic solvents are used. No organic solvents are used or stored in the volatiles laboratory.

4.3 The nitrogen used as purge gas passes through a solvent trap prior to its inlet into the purge and trap units.

4.4 Trip Blanks are shipped to clients with aqueous bottle ware as requested. The purpose of the trip blank is to detect and identify any VOC contamination of the samples while in transit to and from the lab. The blank is created at the laboratory by completely filling the volatile vial container with lab grade organic free deionized water and sealing the container Trip Blanks accompany bottle ware and samples through the sampling, storage and analysis stages as a check on contamination that may occur at these points.

4.5 Individual samples are each handled with a unique syringe that has been baked in a drying oven at 105°C to ensure the absence of volatile compounds.

4.6 Carryover can occur anytime a high level sample is analyzed. Screening procedures are employed to ensure that a sample is analyzed at an appropriate dilution to minimize potential carryover. When a high level sample is analyzed, it is followed by the analysis of a reagent water blank. If another sample was analyzed after the high level sample, this sample is inspected carefully for signs of carryover. If this sample does not contain any of the compounds found in the high level sample, the system can be considered contamination free.

4.7 The analytical system is checked daily with the analysis of a method blank. This blank must meet all quality control criteria for the method before sample analysis may take place.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

Any questions pertaining to safety issues or procedures should be brought to the department manager or Edison Safety Officer.

5.1 Specific Safety Concerns or Requirements

5.1.1 Latex, nitrile and vinyl gloves all provide adequate protection against the methanol used in this method.

5.1.2 Purge vessels on purge-and-trap instruments can be pressurized by the time analysis is completed. Vent the pressure prior to removal of these vessels to prevent the contents from spraying out.

5.1.3 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.1.4 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.1.5 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

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 MSDS 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 MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol (MeOH)	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.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Purge and trap units from several different manufacturers are used, depending upon the sample matrix and preparatory technique required. A purge and trap unit consists of three parts: the sample purge unit, the trap, and the concentrator. Unit configurations currently in use are:

- OI Analytical 4551, 4100 Automatic Sampler/4660,4760 concentrator;
- Archon 5100A Automatic sampler/ OI Analytical 4660,4760 concentrator;
- EST Centurion Autosampler/ EST Encon concentrator;

- Archon Autosampler/EST Encon concentrator.
- Archon/EST Evolution

6.1.2 A VOCARB 3000 trap from Supelco is used in the Encon concentrator. The trap is 25cm long with an inside diameter of 0.105 inches. The trap is packed with 10.0cm Carbopack B, 6.0 cm Carboxin 1000, and 1cm Carboxin 1001.

6.1.3 An OI analytical purge trap #10 is used for the OI 4560,4660 and 4760 concentrator. The trap is 25cm long with an inside diameter of 0.105 inches. The trap is packed to contain the following absorbents: Tenax/silica gel/carbon molecular sieve.

6.1.4 Alternate traps may be used provided the adsorption and desorption characteristics are equivalent to those of the trap recommended by the method.

6.1.5 Both the Encon and OI concentrators are capable of rapidly heating the trap to 260°C and holding at that temperature for the duration of the desorb time.

6.1.6 Gas chromatograph: HP Agilent 6890/7890 equipped with temperature programming capability.

6.1.7 GC column: 30M long x 0.25mm ID, 1.4um film thickness, 20M x 0.18mm x 1um DB-624 and 20M long x 0.18 mm ID Restek Rtx-VMS capillary column with 1um film thickness or similar phase.

6.1.8 Mass Spectrometer (Agilent 5973/5975/5977): scanning from 35-260 amu every 0.9 seconds, utilizing 70 volts (nominal) electron energy in the electron ionization mode and producing a mass spectrum which meets all EPA performance criteria when 50 ng of 4-Bromofluorobenzene (BFB) is injected through the gas chromatograph inlet.

6.1.9 GC/MS Interface: transfer lines heated to 180°C .

6.1.10 Data system: HP Chemstation II for data acquisition and TestAmerica Chrom for data processing.

6.2 Supplies

- Microsyringes: 10 ul to 1000 ul.
- Syringes: 5 ml to 25 ml gas-tight.
- Injection port liners: HP 18740-80200 or equivalent
- Volumetric flasks: Class “A” glassware, 5 ml to 500 ml.

- VOA vials: 20-ml and 40-ml glass with PTFE – faced septum.
- Vials: 2-ml amber glass with screw cap with Teflon-faced septa.
- Top loading analytical balance.
- Spatula: Narrow, stainless steel.
- Stir bars: PTFE coated, small enough to spin freely inside a VOA vial.

7.0 Reagents and Standards

7.1 Reagents

7.1.1 Organic free reagent water: High purity water that meets the requirements for a method blank when analyzed. (See section 9.1.1) Reagent water is obtained from Millipore system. Other methods of preparing reagent water are acceptable, provided that the water produced meets method blank criteria.

7.1.2 Methanol: Ultra Resi-Analyzed, purge and trap grade, purchased from JT Baker or equivalent. (Cat # 9077-02)

7.1.2.1 Each lot of methanol is screened for contaminants before being used for analysis as detailed in TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7.2 Standards

7.2.1 **Calibration Standards** Stock target compound analytical standard solutions are purchased mainly from Restek, Supelco, Inc, Absolute Standards and Spex although standards of similar quality from other suppliers may be substituted as required. Standards noted with an asterisk (*) are custom mixes made especially for TestAmerica Edison.

Target Analyte Standard Name	Concentration	Vendor	Catalog #
8260 List 1 / Std #3 Gases*	2500 ppm	Restek	569722
8260 List 1 / Std #3 Gases – (SS)*	2500 ppm	Restek	569722 sec
8260 List 1 / Std #1 MegaMix*	1250-62500 ppm	Restek	569720
8260 List 1 / Std #1 MegaMix (SS)*	1250-62500 ppm	Restek	569720 sec
8260 List 1 / Std #2 Ketones *	12500 ppm	Restek	569721
8260 List 1 / Std #2 Ketones * (SS)	12500 ppm	Restek	569721 sec
8260 List 1 / Std #5 Acrolein *	20,000 ppm	Restek	568720
8260 List 1 / Std #5 Acrolein (SS)	20,000 ppm	Restek	568720 sec
8260 List 1 /Std #4 2 CEVE *	2500 ppm	Restek	569723
8260 List 1 /Std #4 2 CEVE (SS) *	2500 ppm	Restek	569723 sec

Target Analyte Standard Name	Concentration	Vendor	Catalog #
8260 List 1 /Std #6 Vinyl Acetate *	5000 ppm	Restek	569724
8260 List 1 /Std #6 Vinyl Acetate (SS) *	5000 ppm	Restek	569724 sec
8260 List 2 / Std #1 Additions *	2500-62500ppm	Restek	568725
8260 List 2 / Std #1 Additions (SS) *	2500-62500 ppm	Restek	568725 sec
8260 List 3 / Std #1 Polar Additions *	2500-100000ppm	Restek	568728
8260 List 3 / Std #1 Polar Additions (SS) *	2500-100000 ppm	Restek	568728 sec
VOC Extra Standard 2015 *	2500-5000 ppm	Absolute	98593
VOC Extra Standard 2015 * (SS)	2500-5000 ppm	Absolute	98593
Epichlorohydrin	1000 ppm	Absolute	70377
Acrolein	5000 ppm	Restek	91980
Acrolein *	Neat	Sigma	110221
2-Freon Mix quote # 12258 *	2500ppm	Absolute	12258
2-Freon Mix quote # 12258 * (SS)	2500ppm	Absolute	12258
1,4-Dioxane	Neat	Sigma	360481
Epichlorohydrin	Neat	Sigma	45340
2-Chloroethylvinyl ether	Neat	Sigma	109983
1,4-Dioxane	1000 ppm	Absolute	70373
1,4-Dioxane	10000 ppm	Absolute	92785
Benzene	1000 ppm	Absolute	70025
Chloroform	1000 ppm	Absolute	70076

(1): The separate source for this material is not available as a distinct catalog number. Analyst must ensure that a separate lot of the material is selected and used as required.

An asterisk (*) indicates a custom standard mix.

7.2.1.1. Prepare stock solutions at volumes and concentrations indicated in Table 2 (Working Standards Preparation) by combining the indicated volumes of each stock solution into a volumetric flask corresponding to the total final volume. Dilute to the volume marker with methanol.

7.2.1.2. Prepare individual calibration standards as applicable per Section 9.2.2.1, Table 3, Initial Calibration Standards Preparation, Low Level Soil, Table 3a, Initial Calibration Standards Preparation (Low Level), Aqueous or Table 3B Initial Calibration Standards Preparation, Aqueous.

7.2.1.3. The 'Second Source' standards listed are used in the preparation of the Initial Calibration Verification (ICV) standard (see Tables 4 and 4a for ICV preparation instructions) and the Laboratory Control Standard (LCS) (see Section 9.1.3 and Tables 4 and 4a).

7.2.2 **Surrogate Standards:** Surrogate standard solutions are prepared from the stock solution (2500ppm)

Surrogate Standard Name	Concentration	Vendor	Catalog #
4-Bromofluorobenzene	2500ppm	Restek	567650
Toluene-d8			
1,2-Dichloroethane-d4			
Dibromofluoromethane			

7.2.2.1 A primary surrogate stock solution (2500 ppm each) is prepared from the neat standards as follows:

7.2.2.2 Secondary surrogate standard solutions are prepared at two (2) levels using the 2500 ppm primary stock solution as detailed in the table below:

Standard Name	Vendor	Catalog #	Volume added	Concentration of Stock Std.	Concentration of Standard	Total Volume Volume in MeOH/Total volume of MeOH
8260 Surrogate Mix: 4-Bromofluorobenzene Toluene-d8 1,2-Dichloroethane-d4 Dibromofluoromethane	Restek	567650	1ml	2500ppm	250ppm	10mL 9.0mL TV/M
8260 Surrogate Mix: 4-Bromofluorobenzene Toluene-d8 1,2-Dichloroethane-d4 Dibromofluoromethane	Restek	567650	1ml	2500ppm	50ppm	50mL 9.0mL TV/M

7.2.2.3 Methanol/Surrogate solution (2.5ug/mL): For methanol sampling field kits. Prepared by adding 1mL of 2500 ug/ml primary surrogate stock solution (see Section 7.2.2.1) to 1 L purge and trap grade methanol.

7.2.3 **Internal Standards:** Internal Standards Solutions are purchased from Restek:

Standard Name	Concentration	Vendor	Catalog #
8260 Internal Standard Mix: *Chlorobenzene-d5 *1,4-Dichlorobenzene-d4 *Fluorobenzene *1,4-Dioxane-d8 *TBA-d9	250-5000ppm	Restek	567649

7.2.4 **Internal Standard/Surrogate Mix (125 ppm each):** A solution containing both Internal Standards and Surrogates at 125 ppm is prepared in a 10ml volumetric flask as detailed below using the 2500

ppm surrogate stock solution prepared in Section 7.2.2.1 and the 2500 ppm internal standard mix detailed in Section 7.2.3:

Standard Name	Concentration of Stock Std.	Volume added to final volume of 20ml MeOH	Final Concentration of Standard
8260 Internal Standard/Surrogate Mix (125 ppm) For Aquatek Autosampler	2500 ppm Surrogate Mix	1.0ml	125 ppm each component
	250 Internal Std Mix	10 ml	

7.2.5 **Internal Standard/Surrogate Mix (SIM) (2.5/50 ppm each):** A solution containing both Internal Standards and Surrogates at 25 ppm is prepared in a 10ml volumetric flask as detailed below using the 2500 ppm surrogate stock solution prepared in Section 7.2.2.1 and the 250 ppm internal standard mix detailed in Section 7.2.3:

Standard Name	Concentration of Stock Std.	Volume added to final volume of 10ml MeOH	Final Concentration of Standard
8260 Internal Standard/Surrogate Mix (25 ppm) (SIM)	2500 ppm Surrogate Mix	10ul	2.5/50 ppm each component
	250 Internal Std Mix (Restek)	10ul	
1,4-Dioxane-d8	10000 ppm	50ul	

7.2.6 **GC/MS Instrument Performance Check (BFB):** The instrument performance check solution consists of 4-Bromofluorobenzene in addition to the other three surrogates in methanol. Prepare the solution at **50ppm as specified in section 7.2.2.2**. Assign an expiration date of 6 months.

7.2.7 All standards preparation information must be logged into the TALS Reagent Module. All pertinent information must be entered: Date prepared, Lot #'s, Expiration dates, Solvents used, Lab Lot # (expiration date), Manufacturer and Verification signature. Additionally, all prepped standards are typically given a unique Lot# and all information pertaining to standard preparation is entered into the GC/MS VOA Standard Preparation Log Book. Information such as standard supplier, lot number, original concentration, a description of how the standard was made, are required along with the laboratory lot number, analyst's initials, date prepared, expiration date and verification signature. Class "A" volumetric must be used at all times and syringes, preferably gas-tight syringes when available, should be checked for accuracy using an analytical balance. Class "A" pipettes should also be used if volumes permit.

7.2.8 Please refer to TestAmerica Edison SOP No. ED-GEN-008, *Standard Operating Procedure for Preparation, Purity and Storage of Reagents and Standards*, current revision. For Method 8260C and 8260D::

- Shelf Life of Standard:
  - Stock standards (Non-gases) - 6 months after opening vendor stock of up to 2000ppm, 3 years for 10,000ppm, 5 years for over 50,000ppm, or manufacturer's expiration date whichever comes first.
  - Stock Standards (Gases) - 2 months after opening vendor stock, or manufacturer's expiration date whichever comes first.
  - Working/Secondary dilution Standards (Non-gases) – 6 months after preparation, or manufacturer's expiration date whichever comes first.
  - Working/Secondary dilution Standards (Gases) – 1 week from the date of preparation for 50ppm and 2 weeks for 500ppm, or manufacturer's expiration date whichever comes first.
  - Daily Calibration Standards – 24 hours after preparation.

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- Storage Requirements:

  Aqueous standards are stored at 4°C and Methanol standards are stored at -10°C to -20°C.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Glass 40 ml vials	40 mLs	HCl, pH < 2; Cool 4 °C ± 2°C	14 Days / preserved 7 Days / unpreserved	SW846 Method 5030
Waters	Glass 40 ml vials	40mLs	TSP,pH>11 Cool 4 °C ± 2°C	14 Days / preserved	SW846 Method 5030

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Soils (Low)	Encore or Terracore (40 ml vials)	5 grams in 5 mls DI H_2O	Frozen Stored -7°C to -20°C	14 Days	SW846 Method 5035A
Soils (Med)	Encore or Terracore (40 ml vials)	5 grams in 10 mls MeOH	Cool 4 °C ± 2°C	14 Days	SW846 Method 5030
Soils (High)	Glass (Lab Prepared Kits)	10 grams in 25 mls MeOH	Cool 4 °C ± 2°C	14 Days	SW846 Method 5030

8.1.1 There are several methods of sampling soil. The recommended method is to take samples using an EnCore™ sampler or using a Terra Core™ sampling kit. At specific client request, unpreserved soil samples in 4oz jars may be accepted. For EnCore and Terra Core sampling, a separate jar is required for percent solids/moisture determination, unless one is supplied for another analysis.

8.1.2 For EnCore™ samplers, the 5g sample is extruded into a pre-weighed 40mL vial containing 5mL of methanol (medium level analysis) or reagent water (for low level, <50 µg/kg, analysis). The exact samples weight is determined as the difference between the vial + preservative weight and weight after the sample is added.

- Samples must be transferred (extruded from the sampler) and preserved within 48 hours of sampling.
- Water preserved samples are then frozen at <10°C. Methanol preserved samples may be stored at > 0.0 °C but < 6° °C or frozen.
- Methanol preserved samples are shaken for at least 2 minutes, and a portion of the methanol extract after settling may be transferred to a smaller Teflon-lined capped vial for storage below 6 °C
- Normally one (1) medium level and two (2) low-level samples are taken and preserved.
- One vial with a clean matrix of each preservation type is prepared at the same time as samples, to be used for LCS analysis. Spikes are not added until the time of analysis.
- Samples are spiked with internal standards and surrogates at the time of analysis.

8.1.3 Terra Core™ sampling kits are pre-preserved for use and immediate samples preservation in the field. Kits are shipped that include one (1) methanol preserved and two (2) reagent water preserved vials, along with a 4oz jar for solids/moisture analysis volume.

- Terra Core™ vials are immediately placed in the freezer (<-10°C) upon receipt at the lab. Methanol preserved vials are shaken for at least two (2) minutes to break up the solid and create the methanol extract.

- Terra Core™ vials are labeled with the weight of the vial and preservative. The vials are re-weighed prior to analysis to determine the weight of the solid sample added. It is important that labels NOT be added to these vials prior to weighing, because the weight of the label will add to the sample weight. Vials may be marked with indelible marker, or placed in a labeled, sectioned box until ID labels can be added after weighing.

8.2 Unpreserved soils - At client request, unpreserved soils packed into glass jars or brass tubes may be accepted and subsampled in the lab. A 5g portion of the sample is transferred to a 40mL vial and mixed with reagent water and/or methanol for analysis. Since this procedure is not compliant with SW5035A an NCM and case narrative statement describing the non-conformance must be included with any resulting data reported to the client.

8.3 Aqueous samples are stored in 40mL glass vials with Teflon lined septa at >0 and ≤ 6.0℃. Vials are required to have no headspace lar ger than a small pea.

8.3.1 Regulatory requirements for 2-Chloroethyl vinyl ether:

• 2-Chloroethyl vinyl ether: The stability of this compound is reduced when subjected to low pH, therefore samples for analysis to include 2-CEVE must be taken without acid preservation. Unpreserved samples must be analyzed within 7 days.

• SW846 Update V removed special preservation requirements for Acolein and Acylonitrile. These compounds may be analyzed for using a preserved sample vial.

8.4 Soil samples and water samples preserved to pH <2 with HCl have a maximum holding time is 14 days from sampling until the sample is analyzed. If water samples are known to be unpreserved, the holding time is 7 days from sampling to analysis.

8.4.1 Preserved water samples are checked to confirm the preservation pH AFTER analysis because the vials must not be opened prior to analysis. If the pH is found to be >2, this must be addressed in the case narrative.

8.5 Medium level solid methanol extracts, if taken at the time of preservation, are aliquoted into 4 mL glass vials with Teflon lined caps and stored at > 0.0 °C but ≤6.0℃ or frozen. The extracts are stored with minim um headspace.

8.6 Storage blanks are prepared by filling 40 mL VOA vials with reagent water and placing one in each refrigerator. After 1-2 weeks, the storage blanks are removed and analyzed. Additional details can be found in TestAmerica Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination,* current revision.

9.0 Quality Control

9.1 **Sample QC** - The following quality control samples are prepared with each batch of samples:

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]
Internal Standards	Every samples	Response within -50% to +100% of CCV

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into LIMS.

9.1.1. **Method blanks** are analyzed every 12 hours immediately after successful calibration verification (ICV and CCV) and before any samples are analyzed during the 12 hour clock. Analyze the blank in the same manner as the associated samples.

9.1.1.1. Prepare an aqueous blank by filling a 40 mL vial with reagent water and placing it in the autosampler. The autosampler will add the internal standard and/or surrogate standard.

9.1.1.2. Prepare a medium or high level blank in a 50 mL volumetric flask by adding 1.0 mL of purge and trap grade methanol to reagent water and bringing up to volume with the reagent water. The appropriate volume of this mix is added to the purge vessel. The autosampler will automatically internal standard and/or surrogate standard.

9.1.1.3. Prepare a low- level soil blank in a 40 ml VOA vial by adding a magnetic stir bar and 5 ml of reagent water and placing the vial in the autosampler tray. An additional 5mL of reagent water plus 1uL of 250ppm Internal Standard/Surrogate Mix (see Section 7.2.4) will be added by the Archon prior to purging.

9.1.1.4. To be considered acceptable, the method blank must not have any target analytes above the reporting limit. For method 8260D method blank is acceptable when target analyte concentrations are less than one-half of the reporting limit. Method blanks may contain analyte concentrations greater than acceptance limits if the associated samples in the batch are unaffected or sample

concentrations/responses are >10x the blank If method blanks are unacceptably contaminated with target compounds that are also present in field samples, all affected samples must be re-extracted and re-analyzed. Re-analysis is not necessary if the analyte concentration falls well below the action or regulatory limit or if the analyte is deemed not important for the project. Corrective action must be taken to identify and eliminate the contamination source. Demonstrate that acceptable blanks can be obtained before continuing with sample extraction and analysis. Method blanks must be analyzed on each instrument on which the associated samples are analyzed. Method blanks, trip blanks and other field blanks must be carried out through all stages of sample preparation and analysis.

9.1.1.5. Surrogate recoveries for the method blank must be within the laboratory generated limits. (Method 8260C /8260D requires the use of a minimum of three (3) surrogates. Since we are spiking with four (4) surrogates, either 1,2-Dichloroethane-d4 or dibromofluoromethane can be recovered outside of control limits without corrective action) .Internal standard area counts in the method blank must be within method specified limits. If any surrogate or internal standard is outside the limits, the method blank must re-analyzed.

9.1.2. Matrix Spike (MS)/Matrix Spike Duplicate (MSD): A matrix spike/matrix spike duplicate (MS/MSD) pair is extracted and analyzed with every 20 environmental samples of a specific matrix (defined as a sample batch which may contain up to 20 samples, and additional samples can be added to the batch for 14 days after the first sample was analyzed). Full compound list spiking is employed for MS/MSDs and LCSs. These spikes are prepared (as described in Section 9.1.2.1) concurrent with sample preparation. MS and MSD recoveries are calculated and compared to lab generated acceptance criteria which are updated annually. For acceptance limits, reference the current TALS (LIMS) active Method Limit Group database.

9.1.2.1. Prepare the MS/MSD as follows:

9.1.2.1.1 Low Level Soil: The low level soil MS/MSD is prepared as detailed in the following table. This is prepared in duplicate (one for the MS, the other for the MSD) in a 5 ml syringe filled with reagent water. Once prepped the solution is added to separate 40 ml vials each containing 5 gram aliquots of the sample to be spiked :

Standard Solution (Reference Table 2, Lab Names)	Concentration	Volume of Standard (ul) Added to 5.0 ml of Reagent Water	Final Concentration (ug/kg)
Gas Mix Li	50ppm	2	20

Standard Solution (Reference Table 2, Lab Names)	Concentration	Volume of Standard (ul) Added to 5.0 ml of Reagent Water	Final Concentration (ug/kg)
8260 combined	50ppm	2	20
Acrolein	500 ppm	3	300
Propenes	50ppm (varied)	2	20 (varied)
Freons	50 ppm	2	20

9.1.2.1.2 **Aqueous Samples:** The MS/MSD for aqueous samples is prepared as detailed in the following table. This is prepared in duplicate (one for MS, the other for MSD) in 50 ml volumetric flasks filled with an aliquot of sample to be spiked. Once prepped the solution is poured into a 40 ml VOA vial and loaded onto the purge and trap autosampler:

Standard Solution (Reference Table 2, Lab Names)	Concentration	Volume of Standard (ul) Added to 50 ml of Sample	Final Concentration(ug/L)
Gas Mix Li	50ppm	20	20
8260 combined	50ppm	20	20
Acrolein	500 ppm	4	40
Propenes	50ppm (varied)	20	20 (varied)
Freons	50	20	20

9.1.2.1.3 **Medium & High Level Soils:** The MS/MSD for medium/high level soils is prepared as detailed in the following table. This is prepared in duplicate (one for MS, the other for MSD) in 50 ml volumetric flasks filled with reagent water which has been previously spiked with the methanol sample extract. Once prepped the solution is poured into a 40 ml VOA vial, the and loaded onto the purge and trap autosampler:

Standard Solution (Reference Table 2, Lab Names)	Concentration	Volume of Standard (ul) Added to 50 ml of Reagent Water containing sample methanol extract	Final Concentration (ug/L)
Gas Mix Li	50ppm	20	20
8260 conbined	50ppm	20	20
Acrolein	500ppm	4	40
Propenes	50ppm (varied)	20	20 (varied)
Freons	50 ppm	20	20

9.1.2.1.4 **SIM:** The MS/MSD for SIM samples is prepared as detailed in the following table. This is prepared in duplicate (one for MS, the other for MSD) in 100 ml

volumetric flasks filled with an aliquot of sample to be spiked. Once prepped, two separate 10ml solution is poured into 40 ml VOA vials, 2ul of SIM IS/S is then added to each vial and loaded onto the purge and trap autosampler:

Standard Solution	Concentration	Volume of Standard Added to 100 ml of Sample (ul)	Final Concentration (ug/L)
8260SIM Mix1	10ppm	0.5	0.05
1,4-Dioxane	50ppm (varied)	10	5
Benzene/Chloroform	10ppm	0.5	0.05

9.1.2.2. An Laboratory Control Sample (LCS) /Laboratory Control Sample Duplicate (LCSD) may be substituted for the MS/MSD if insufficient sample volume is available (see Section 9.1.3).

9.1.3. Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD): A Laboratory Control Sample (LCS) (aka blank spike) must be prepared analyzed with each batch of 20 environmental samples. The LCS data is used to assess method performance if the MS/MSD recoveries fall outside of the lab generated limits (see For acceptance limits, reference the current TALS (LIMS) active Method Limit Group database). If the LCS recovery is within the current lab generated limits, the MS/MSD recoveries are attributed to matrix interference. If the LCS recovery results are outside the method specified, the LCS is reanalyzed. If, upon reanalysis, the LCS is it is still outside of limits the entire batch must be reanalyzed. For 8260D, when an LCS is prepared in the same manner as CCV, the same standard can be used as both the LCS and CCV.

9.1.3.1 For LCS preparation instructions please refer to Section 9.1.2.1 for low level soil introduction technique (note: use reagent water only, no solid matrix is used when preparing the LCS) and Sections 9.1.2.1.2 and 9.1.2.1.3 as applicable for aqueous/medium or high level solids introduction (note: use reagent water only, no sample or sample extract is used when preparing the LCS).

9.1.3.2 The LCS for SIM samples is prepared as detailed in the following table. This is prepared in a 200 ml volumetric flasks filled with organic free reagent water. Once prepped, 10ml of the solution is poured into a 40 ml VOA vial and 2ul IS/SS added manually and loaded onto the purge and trap autosampler

Standard Solution	Concentration	Volume of Standard Added to 200 ml of Reagent Water (ul)	Final Concentration (ug/L)
8260 Mix1	10ppm	1	0.05
1,4-Dioxane	50ppm	20	5
Benzene/Chloroform	10ppm	1	0.05

9.1.3.3 A Laboratory Control Sample Duplicate (LCSD) is analyzed only when insufficient client sample is available for preparation of an MS/MSD pair. The LCS/LSCD is evaluated in the same manner as the MS/MSD (see Section 9.1.2)

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with a four (4) component surrogate standard mix (see Section 7.2.2). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (For acceptance limits, reference the current TALS (LIMS) active Method Limit Group database).

9.1.4.1. Surrogate recovery limits are lab generated and are updated annually.

9.1.4.2. Surrogate recoveries are calculated for the blank, samples, and QC samples. Surrogate recovery is calculated as:

$$\frac{\text{Concentration found}}{\text{Concentration added}} \times 100 = \%\ \text{RECOVERY}$$

9.1.4.3. If the surrogate recoveries of any blank, sample, or QC sample fails to meet the current recovery criteria, the sample must be re-analyzed. If a surrogate is diluted to a concentration below that of the lowest calibration standard, no corrective action is necessary. Methods 8260C and 8260D requires the use of a minimum of three (3) surrogates. As we spike with four (4) surrogates, one can be recovered outside of control limits without corrective action.

9.1.5. Internal Standards: All samples, blanks, standards and QC samples are spiked with a five (5) component internal standard mix (See Section 7.2.3). The response (area count) and retention time of each internal standard in all samples, standards, blanks and QC samples are monitored.

9.1.5.1. The internal standard responses must be within -50 +100% of its corresponding internal standard in the mid-level calibration standard or the active calibration curve. Failure to meet these criteria is indicative of sample matrix effects. All samples failing these criteria must be reanalyzed to confirm matrix effects.

9.1.5.2. Internal standard retention time is evaluated immediately after acquisition. The retention times of the internal standards must be within ±30 seconds of the internal standards from the mid point standard of the initial calibration or the calibration verification standard. Any blank, sample, or QC sample that fails to meet these criteria must be re-analyzed.

9.2 Instrument QC

9.2.1 GC/MS Instrument Performance Check (BFB): The GC/MS system is tuned using Perfluortributylamine (PFTBA) such that an injection or purging of 50ng of 4-Bromofluorobenzene (BFB) meets the abundance criteria listed in the table below. Prior to the analysis of any calibration standards or samples, the GC/MS system must meet all BFB key ion abundance criteria. This analysis will verify proper tuning of the system for a period of 12 hours post-injection. After 12 hours, the instrument performance must again be verified prior to the analysis of standards, QC or samples. For method 8260D tune checks are only required prior to initial calibration. (**NOTE**: see Method Modifications in Section 16.0).

BFB Key Ions and Abundance Criteria	
Mass	**Ion Abundance Criteria**
50	15.0-40.0 percent of the base peak
75	30.0-60.0 percent of the base peak
95	Base peak, 100% relative abundance
96	5.0-9.0 percent of the base peak
173	Less than 2.0% of mass 174
174	Greater than 50% of the base peak
175	5.0-9.0 percent of mass 174
176	Greater than 95.0% but less than 101% of mass174
177	5.0-9.0 percent of mass 176

9.2.1.1. The BFB mass spectrum may be evaluated using one of the procedures listed below. The spectrum may be background subtracted using a single peak no more than 20 scans before the peak apex. The BFB spectrum must meet the technical acceptance criteria listed in the table above:

- A single scan on the peak;
- An average of the peak;
- Use of three scan averaging and background subtraction techniques. Select the scan at the BFB peak apex, add +1 scan from the apex and -1 scans from the apex;

9.2.1.2. BFB parameter settings are stored in a tune file, which ill be used in all subsequent analysis of standards and samples.

9.2.2 Initial Calibration Range and Initial Calibration Verification

9.2.2.1. **Initial Calibration:** The initial calibration range consists of a five-point concentrations (six points for second order regression) of analytical standards prepared as described in Tables 3, 3A and 3B as applicable (attached). The initial calibration range must be analyzed only after the BFB instrument performance check has met the criteria in Section 9.2.1. A separate initial calibration range is analyzed for each sample introduction technique. The last initial calibration standard may be used to be the start of the 12 hour clock for samples analyzed after initial calibration. Verify closely eluting isomers resolution in the mid-point concentration of the ICAL. Isomers are considered resolved if the peaks are at least 50% resolved (i.e., the height of the valley between two isomer peaks is less than or equal to 50% of the average of the two peak heights. This should also be checked in the daily CC's.

9.2.2.2. If analysis by the SIM technique is required, prepare calibration standards for, Vinyl Chloride, Chloroform, Benzene 1,2-dibromoethane, 1,2,3-Trichloropropane and 1,2-dibromo-3-chloropropane at concentrations of 0.02, 0.04, .05, 0.10, 0.20, 0.50, 1.0 and 2.0 ppb; 1,4-Dioxane at 0.4, 1, 5, 10, 20, 30, 40 and 50ppb. See Table 5 that summarizes the preparation information.

9.2.2.3. **Initial Calibration Verification (ICV):** An Initial Calibration Verification (ICV) standard is analyzed immediately after the Initial Calibration Range and before any samples are analyzed. The ICV is prepared as detailed in Section 7.2.1.3 and Tables 4 and 4a (full scan) and Table 6 (SIM) (attached). The ICV must be from a source separate from the standards used in the Initial Calibration Range.

9.2.3 **Continuing Calibration Verification (CCV):** A approximately mid-point (20ug/ml and 0.050/5ug/ml for SIM) Continuing Calibration Verification (CCV) must be analyzed every 12 hours after the BFB instrument performance check. BFB is not a requirement for 8260D CCV verification. The CCV is prepared as detailed in Section 7.2.1.1 and Table 3 (attached).

9.2.4 **Calibration Acceptance Summary**

9.2.4.1. **Retention Time:** The relative retention times of each compound in the five calibration standards must agree within 0.06 relative retention time units.

9.2.4.2. **Initial Calibration Range:** Internal standard calibration is employed for this method. After the initial calibration range has been analyzed as detailed in Section 10.3.3 the relative response factor (RRF) for each target/surrogate compound at each concentration level is determined using the following equation.

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:
A_x = Area characteristic ion for the compound (see attached Table 7)
Ais = Area characteristic ion of internal standard (see attached Table 7)
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

9.2.4.2.1. Determine the mean RRF for each compound using the five or six RFs from the initial calibration range.

9.2.4.2.2. The average RFs of the target analytes listed in the table below must meet the indicated minimum RF criteria:

Minimum Relative Response Factor	
Common Target Analytes	**Minimum RF**
Dichlorodifluoromethane	0.100
Chloromethane	0.100
Vinyl Chloride	0.100
Bromomethane	0.100
Chloroethane	0.100
Trichlorofluoromethane	0.100
1,1-Dichloroethene	0.100
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100
Acetone *	0.050
Carbon disulfide	0.100
Methyl Acetate *	0.005
Methylene chloride	0.100
trans-1,2-Dichloroethene	0.100
cis-1,2-Dichloroethene	0.100
Methyl tert-Butyl Ether	0.100
1,1-Dichloroethane	0.200
2-Butanone *	0.050
Chloroform	0.200
1,1,1-Trichloroethane	0.100
Cyclohexane	0.100
Carbon tetrachloride	0.100
Benzene	0.500
1,2-Dichloroethane	0.100
Trichloroethene	0.200
Methylcyclohexane	0.100
1,2-Dichloropropane	0.100
Bromodichloromethane	0.200
cis-1,3-Dichloropropene	0.200
trans-1,3-Dichloropropene	0.100
4-Methyl-2-pentanone *	0.050
Toluene	0.400

Minimum Relative Response Factor	
Common Target Analytes	**Minimum RF**
1,1,2-Trichloroethane	0.100
Tetrachloroethene	0.200
2-Hexanone*	0.050
Dibromochloromethane	0.100
1,2-Dibromoethane	0.100
Chlorobenzene	0.500
Ethylbenzene	0.100
meta-/para-Xylene	0.100
ortho-Xylene	0.300
Styrene	0.300
Bromoform	0.100
Isopropylbenzene	0.100
1,1,2,2-Tetrachloroethane	0.300
1,3-Dichlorobenzene	0.600
1,4-Dichlorobenzene	0.500
1,2-Dichlorobenzene	0.400
1,2-Dibromo-3-chloropropane	0.050
1,2,4-Trichlorobenzene	0.200

Note: Alternate ions chosen for the analytes in the table above may result in lower than recommended value

* These values are lower than method recommended values.

9.2.4.2.3. Any individual analyte that fails the minimum response factor above must have a demonstration of sensitivity in the analytical batch to report non-detects. The demonstration of sensitivity is analysis of a low level CCV (at or below the reporting limit). The criterion for a passing LLCCV is detection only, and a passing LLCCV allows non-detects to be reported without flagging. The low level CCV would normally be analyzed immediately after the mid-level CCV

9.2.4.2.4. Calculate the Standard Deviation (SD) and Percent Relative Standard Deviation (% RSD) of the response factors for each compound:

$$\%\ RSD = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

The % RSD of the common target compounds listed above must be ≤20% in order for the calibration range to be acceptable. If more than 10% of the compounds exceed the 20%RSD limit and do not meet the minimum correlation coefficient (0.99) for alternative curve fits, appropriate instrument maintenance like source cleaning should be performed. Any compound that do not meet the 20%

RSD or 0.99 correlation coefficient criteria must be flagged as estimated for detects.

9.2.4.2.5. For all compounds (including those analyzed by SIM): in order to assume linearity, the % RSD of the RRF's for each target analyte must be ≤20%.

9.2.4.2.6. If the above listed criteria is met, the system can be assumed to be linear, sample analysis may begin and the average RF from the initial calibration range may be used to quantitate all samples.

9.2.4.2.7. An alternative calibration technique may be employed for those any compounds exceeding the 20% RSD criteria:

9.2.4.2.6.1 Linear regression: Calculate the first order linear regression for any compound which did not meet the 20% RSD criteria. The r value (Correlation Coefficient) of the equation must be ≥0.99 for linear regression to be employed.

9.2.4.2.6.2 Quadratic (or second order) regression: may be used if the linear regression correlation coefficient exceeds criteria. Quadratic regression requires the use of a minimum six calibration points. If second order regression calibration is used, the r^2 (Correlation Coefficient) value must be $\geq$ 0.99

9.2.4.2.8. If neither of the alternative calibration techniques meets acceptance criteria i.e for more than 10% of the analytes fail both 20%RSD and 0.990 the calibration is not valid. Corrective action must be taken and the initial calibration range reanalyzed.

9.2.4.2.9. Non-detect results for any analyte that fails both 20%RSD and 0.990 correlation coefficient may be reported without flagging if (and only if) there has been a successful analysis of a LLCCV (CCV at the reporting limit) in the same analytical batch. The criterion for the LLCCV is detection only (%D criteria are not applied) but the standard qualitative criteria in the method must be met. Flagging of detected analytes results as estimated is discouraged when the 20%RSD and 0.990 criteria fails. In general no more than one or two of the poorest performing analytes should fail both criteria.

9.2.4.2.10. Due to significant bias to the lower portion of a calibration curve using the linear regression fit model a quantitation check on the viability of the lowest calibration point should be performed by re-fitting the

response from the low concentration calibration standard back into the curve as if it were an unknown sample (rename the lower point calibration file as a separate data file before re-processing). The results should be within ±30% of the standard's true concentration. This is not required for average RF or quadratic fits. Additionally forcing a linear regression through zero will meet the requirement of not re-fitting. Analytes which do not meet the minimum quantitation calibration re-fitting criteria should be considered 'out of control'. Report those target analyte outliers as estimated when the concentration is at or near the lowest calibration point and/or report to the next reporting level (i.e., the next higher calibration point for the analyte).

9.2.4.2.11. For additional detail refer to TestAmerica Edison Work Instruction No. EDS-WI-096, *8260C ICAL Procedure*, latest revision.

9.2.4.3. Calibration Point Read-back Criteria: Whichever calibration model above is selected, it should be subjected to an additional check to establish the representativeness of the data that were used to produce it. This check is the refitting of each calibration point response back to the calibration model, or the comparison of the calculated amount of the standard against the expected amount.

- CHROM software provides an Initial Calibration %Drift report which shows the % Error for each calibration point. This report must be reviewed in addition to the %RSD Linear Response Factor.

- The absolute value of the % Error for each calibration point should be < 30%. For the lowest calibration point, the % Error may be < 50%. Relative standard error (RSE) can also be used and must be ≤ 20% for each calibration point. See section 11.10 for the Calculation of the %Error.

9.2.4.4. Initial Calibration Verification (ICV): Once the initial calibration has been analyzed and has met the above criteria, a second source Initial Calibration Verification (ICV) (as prepared in Section 9.2.2.2) must be analyzed and evaluated. The ICV must meet the criteria of 70-130% recovery for all compounds however up to 10% of the compounds are allowed to exceed this criteria as long as their recoveries are within 65-135%. For the poor performers the range is 50-150%. If the criterion is not met, a second ICV may be analyzed after corrective measures are taken. If a second ICV analysis fails to meet criteria

proceed with corrective action and the analysis of a new initial calibration range. Flagging: If the ICV limits are outside of criteria (high) for an analyte and that analyte is undetected in the sample, no flagging or narration is required. If the ICV limits are outside of criteria (low) for an analyte and that analyte is undetected in a sample, narrate the non-conformance in an NCM. When that out of spec analyte is detected in a sample, describe the issue in the narrative, or flag as estimated.

9.2.4.5. Continuing Calibration Verification (CCV): A CCV consisting of a standard at or near the midpoint of the Initial Calibration Range is analyzed every 12 hours of instrument operation or at the beginning of an analytical sequence to verify the initial calibration. The calibration verification consists of a BFB instrument performance check, and analysis of a calibration verification standard.

9.2.4.4.1 Tune Verification: Follow the procedure for verifying the instrument tune described in section 9.2.1 using a 50 ng injection of BFB. If the tune cannot be verified, analysis must be stopped, corrective action taken and a return to "control" demonstrated before continuing with the calibration verification process. For method 8260D, tune verification is not required for daily CCV.

9.2.4.4.1.1 Calibration Verification: Analyze the calibration verification standard immediately after a BFB that meets criteria. For method 8260D, BFB is not needed. Use the mid point calibration standard (20ug/L). **NOTE:** The same sample introduction technique employed for the initial six-point calibration must be used for the calibration verification.

9.2.4.4.1.2 Calculate response factors (RF) for each compound using the internal standard method.

9.2.4.4.1.3 The RFs must meet the minimum RF criteria listed in the table in Section 9.2.4.2.2.

9.2.4.4.1.4 Calculate the % Difference for each response factor in the calibration check standard vs. the response factors from the initial calibration.

9.2.4.4.1.5 If the percent difference/drift (%D) for the compounds listed in the table in Section 9.2.4.2.2 is ≤20%, the initial calibration is assumed to be valid. If the ≤20% D criteria is not met for more than 20% of the compounds

in the initial calibration, corrective action/ investigation may be taken. After corrective action, another calibration verification standard may be injected. If the response for the analyte is still not ≤20%, a new initial calibration range must be generated.

9.2.4.4.1.6 For the poor performing compounds listed below that fail the 20%D or 50%D criteria adequate sensitivity may be demonstrated by including a low level standard (LLCCV) in the analytical batch.

Poor Performers	
Acetone	Acrolein
Carbon disulfide	1,4-Dioxane
2-Butanone	Cyclohexane
2-Hexanone	Methyl cyclohexane
4-Methyl-2-pentanone	Benzyl chloride
Chlorodibromomethane	Naphthalene
1,2-Dibromo-3-chloropropane	Cis-Dichloropropene
Bromomethane	Trans-Dichloropropene
Chloroethane	All Alcohols

When samples have non-detects for an analyte that fails the SOP criteria with low recovery a low level CCV must be analyzed in the batch as a demonstration of adequate sensitivity. The criterion for a passing LLCCV is detection only, and a passing LLCCV allows non-detects to be reported without flagging. Any sample detects for an analyte that fails the SOP criteria must be flagged as estimated, or detailed in the case narrative. In all cases every effort should be made to re-analyze on an instrument with a passing CCV.

9.2.4.4.1.7 Percent drift is used instead of percent difference in calibrations employing either the linear or second order regression modes.

9.2.4.4.1.8 For the compounds not listed in the table in Section 9.2.4.2.2: No one individual compound of interest may exceed 50%D. For SIM analysis the %D is 20%.

9.2.4.4.1.9 The retention times of the internal standards from the calibration check must be within ±30 seconds of the internal standards from the mid point standard of the original calibration. If the retention time for any internal standard changes by more than 30 seconds from the

latest daily (12 hour) calibration standard, the chromatographic system is inspected for malfunctions, and corrections made as required. If corrective action does not result in the retention time criteria being achieved, the system must be re-calibrated using four additional standards.

9.2.4.4.1.10 Internal standard area response is also evaluated immediately after acquisition. The response (area count) of each internal standard in the calibration verification standard must be within 50% - 100% of its corresponding internal standard in the mid-level calibration standard of the initial calibration curve. If the EICP area for any internal standard changes by more than a factor of two (-50% to +100%), the mass spectrometer system must be inspected for malfunction and corrections made as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning is required.

10.0 Procedure

10.1. Gas Chromatograph/Mass Spectrometer Operation

10.1.1. The instrument operating parameters are set as follows at the beginning of a method of analysis and remain constant throughout the entire analytical procedure

10.1.1.1 Full Scan Operating Mode

Purge and trap unit

Purge Time:	11 minutes
Dry Purge:	1 Minutes
Purge Gas:	Nitrogen
Purge Flow:	40-45 ml/min
Purge Temp:	Water: Ambient; Solids: 40°C
Trapping Temp:	Ambient, <30°C
Desorb Time:	1 Minute
Desorb Temp:	VOCARB: 260°C, #10: 190°C

Gas chromatograph

Injector:	180°C

Carrier Gas:	Helium
Carrier Flow:	6 ml/min, 6890: 0.8 ml/min
Oven Program:	40°C for 1 min, 8°C/min to 90°C, 20°C/ min to 250°C for 3 min; 6890: 40°C for 1 min, 8°C/min to 100°C, 24°C/min to 220°C for 2 min
Run Time:	15 - 20 Minutes

Mass Spectrometer

Electron Energy:	70 volts (nominal)
Mass range:	35-260 AMU
Scan time:	0.9 sec./scan
Source Temp:	200°C
Separator Temp:	180°C

10.1.1.2 SIM Operating Mode

Purge and trap unit

Purge Time:	11 minutes
Dry Purge:	1 Minutes
Purge Gas:	Nitrogen
Purge Flow:	40-45 ml/min
Purge Temp:	Water: Ambient; Solids: 40°C
Trapping Temp:	Ambient, <30°C
Desorb Time:	1 Minute
Desorb Temp:	VOCARB: 260°C, #10: 190°C

Gas chromatograph

Injector:	180°C
Carrier Gas:	Helium
Carrier Flow:	6 ml/min, 6890: 0.8 ml/min
Oven Program:	40°C for 1 min, 8°C/min to 90°C, 20°C/ min to 250°C for 3 min; 6890: 40°C for 1 min, 8°C/min to 100°C, 24°C/min to 220°C for 2 min
Run Time:	15 - 20 Minutes

Mass Spectrometer

Electron Energy:	70 volts (nominal)
Mass range:	35-260 AMU
Scan time:	0.9 sec./scan
Source Temp:	200°C
Separator Temp:	180°C

SIM Parameters:

Group 1
Plot 1 Ion: 51.0/96

Ions/Dwell in Group	(Mass	Dwell)	(Mass	Dwell)	(Mass	Dwell)
	51.0	100	58.0	100	65.0	100
	67.0	100	70.0	100	88.0	100
	96.0	100	78.0	100	83.0	100
	85.0	100	62.0	100	64.0	100

Group 2
Group Start Time: 6.20
Plot 1 Ion: 82/117

Ions/Dwell in Group	(Mass	Dwell)	(Mass	Dwell)	(Mass	Dwell)
	82.0	100	107.0	100	109.0	100
	117.0	100				

Group 3
Group Start Time: 8.50
Plot 1 Ion: 75/157

Ions/Dwell in Group	(Mass	Dwell)	(Mass	Dwell)	(Mass	Dwell)
	75.0	100	95.0	100	150.0	100
	152.0	100	152.0	100	157.0	100
	174.0	100				

10.2. Sample Preparation

10.2.1. Screening: All samples extracts must be screened by GC/FID static headspace analysis to provide the analyst with appropriate initial dilution factors. For additional details see TestAmerica Edison SOP No. ED-GCV-001, *Screening for Volatile Organics, Static Headspace with GC FID, SW846 Method 5021*, current revision.

10.2.2. Aqueous Samples: Unopened 40 mls vials with aqueous samples are placed in an Archon autosampler. 1 uL of Internal Standard/Surrogate Mix (see Section 7.2.4) is added by the Archon as the 5 mL of the sample passes through the sample loop.

10.2.3. Medium or high level soils: Medium or high level extracts that will be run on an Archon autosampler are prepared in 50mL volumetric flasks. The Archon can be set up to add 1uL of 250ppm Internal Standard/Surrogate separately (see Section 7.2.3 and 7.2.2.2) to each sample as the 5mL portion passes through the sample loop.

10.2.4. Low level soils: Low level soils must be run on an Archon autosampler. 1uL of 250ppm Internal Standard/Surrogate separately (see Section 7.2.3

and 7.2.2.2) and 5mL reagent water is added to each sample vial by the Archon immediately before the sample is purged.

10.2.5. **SIM analysis:** Aliquot 10ml of sample and manually add 2ul of 2.5/50ppm of internal standard/surrogate mix. Load to soil section of the autosampler for heated purge.

10.3. Instrument Performance and Calibration Sequence

10.3.1. Once the GC/MS instrument has been setup and maintained as detailed in Section 10.1, the first operations to be performed are the performance checks and calibration standards.

10.3.2. Analyze the Instrument Performance Check Standard (BFB) as discussed in Section 9.2.1.

10.3.3. A unique initial calibration is then prepared for each sample introduction technique.:

10.3.3.1 **40 ml VOA Vial (Aqueous/Medium-High Level Soils):** Prepare aqueous calibration standards at six concentration levels for each parameter by adding the volumes of working standards listed in Table 3 to a 50mL volumetric flask of reagent water. Pour the calibration standards into 40mL VOA vials and load into the autosampler tray. If the internal standard is to be added by the Archon/OI autosamplers the addition of internal standard into the 50ml volumetric flaks may be omitted.

10.3.3.2 **40 ml VOA Vial (Low Level Soils):** If the calibration is for low-level soils prepared according to Method 5035AA, the calibration standards must be prepared by adding the volumes of working standards listed in Table 3 into a 5 mL syringe filled with reagent water and pouring the prepared standards into 40 mL VOA vials containing a magnetic stir bar.

10.3.4. Purge the standard for 11 minutes.

10.3.5. After purging is complete, desorb the sample onto the GC column by rapidly heating the trap to 260°C for VOCARB, 190°C for #10 and backflushing it with helium.

10.3.6. Begin the GC temperature program and data acquisition.

10.3.7. Re-condition the trap by baking for 12 minutes at 260°C for VOCARB, 210°C for #10.

10.3.8. Cool the trap to (<31°C). The trap is now ready for the next sample.

10.3.9. Transfer data to network, and process using CHROM software.

10.4. Sample Analysis Sequence

10.4.1. Once the initial calibration has been verified by successful analysis of an ICV and Method Blank, analysis of samples may begin.

10.4.2. Samples must be analyzed under the same instrument conditions and using the same injection volume as the calibration standards.

10.4.3. Equilibrate all samples to room temperature prior to analysis.

10.4.4. If the sample concentration exceeds that of the range, the sample must be diluted and re-analyzed.

10.4.5. The analytical run log is printed as a record of samples analyzed. The analyst will annotate the run log with any required information regarding anomalies or unusual events. The run log must be signed by the analyst and a reviewed and signed by a trained peer or manager

10.5. Data Processing

10.5.1. Prior to processing any standards or samples, target compound lists and sublists must be assembled in the Chrom system. These lists are required for processing of all data files including calibration files. The data includes compound names, retention time data, quantitation ions, qualitative identification ions, and the assigned internal standard for qualitative and quantitative identification.

10.5.2. Key data is manually entered the first time a compound list is used for data processing. Processing data using a compound list automatically generates response factor data and updates retention information.

10.5.3. Data is transferred from the acquisition PC to the network for auto-processing with CHROM software.

10.5.4. Each data file is checked for correct information including sample number, job number, QA batch, dilution factor, initial volume, final volume, and % moisture.

10.5.5. The data processing service from Chrom queries LIMS for the sample processing parameters.

10.5.6. Each data file is processed using calibration factors from the most recent initial calibration, quantitation from the daily calibration verification standard is not permitted.

10.5.7. The characteristic ions for target compounds, surrogate compounds, and internal standards which can be determined using SW8260C and 8260D are listed in Table 7.

10.6. Interpretation and Qualitative Identification:

10.6.1 Target Analytes: Qualitative identification of target compounds is based on retention time and mass spectral comparison with characteristic ions in the target compound list. The reference mass spectrum is taken from a standard of the target compound analyzed by this method. The characteristic ions are the three ions of greatest relative intensity or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met:

10.6.1.1. Once the GC/MS instrument has been setup and maintained as detailed in Section 10.1, the first operations to be performed are the performance checks and calibration standards.

10.6.1.2. The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other.

10.6.1.3. The relative retention time (RRT) of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

10.6.1.4. The most abundant ion in the standard target spectrum that equals 100% MUST also be present in the sample target spectrum.

10.6.1.5. All other ions that are greater than 10% in the standard target spectra should also be present in the sample.

10.6.1.6. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

10.6.1.7. Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Otherwise, structural isomers are identified as isomeric pairs.

10.6.1.8. If the compound does not meet all of the criteria listed above, but is deemed a match in the technical judgment of the mass spectral interpretation specialist, the compound will be positively identified and reported with documentation of the identification noted in the raw data record.

10.6.2 Non-Target Analytes: Upon client request a library search to identify non-target Tentatively Identified Compounds (TIC) is performed. The NIST/EPA/NIH mass spectral library is used to identify non-target compounds (not including internal standard and surrogate compounds) of

greatest apparent concentration by a forward search of the library. The following guidelines are used by the analyst when making TIC identifications:

10.6.2.1 Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

10.6.2.2 The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

10.6.2.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

10.6.2.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

10.6.2.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

10.6.2.6 If, in the technical judgement of the mass spectral interpretation specialist, no tentative identification can be made, the compound will be reported as 'Unknown'. If the compound can be further classified the analyst may do so (i.e, 'Unknown hydrocarbon', 'Unknown acid' , etc..).

10.7. Data Reporting

10.7.1. Final Report. LIMS TALS system automatically produces a data report consisting of key, hardcopy reports corresponding to specific data reporting requirements.

10.7.1.1. Total Ion Chromatogram. Full length chromatogram depicting the full length of the GC/MS acquisition.

10.7.1.2. Spectra of all detected target compounds. A page for each detected target compound spectra with a standard reference spectrum for comparison.

10.7.1.3. The calculations of the concentrations of each target compound in the sample, reported in units of ppb, ug/kg or ug/l.

10.7.1.4. Data summaries for each method blank indicating which samples were extracted with the indicated blank.

10.7.1.5. A copy of the initial calibration range together with the calibration verification report, and tune report.

10.7.1.6. Quality Control (QC) data report for each batch including surrogate recoveries, internal standard area summaries, LCS, MS/MSD and RPD summaries.

11.0. Calculations / Data Reduction

11.1. Target Compounds: are quantitated using the internal standard method.

11.1.1. Identified target compounds are quantitated using the integrated abundance from the EICP of the primary characteristic ion. The internal standard used shall be the one nearest the retention time of the analyte).

11.1.2. The average response factor (RRF) from the initial calibration is used to calculate the target analyte concentration in client samples using the formula found in Section 11.3.. See Section 9.2.4.2 for discussion of RRF.

11.1.3. Secondary ion quantitation is utilized only when there are sample interferences preventing use of the primary characteristic ion. If secondary ion quantitation is used an average relative response factor (RRF) must be calculated using that secondary ion.

11.1.4. Aqueous Samples

$$\text{Concentration } (\mu g/L) = \frac{(As)(Cis)(D)}{(Ais)(RRF)(Vs)}$$

Where:

As	=	Area of the characteristic ion for the target analyte in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Ais	=	Area of the characteristic for the associated internal standard

RRF = Average relative response factor from the initial calibration.

Vs = Volume of sample purged (ml)

11.1.5. Low Level Solid Samples

$$\text{Concentration (µg/Kg) (dry wt)} = \frac{(As)(Cis)}{(Ais)(RRF)(Ws)(DW)}$$

Where:

As = Area of the characteristic ion for the target analyte in the sample

Cis = Concentration of the internal standard (ug/L)

DW = Dry wt correction = $\frac{100 - \%\ \text{moisture}}{100}$

Ais = Area of the characteristic for the associated internal standard

RRF = Average relative response factor from the initial calibration.

Ws = Weight of sample purged (g)

11.1.6. Medium Level Solid Samples

$$\text{Concentration (µg/Kg) (dry wt)} = \frac{(As)(Cis)(Vt)(1000)(D)}{(Ais)(RRF)(Va)(Ws)(DW)}$$

Where:

As = Area of the characteristic ion for the target analyte in the sample

Cis = Concentration of the internal standard (ug/L)

D = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1

DW = Dry wt correction = $\frac{100 - \%\ \text{moisture}}{100}$

Ais = Area of the characteristic for the associated internal standard

RRF	=	Average relative response factor from the initial calibration.
Va	=	Volume of the aliquot of sample methanol extract added to reagent water for purging in ul
Vt	=	Total volume of methanol extract in milliliters
Ws	=	Weight of sample purged (g)

11.2. Non-Target Compounds (Tentatively Identified Compounds): An estimated concentration for non-target (tentatively identified compounds) is calculated using the internal standard method . For quantiation, the nearest eluting internal standard free of interferences is used. The procedure used for calculating the concentration of non-target compounds is the same as that used for target compounds (see Section 11.1) with the following revisions:

11.2.1. The total area count of the non-target compound is used for As (instead of the area of a characteristic ion).

11.2.2. The total area count of the chosen internal standard is used as Ais (instead of the area of a characteristic ion).

11.2.3. A RF on 1.0 is assumed.

11.2.4. The resulting concentration is qualified as estimated ('J') indicating the quantitative uncertainties of the reported concentration.

11.3. Relative Response Factors

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

A_x = Area characteristic ion for the compound (see Table 7)
Ais = Area characteristic ion of associated internal standard (See Table 7)
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

11.4. Percent Relative Standard Deviation (% RSD) : as discussed in Section 9.2.4.2. (Initial calibration):

$$\% \text{ RSD} = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

11.5. Percent Difference (% D):as discussed in Section 9.2.4.4 (Continuing calibration):

$$\% D = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} X\ 100$$

Where: RRFc = RRF from continuing calibration

$\overline{RRF_i}$ = Mean RRF from current initial calibration

11.6. Percent Recovery (% R): Surrogates and Spikes

$$\text{Recovery (\%)} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}} \times 100$$

11.7. Dry Weight Correction: All solid samples must be corrected for dry weight using the following formula for dry weight determination.

$$DW = \frac{Gd}{Gw} \times 100$$

Where:

DW	=	Percent % Dry Weight
Gd	=	Dry weight of selected sample aliquot
Gw	=	Wet weight of selected sample aliquot

Multiply the DW value times the wet weight of the sample extracted. NOTE: This calculation can also be performed automatically by the target system provided the DW value is available and entered into the system.

11.8. Accuracy:

$$\text{ICV , CCV and LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.9. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.10. Calculation of Percent (%) Error:

$$\%Error = \frac{x_i - x_i'}{x_i} x\,100$$

Where:

x_i ' = Measured amount of analyte at calibration level *i*, in mass or concentration units

x_i = True amount

12.0 Method Performance

12.1. Method Detection Limit Study (MDL)

A Method Detection Limit (MDL) study, as described in the TestAmerica corporate Detection and Quantitation Limits SOP, CA-Q-S-006, must be performed initially and whenever a significant change affecting sensitivity is made to the analytical system. The MDL must be re-evaluated from quarterly MDL points at least every 12 months.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Lower Limit of Quantitation Verification

The lowest calibration standard analyzed establishes the LLOQ or Reporting Limit. The capability to reliably detect this concentration through the preparation, clean-up and analytical procedure is verified through the annual analysis of a standard at the LLOQ/RL. The LLOQ verification shall also be performed whenever significant changes are made to the preparation and/or analytical procedure.

12.3.1 The LLOQ verification standard shall be prepared at a concentration 0.5-2 times the LLOQ/RL, and be taken through all of the same preparation and clean-up methods as client samples.

12.3.2 The LLOQ verification standard for aqueous matrix shall be prepared using laboratory deionized water and for the solid matrix using clean Ottawa sand. Other clean matrices may be used in addition, for project specific requirements.

12.3.3 The LLOQ shall be verified annually on each instrument used for client sample analysis.

12.3.4 Recovery of each analyte must meet the laboratory established LCS

recovery limits + 20%. (For example, if the LCS recovery limits are 70-130%, the LLOQ verification must meet recovery limits of 50-150%.) Once sufficient points have been generated, LLOQ based statistical limits may be used in place of limits based on LCS recovery.

NOTE: The lower recovery limit for the LLOQ can be no lower than 10%.

12.4. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, (*Training*), for the laboratory's training program.

13.0 Pollution Control

13.1. 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."

14.0 Waste Management

14.1. 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 TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Practices*, current revision. The following waste streams are produced when this method is carried out.

- Laboratory Generated Aqueous Waste (aqueous VOA vials – used and unused). This waste may have a pH of less than 2.0. These vials are collected in satellite accumulation. The vials are then transferred to the waste room. These vials are passed through a vial crusher and the liquid portion is separated from the solid portion. The solid is dumped into the municipal garbage. The liquid is pumped into the neutralization system where it is neutralized to a pH of 6 to 9 with sodium bicarbonate (Seidler Chemical SC-0219-25). When neutralization is complete, the material is transferred to the municipal sewer system.

- Expired Standards – The vials are collected in a 1 gallon polyethylene bucket. These vials are then transferred to an open top 55 gallon steel or polyethylene waste drum. These drums are transported to a waste facility for proper disposal.

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These

boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

Teris Profile Number (incineration): 50016710
Onyx Profile Number: (stabilization) 402535

- Methanol Preserved Samples/Returned Methanol Preservative - Methanol preserved sample vials are collected in satellite accumulation and then transferred to a 55 gallon open top steel waste drum in the waste room. This drum is then removed by a waste vendor for incineration.

  Teris Profile Number: 50016652
  Onyx Profile Number: 282493

15.0 References / Cross-References

15.1. United States Environmental Protection Agency, “Method 8260C, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)”, Test Methods for Evaluating Solid Wastes, SW846, August 2006.

15.2. .United States Environmental Protection Agency, “Method 8260D, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)”, Test Methods for Evaluating Solid Waste, SW846, Update VI, Revision 4, June 2018.

15.3. United States Environmental Protection Agency, “Method SW8000D: Determinative Chromatographic Separations”, Test Methods for Evaluating Solid Wastes, SW846, Laboratory Manual, Physical/Chemical Methods, Update V, Revision 4, October 2012.

15.4. U.S. EPA. 2003. "Method 5030C (SW-846): Purge-and-Trap for Aqueous Samples," Revision 3. Washington, DC.

15.5. U.S. EPA. 2002. "Method 5035A (SW-846): Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples," Draft Revision 1. Washington, DC.

15.6. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.7. TestAmerica Document No. CW-E-M-001, Corporate Environmental Health and *Safety Manual,* current revision.

15.8. TestAmerica Edison SOP Nos. ED-MSV-001*, Purge and Trap for Aqueous Samples, SW846 Method 5030*, current revision.

15.9. TestAmerica Edison ED-MSV-002, *Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, SW846 Method 5035A,* current revision.

15.10. TestAmerica Edison SOP No. ED-GCV-001, *Screening for Volatile Organics, Static Headspace with GC FID, SW846 Method 5021*, current revision.

15.11. TestAmerica Corporate Quality SOP No. CA-Q-S-001, *Solvent & Acid Lot Testing & Approval*, current revision.

15.12. TestAmerica Edison SOP No. ED-GEN-023, *Bulk Solvent Testing and Approval*, current revision.

15.13. TestAmerica Edison SOP No. ED-GEN-008, *Standard Operating Procedure for Preparation, Purity and Storage of Reagents and Standards*, current revision

15.14. TestAmerica Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination,* current revision

15.15. TestAmerica Edison Work Instruction No. EDS-WI-096, *8260C ICAL Procedure*, current revision.

15.16. TestAmerica Edison SOP No. ED-GEN-022, *Training*, current revision.

15.17. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Practices*, current revision

15.18. TestAmerica Edison Work Instruction Document No. EDS-WI-012, *Client Complaint/Corrective Action Form,* current revision.

15.19. TestAmerica Corporate Quality Memorandum, CA-Q-QM-002, *GC/MS Tuning Policy*, current revision.

15.20. TestAmerica Edison SOP No. ED-GEN-003, *Standard Operating Procedure for Control of Non-Conformances and Corrective Action,* current revision

16.0 Method Modifications:

16.1 Method 8260D requires the BFB tune standard to be analyzed once prior to an ICAL and not daily after that prior to sample analysis. The laboratory will analyze the BFB tune daily, prior to QC and sample analysis. The laboratory will use the tighter criteria from Methods 8260B/8260C for tune evaluation, rather than the criteria suggested in Table 3 of Method 8260D.

17.0 Attachments

N/A

18.0 Revision History

- Revision 8, dated 07/16//2020

    - Updated throughout to include requirements of SW 8260D and 8000D.
    - Updated Table 1 with full current analyte list.
    - Add text to Section 1.1.6 detailing procedures for documenting method variations via NCMs.
    - Section 2.6: clarified text regrading lower than standard RLs.
    - Section 4.4: clarified text regarding trip blanks.
    - Section 7.1.1: revised source and details of organic free water.
    - Section 8.0: added handling and preservation details for various soil and aqueous sample types.
    - Section 9.1.1.4: Added that concentrations allowed in blanks (one half of RL), how blank concentration relates to sample concentration (<1/10) and some guidance on re-analysis when concentration exceeds criteria.
    - Section 9.1.3: added that CCV/LCS can be the same run.
    - Tune verifications as not required for daily CCV updated throughout.
    - Section 9.2.2.1.: Allowance for last calibration standard to be the start of 12-hour clock for samples analyzed after initial calibration. Calculations for verifying peak resolution
    - New Section 9.2.4.2 added: Calibration Point Read Back Criteria
    - Section 11.10: added formula for calculation of Percent Error.
    - Section 12 (Method Performance) updated to include new MDL procedure and annual LLOQ procedure.
    - Updated references in Section 15 as necessary.

- Revision 7, dated 04/01/2020:

    - Updated formatting and branding to Eurofins
    - Sec 7.2.8: Revised Expiration dates based on concentration level and corrected storage requirements.
    - Sec 8.1: Changed Storage Blanks storage period from 1 week to 1-2 weeks.

- Revision 6, dated 01/17/2018:

    - Revised Table 5 and 6: revised to add additional levels plus Benzene and chloroform and to updated concentration level of ICV to current level.

    - 7.2.1 and Table 2: Table in Section 7.2.1 and Table 2 updated to include complete list of standards currently in use as well as to update vendor catalog number for several items.

    - Section 7.2.5: SIM IS/SS mix corrected to reflect lower concentration of IS mix .

    - Section 9.1.2.1.4: SIM MS/MSD preparation revised.

    - Section 9.1.3.2: SIM LCS/LCSD preparation revised.

    - Section 9.2.2.2: additional SIM levels added.

- Section 9.2.3: SIM CCV level concentration revised to reflect lower concentration.
- Section 10.2.5: New SIM analysis preparation narrative added
- Section: 6.1: New instrumentation added.
- Section: Section: 9.1.4.3: Revised to have any one surrogate out without the need for corrective action. This corrects previous narrative of one surrogate out of two.

- Revision 5, dated 12/11//2015:
  - Revised Table 5: new concentration of low standard (1,4-dioxane only).
- Revision 4, dated 12/08//2014:
  - Section 9.2.4.2.2: Table revised to reflect minimum RF of 0.050 for following compounds: acetone, 2-butanone, 4-methyl-2-pentanone, 2-hexanone.
  - Section 9.2.4.3: added statement 'for poor performers the range is 50-150%'.
- Revision 3, dated 11/10/2014:
  - Tables 1 and 7: added 1,2,4,5-Trimethylbenzene, 1,4-Diethylbenzene, Butadiene, 1,4-Difluorobenzene, 1-Chlorohexane, Freon 114, Freon 123a, Isooctane, 4-Ethyltoluene, t-Amyl Alcohol, Chlorofluoroethylene to list of target compounds and list of standard sources.
  - Section 2.5: added chloroform, vinyl chloride and benzene to the list of SIM analytes addressed in this section.
  - Section 2.6: revised the concentration of the low ketone standard to 2.5 ug/l.
  - 7.2.1 and Table 2: Table in Section 7.2.1 and Table 2 updated to include complete list of standards currently in use as well as to update vendor catalog number for several items. All standards prep tables revised to reflect current standard prep instructions.
  - Section 8. Preservation by TSP and holding time is added.
  - Section 9.1.2.1: updated source of standards used in various spiking solutions.
  - Section 9.1.3: LCS/MS/MSD. Preparation tables now indicate using calibration mix and not the second source mix.
  - Sections 9.1.4.3 and 9.1.1 : Revised to indicate that we are now spiking with 4 surrogates instead of the method required 3. One surrogate is now allowed to be out of limit criteria for either 1,2-Dichloroethane-d4 and Dibromofluoromethane.
  - Section 9.2.2: Chloroform, Vinyl Chloride and Benzene added as SIM compounds.

- Section 9.2.4.2.3.1. A list of 'poor performing compounds' is added with a ICAL RSD criteria of 50%.
- Section 9.2.4.3: now specifies that up to 10% of the compounds are allowed to exceed the 70-130% ICV recovery criteria as long as their recoveries are within 65-135%..
- Section 9.2.4.4.1.6: Added the following to the first sentence: '…or 50%D for the poor performing compounds'.
- Section 10.1.1.2: updated masses/dwell time for Group 1 under SIM Parameters.
- Throughout document as appropriate: Replaced references to Target with references to CHROM
- Added Section 10.5.5: "The data processing service from Chrom queries LIMS for the sample processing parameters."

- Revision 2, dated 11/04/2013:
  - Tables 1 and 7: added methyl acrylate, 1-methylnaphthalene and 2-methylnapthalene.
- Revision 1, dated 09/16/2011:
  - Tables 1 and 7: added cyclopentene, 2-chloro-1,3-butadiene, methacrylonitrile, propionitrile, ethyl methacrylate, 2-nitropropane, indan and isobutyl alcohol to list of target compounds and list of standards sources.
  - Section 7.2.1 and Table 2: Table in Section 7.2.1 and Table 2 updated to include complete list of standards currently in use as well as to update vendor catalog number for several items.
  - Table 3: Initial Calibration Standards Preparation: is now split into three tables to include aqueous low level analysis.
  - Table 5: added following footnote:

    Levels 1 and 2 respectively are prepared in 500ml and 100ml final volumes
    [1]This level is also used as the Continuing Calibration Verification.

- Revision 0, dated 02/15/2011: New

Table 2: Working Standards Preparation

Target Compound Standard Name	Lab Name	Vendor	Cat. #	Vol. Std. Added	Conc. of Stock Std.	Concentration of Standard	Final Vol/ Total vol of MeOH
Gas Mix Hi	Gas (Hi)	Restek	567645	5ml mL	2000 ppm	500 ppm	20mL 15mL TV/M
Gas Mix Li	Gas (Li)	Restek	567645	500 uL	2000 ppm	50 ppm	20mL 19.5mL TV/M
8260Mix 1	Mix 1 (Hi)	Restek	567641 567646 567642 568022	2.5ml 2.5 ml 2.5 ml 2.5 ml	2000 ppm	500 ppm	10ml
8260 combined	Mix 1 (Li)	Restek	567641 567646 567642 568022 567643 568018 568713 568722 568723	1.0ml 1.0ml 1.0ml 1.0ml 1.0ml 1.0ml 1.0ml 1.0ml 1.0ml	2000 ppm	50 ppm	40ml 31ml TV/M
Acrolein	AC	Restek	82402	1.0ml	20000 ppm	500 ppm	40ml 39ml TV/M
8260 Mix 2	Mix 2 (Hi)	Restek	567643 568722 568019-fl 568713-fl	2.5ml 2.5 ml 2.5 ml 2.5 ml	2000 ppm	500 ppm	10mL
8260 Mix 3	Mix 3 (Hi)	Restek	568723 568021-fl	2.5ml 2.5ml	2000 ppm	500 ppm	10ml 5ml TV/M
1,4-Dioxane	1,4-Dioxane	Supelco	360481	483.6ul	Neat	50000 ppm	10ml/9.52TVM
1,4-Dioxane	1,4-Dioxane	Supelco	NA	100ul	50000 ppm	500 ppm	10ml/9.90TVM
Propenes*	Propenes	Supelco	21240202	NA	1000/2000 ppm	NA	NA
Propenes*	Propenes	Supelco	21240202	1ml	1000/2000 ppm	50 ppm (varied)	20ml/ 19ml
Gas SS	Gas SS	Restek	567645.sec	1ml	2000ppm	50 ppm	40ml 39ml/TV/M
8260 Mix 1 SIM	8260 Mix 1 SIM	Supelco	5-02111	50 ul	2000ppm	10 ppm	10ml 9.95 TV/M
1,4-Dioxane SIM	1,4-Dioxane	Supelco	NA	100 ul	50000 ppm	500 ppm	10ml/9.90TVM

Table 2: Working Standards Preparation

Target Compound Standard Name	Lab Name	Vendor	Cat. #	Vol. Std. Added	Conc. of Stock Std.	Concentration of Standard	Final Vol/ Total vol of MeOH
8260 SS	8260 SS	Restek	567641.sec 567646.sec 567642.sec 568022- sl 567643.sec 568019- sl 568713- sl 568722.sec 568723.sec 568021- sl	1 ml 1 ml 1 ml 1 ml 1 ml 1 ml 1 ml 1 ml 1 ml 1 ml	2000 ppm	50 ppm	40 ml 30 ml TV/M
Acrolein SS	AC SS	Restek	568720.sec	1 ml	20000 ppm	500 ppm	40 ml 39 ml TV/M
Propenes SS	Propenes SS	Supelco		1 ml	1000/2000 ppm	50/100 ppm	40 ml 39 ml TV/M
8260Mix 1 SIM SS	SIM MIX1 SS	Supelco	5S-02111	50ul	2000 ppm	10 ppm	10ml 9.95 TV/M
Benzene/ Chloroform	Ben/chl	Absolute	70025/ 70076	100ul each	1000ppm	10ppm	10ml 9.90 TV/M
1,4-Dioxane (SS)	1,4-Dioxane	Absolute	70373	1ml	1000 ppm	500 ppm	2ml/1ml TV/M

Asterisk (*) indicates a custom standard mix.

Table 3: Initial Calibration Standards Preparation, Low Level Soil

Standard Solution	Final Volume Reagent Water (ml)	Volume of Standard Added to Reagent Water (ul)					
		1ppb *	5ppb*	20ppb[1]	50ppb	200ppb	500ppb
Gas Mix (50ppm)	5	-	-	2.0	5	-	-
	50	1.0	5.0			-	-
Gas Mix (500ppm)	5	-	-	-		2.0	5.0
		-	-	-			
Mix 1 (combined) (50ppm)	5	-	-	2.0	5	-	-
	50	1.0	5.0			-	-
Mix 1 (Hi) (500ppm)	5	-	-	-	-	2.0	5.0
		-	-	-	-	-	-
Freon Mix							
AC (500ppm)	5	-	-	3.0	4.0	5.0	6.0
	50	10	20			-	-
Mix 2 (Hi) (500ppm)	5	-	-	-	-	2.0	5.0
		-	-	-	-		
Mix 3 (500ppm)	5					2.0	5
Propenes (50ppm)	-	-	-	-	-	-	-
	50	10.0	20.0		-	-	-
Propenes (Hi)(500ppm)	5	-	-	2.0	5.0	20	50
	-	-	-	-	-	-	-

[1]**This level is also used as the Continuing Calibration Verification.**

Table 3a: Initial Calibration Standards Preparation, Aqueous (LOW LEVEL)

Standard Solution	Volume of Standard Added to Reagent Water (ul)						
	0.5ppb*	1ppb*	5ppb*	20ppb[1]	50ppb	200ppb	500ppb
Gas Mix (500ppm)	0.5	1	1	2	5	20	50
Mix 1 (Hi) (500ppm)	0.5	1	1	2	5	20	50
Mix 2 (Hi) (500ppm)	0.5	1	1	2	5	20	50
Mix 3 (varied)	0.5	1	1	2	5	20	50
AC (500ppm)	2	4	4	4	10	20	40
1,4-Dioxane (500ppm)	15	30	-	-	-	-	-
Freons mix	0.5	1	1	2	5	20	50
Propenes (1000/2000ppm)	0.5	0.5	0.5	1	2.5	10	25
Methanol Compensate	3000	2800	610	300	280	190	0
Final vol. (reagent water)	500ml	500 ml	100ml	50 ml	50ml	50ml	50ml

[1]This level is also used as the Continuing Calibration Verification.

Table 3b: Initial Calibration Standards Preparation, Aqueous

Standard Solution	Volume of Standard Added to Reagent Water (ul)					
	1.0ppb*	5ppb*	20ppb[1]	50ppb	200ppb	500ppb
Gas Mix (500ppm)	1	1	2	5	20	50
Mix 1 (Hi) (500ppm)	1	1	2	5	20	50
Mix 2 (Hi) (500ppm)	1	1	2	5	20	50
Mix 3 (varied)	1	1	2	5	20	50
AC (500ppm)	4	4	4	10	20	40
1,4-Dioxane (500ppm)	30	-	-	-	-	-
Freons Mix	1	1	2	5	20	50
Propenes (1000/2000ppm)	0.25	0.5	1	2.5	10	25
Methanol Compensate	2800	610	300	280	190	0
Final vol. (reagent water)	500 ml	100ml	50 ml	50ml	50ml	50ml

[1]This level is also used as the Continuing Calibration Verification.

Table 4 : ICV Standard Preparation, Low Level Soil

Standard Solution	Concentration	Volume of Standard Added to 5.0 ml of Reagent Water (ul)	Final Concentration (ug/L)
GAS SS (Separate lot)	50ppm	2	20
8260 SS (Separate lot)	50ppm (+varied)	2	20
AC SS (separate lot)	500ppm	3	300
Freon SS (Separate lot)	50ppm	2	20
Propenes SS(separate lot)	50ppm (varied)	2	20 (varied)

Table 4a: ICV Standard Preparation, Aqueous

Standard Solution	Concentration	Volume of Standard Added to 50 ml of Reagent Water (ul)	Final Concentration (ug/L)
GAS SS (Separate lot)	50ppm	20	20
8260 SS (Separate lot)	5000ppm (varied)	20	20
AC SS (separate lot)	500ppm	4	400
Freons SS (Separate lot)	50ppm	20	20
Propenes (second source)	50ppm (varied)	20	20 (varied)

Table 5: SIM Initial Calibration Standards Preparation

Standard Solutions	Volume Standard Solution Added to Reagent Water (Final Concentration)							
8260 Mix 1 SIM (10ppm)	1 ul (0.02 ppb)	2 ul (0.04 ppb)	1 ul (0.05 ppb)	1 ul (0.1 ppb)	1 ul (0.2 ppb)	2.5 ul (0.5 ppb)	5 ul (1 .0 ppb)	10 ul (2.0 ppb)
1,4-Dioxane (500ppm)	4 ul (0.4 ppb)	2 ul (1.0 ppb)	1 ul (5.0 ppb)	1 ul (10 ppb)	1 ul (20 ppb)	2.5 ul (30 ppb)	5 ul (40 ppb)	10 ul (50 ppb)
SIM (ben/chl) 10ppm	1 ul (0.02 ppb)	1 ul (0.02 ppb)	1 ul (0.05 ppb	1 ul (0.1 ppb)	1 ul (0.2 ppb)	2.5 ul (0.5 ppb)	5 ul (1.0 ppb)	10 ul (2.0 ppb)
Final Vol. (reagent water)	**500ml**	**500ml**	**200ml**	**100ml**	**50ml**	**50ml**	**50ml**	**50ml**

levels 1 and 2 are respectively prepared in 500ml and 100ml final volumes
[1]**This level is also used as the Continuing Calibration Verification.**

Table 6 : SIM ICV Standard Preparation

Standard Solution	Concentration	Volume of Standard Added to 200 ml of Reagent Water (ul)	Final Concentration (ug/L)
SIM MIX1 SS (Second source)	10ppm	1	0.05
1,4-Dioxane SS	50ppm	20	5

TABLE 7
Characteristic Ions of Volatile Organic Compounds

Parameter	Primary ion	Secondary ion
1,1,1-Trichloroethane	97	99,117,119
1,1,2,2-Tetrachloroethane	83	85,131,133,166
1,1,2-Trichloroethane	97	83,85,99,132,134
1,1-Dichloroethane	63	65,83,85,98,100
1,1-Dichloroethene	96	61,98
1,1-Dichloropropene	75	110. 77
1,2,3-Trichlorobenzene	180	182
1,2,3-Trichloropropane	110	75
1,2,4-Trichlorobenzene	180	182, 145
1,2,4-Trimethylbenzene	105	120
1,2-Dibromo-3-Chloropropane	75	155, 157
1,2-Dibromomethane	107	109
1,2-Dichloroethane	62	64,100,98
1,2-Dichloroethene	96	61,98
1,2-Dichloropropane	63	65,114
1,2-Dichlorotrifluoroethene	67	117
1,2-Difluorotetrachloroethene	101	103, 167
1,3,5-Trimethylbenzene	105	120
1,3-Dichlorobenzene	146	148, 111
1,4-Dichlorobenzene	146	148, 111
1,4-Dioxane	88	58
1-Chloropropane	63	78
1-Methylnaphthalene	142	141
1-Propene	41	42
2,2-Dichloropropane	77	97

TABLE 7 Characteristic Ions of Volatile Organic Compounds		
2,4,4-trimethyl-1-pentene	41	57, 97
2-Butanone	72	57
2-Chloroethyl vinyl ether	63	65, 106
2-Chloropropane	78	63
2-Chlorotoluene	91	126
2-Chloro-1,3-butadiene	88	53
2-Hexanone	43	58,100
2-Methylnaphthalene	142	141, 115
2-Nitropropane	39	42, 44
2-Octane	43	58
2-Octanol	45	55
4-Chlorotoluene	91	126
4-Methyl-2-Pentanone	43	58,100
Methacrylonitrile	67	41
Acetone	43	58
Acetonitrile	39	40, 41
Acrolein	56	55
Acrylonitrile	53	52
Allyl Alcohol	57	40, 39
Allyl Chloride	76	41
Amyl Acetate	43	70, 61
Benzene	78	--
Benzyl Chloride	91	126, 65
Bromobenzene	156	77, 158
Bromochloromethane	129	49, 130
Bromodichloromethane	83	85
Bromoform	173	171,175,
Bromomethane	94	96
Butyl Acetate	73	56, 43
Butyl Acrylate	73	56, 55
Butyl methacrylate	87	69
Camphene	93	121
Camphor	95	81
Carbon disulfide	76	78
Carbon tetrachloride	117	119,121
Chlorobenzene	112	114
Chloroethane	64	66
Chloroform	83	85
Chloromethane	50	52
Chlortrifluoroethene	116	118
cis-1,3-Dichloropropene	75	77

TABLE 7 Characteristic Ions of Volatile Organic Compounds		
Cyclohexane	56	84, 69
Cyclopentene	67	68, 68, 53
Dibromochloromethane	129	208,206
Dibromomethane	93	95, 174
Dichlorodifluoromethane	85	87
Dimethylnaphthalene (total)	141	156, 155
Epichlorohydrin	57	62, 49
Ethanol	46	45
Ethyl Acetate	70	61, 43
Ethyl Acrylate	55	56
Ethyl Ether	59	74, 75
Ethylbenzene	106	91,
Ethyl methacrylate	69	41, 99
Freon TF	101	103, 151, 85
Hexachlorobutadiene	225	223
Hexane	56	57, 86
Indan	117	118, 58
Iodomethane (methyl iodide)	142	127
Isobutyl Alcohol (Isobutanol)	43	41, 42
Isoprene	67	53, 59
Isopropanol	45	59
Isopropyl Acetate	43	61, 87
Isopropyl Ether (DIPE)	45	87
Isopropylbenzene	105	120
Methyl Acetate	43	74
Methyl Acrylate	55	85, 42
Methyl cyclohexane	83	55, 98
Methyl Methacrylate	100	69
Methyl tert-butyl ether (MTBE)	73	57
Methylene chloride	84	49,51,86
Methylnaphthalene (total)	142	141, 115
Naphthalene	128	--
n-Butanol	56	41, 43
n-Butylbenzene	91	92, 134
n-Heptane	57	43, 71
n-Pentane	72	57
N-Propanol	60	59
n-Propylbenzene	91	120
P-Isopropyltoluene`	119	134, 91
Propyl Acetate	43	61, 73
Propionitrile	54	52, 54

TABLE 7 Characteristic Ions of Volatile Organic Compounds		
sec-Butylbenzene	105	134
Styrene	104	78,103
Tert-Amyl Methyl Ether	73	55, 87
Tert-butyl Alcohol	59	--
Tert-Butyl Ethyl Ether	59	87
Tert-Butylbenzene	119	91, 134
Tetrachloroethene	164	129,131,166
Tetrahydrofuran	42	72, 71
Toluene	92	91
Total Xylenes	106	91
trans,-1,3-Dichloropropene	75	77
Trans-1,4-dichloro-2-butene	53	75
Trichloroethene	130	95,97,132
Trichlorofluoromethane	101	103
Vinyl acetate	43	86
Dichlorofluoromethane	67	69
Chlorotrifluoroethene	116	118
1,2-tetrachlorodifluoroethane	101	103,167
1,2-Dichlorotrifluoroethane	67	117
Vinyl chloride	62	64
Isooctane	57	41, 56
1- Chlorohexane	91	93, 55, 56
1,2,4,5-Tetramethylbenzene	119	134, 91
4-EthylToluene	105	120, 77
Chlorotrifluoroethylene	66	116,118,85
Freon 114	85	87,135,137
t-Amyl Alcohol	59	55, 73, 43
1,4-Difluorobenzene	114	63
1,4-Diethylbenzene	119	105,134
Freon 123a	67	69, 117, 119
Butadiene	54	53, 39
4-Bromofluorobenzene (sur)	95	174,176
1,2-Dichloroethane-d4 (sur)	65	102, 104
Toluene-d8 (sur)	98	70,100
Fluorobenzene (istd)	96	77
Chlorobenzene-d5 (istd)	117	82,119
1,4-Dichlorobenzene-d4 (istd)	152	115,150

Title: Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometery (GC/MS), SW846 Methods 8270D and 8270E

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Facility Distribution No. : Electronic **Distributed To: Facility Intranet**

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

USEPA Methods 8270D and 8270E are analytical methods which employ the use of GC/MS to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, and water samples

TestAmerica Edison has the capability to analyze and report the compounds listed in Table 1 via Methods 8270D and 8270E.

Table 1

Compound	CAS No.	Compound	CAS No.
1,1'-Biphenyl	92-52-4	Anthracene **(1)**	120-12-7
1,2,4,5-Tetrachlorobenzene	95-94-3	Atrazine	1912-24-9
1,2,4-Trichlorobenzene	120-82-1	Benzaldehyde	100-52-7
1,2-Dichlorobenzene	95-50-1	Benzidine	92-87-5
1,2-Diphenylhydrazine	122-66-7	Benzo[a]anthracene **(1)**	56-55-3
1,3-Dichlorobenzene	541-73-1	Benzo[a]pyrene **(1)**	50-32-8
1,3-Dimethylnaphthalene	575-41-7	Benzo[b]fluoranthene **(1)**	205-99-2
1,4-Dichlorobenzene	106-46-7	Benzo[g,h,i]perylene **(1)**	191-24-2
1,4-Dichlorobenzene-d4 **(ISTD)**	3855-82-1	Benzo[k]fluoranthene **(1)**	207-08-9
1,4-Dioxane **(1) (2)**	123-91-1	Benzoic acid	65-85-0
1-Methylnaphthalene	90-12-0	Benzyl alcohol	100-51-6
1-Naphthylamine	134-32-7	Bis(2-chloroethoxy)methane	111-91-1
2,2'-oxybis[1-chloropropane]	108-60-1	Bis(2-chloroethyl)ether **(1)**	111-44-4
2,3,4,6-Tetrachlorophenol	58-90-2	Bis(2-ethylhexyl) phthalate	117-81-7
2,3,7,8-TCDD	1746-01-6	Bisphenol-A	80-05-7
2,3-Dihydroindene	496-11-7	Butyl benzyl phthalate	85-68-7
2,3-Dimethylaniline	87-59-2	Caprolactam	105-60-2
2,4,5-Trichlorophenol	95-95-4	Carbamazepine	298-46-4
2,4,5-Trimethylaniline	137-17-7	Carbazole	86-74-8
2,4,6-Tribromophenol **(Surrogate)**	118-79-6	Chrysene **(1)**	218-01-9
2,4,6-Trichlorophenol	88-06-2	Chrysene-d12 **(ISTD)**	1719-03-5
2,4-Dichlorophenol	120-83-2	Coumarin	91-64-5
2,4-Dimethylphenol	105-67-9	Dibenz(a,h)anthracene **(1)**	53-70-3
2,4-Dinitrophenol	51-28-5	Dibenzofuran	132-64-9
2,4-Dinitrotoluene	121-14-2	Diethyl phthalate	84-66-2
2,4-Xylidine	95-68-1	Dimethyl phthalate	131-11-3
2,6-Dinitrotoluene	606-20-2	Di-n-butyl phthalate	84-74-2
2-Chloronaphthalene	91-58-7	Di-n-octyl phthalate	117-84-0
2-Chlorophenol	95-57-8	Fluoranthene **(1)**	206-44-0
2-Ethylaniline	578-54-1	Fluorene **(1)**	86-73-7
2-Fluorobiphenyl **(Surrogate)**	321-60-8	Hexachlorobenzene **(1)**	118-74-1
2-Fluorophenol **(Surrogate)**	367-12-4	Hexachlorobutadiene	87-68-3
2-Methylnaphthalene	91-57-6	Hexachlorocyclopentadiene	77-47-4
2-Methylphenol	95-48-7	Hexachloroethane	67-72-1
2-Naphthylamine	91-59-8	Indeno[1,2,3-cd]pyrene **(1)**	193-39-5
2-Nitroaniline	88-74-4	Isophorone	78-59-1
2-Nitrophenol	88-75-5	n,n'-Dimethylaniline	121-69-7
2-tertbutyl-4-methylphenol	2409-55-4	Naphthalene **(1)**	91-20-3
2-Toluidine	95-53-4	Naphthalene-d8 **(ISTD)**	1146-65-2

Table 1

Compound	CAS No.	Compound	CAS No.
3 & 4 Methylphenol	15831-10-4	n-Decane	124-18-5
3,3'-Dichlorobenzidine	91-94-1	Nitrobenzene	98-95-3
3,4-Dimethylaniline	95-64-7	Nitrobenzene-d5 **(Surrogate)**	4165-60-0
3,5-di-tert-butyl-4-hydroxytol	128-37-0	N-Nitrosodimethylamine **(1)**	62-75-9
3-Nitroaniline	99-09-2	N-Nitrosodi-n-propylamine	621-64-7
4,6-Dinitro-2-methylphenol **(1)**	534-52-1	N-Nitrosodiphenylamine	86-30-6
4-Bromophenyl phenyl ether	101-55-3	n-Octadecane	593-45-3
4-chloro-2-methylaniline	95-69-2	o-Toluidine-d9 **(Surrogate)**	194423-47-7
4-Chloro-3-methylphenol	59-50-7	Pentachloronitrobenzene	82-68-8
4-Chloroaniline	106-47-8	Pentachlorophenol **(1)**	87-86-5
4-Chloroaniline–d4 **(Surrogate)**	191656-33-4	Perylene-d12 **(ISTD)**	1520-96-3
4-Chlorophenyl phenyl ether	7005-72-3	Phenanthrene **(1)**	85-01-8
4-Methylphenol	106-44-5	Phenanthrene-d10 **(ISTD**)	1517-22-2
4-Nitroaniline	100-01-6	Phenol	108-95-2
4-Nitrophenol	100-02-7	Phenol-d5 **(Surrogate)**	4165-62-2
Acenaphthene **(1)**	83-32-9	Phenyl ether	101-84-8
Acenaphthene-d10 **(ISTD)**	15067-26-2	Pyrene **(1)**	129-00-0
Acenaphthylene **(1)**	208-96-8	Pyridine	110-86-1
Acetophenone	98-86-2	Terphenyl-d14 **(Surrogate)**	1718-51-0
Aniline	62-53-3	Total Cresols	STL00160
Aniline-d5 **(Surrogate)**	4165-61-1		

(1) Compound can be analyzed by full scan or Selected Ion Monitoring (SIM).
(2) Compound can also be analyzed by Isotope Dilution/SIM.

1.2 For a listing of method detection limits (MDLs) and Reporting Limits (RLs) please refer to the currently active Method 8270 Method Limit Groups in TALS (TestAmerica LIMS).

1.3 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work)*, and Section 19 (*Test Methods and Method Validation)* in TestAmerica Edison's Quality Assurance Manual (TestAmerica Edison Document No. ED-QA-LQM).

1.4 Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP ED-GEN-003. The NCM shall be filed in the project file and addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

2.0 Summary of Method

2.1 This method is used for the analysis of aqueous and solid matrices for semi-volatile base, neutral and acid organic compounds that are extracted from the sample matrix with an organic solvent.

2.2 An aliquot of sample containing surrogate spiking compounds is extracted with an organic solvent. The extract is concentrated on a steam bath to a suitable volume. Internal standards are added to the extract.

2.3 Sample extraction techniques are specified for each matrix in the following TestAmerica Edison SOPs:

- ED-ORP-002 (*Extraction of Semivolatile Organic Compounds in Water by Separatory Funnel, SW846 Method 3510C*);
- ED-ORP-043 (*SW846 Method 3580A - Waste Dilution Prep for Analysis of BNAs by SW846 Method 8270);*
- ED-ORP-0044 (*Microwave Extraction for Solids, SW846 Method 3546*);

2.4 A small aliquot of the extract is injected into a gas chromatograph (GC) equipped with a capillary column. The GC is temperature programmed to separate the compounds which were recovered during the extraction step by boiling point. The effluent of the gas chromatograph is interfaced to a mass spectrometer (MS) which is used to detect the compounds eluting from the GC. The detected compounds are fragmented with an electron beam to produce a mass spectrum which is characteristic of the compound introduced into the MS. Identification of target analytes is accomplished by comparing their mass spectra with the electron ionization spectra of authentic standards. Quantitation is accomplished by comparing the response of a major ion (quantitation ion) relative to an internal standard established through a five-point calibration (six points for second order regression). Specific calibration and quality control steps are included in the method that must be performed and must meet the specifications of SW846 Methods 8270D or 8270E as applicable.

2.5 The standard preparation procedure for aqueous samples involves use of a Reduced Volume Extraction (250 ml) (RVE) followed by analysis using a Large Volume Injection (LVI). Optionally, a full volume (1000 ml nominal) may be employed. The details of the extractions are outlined in the applicable prep SOPs while the analytical details for 8270D and 8270E are presented in this SOP.

2.6 These methods are also applicable to the analysis of samples by Selected Ion Monitoring (SIM) for the purpose of obtaining lower reporting limits for the following compounds:

Table 2 – SIM Analytes

SIM Analytes	CAS #
1,4-Dioxane	123-91-1
4,6-Dinitro-2-methylphenol	534-52-1
Acenaphthene	83-32-9

Table 2 – SIM Analytes	
SIM Analytes	**CAS #**
Acenaphthylene	208-96-8
Anthracene	120-12-7
Benzo[a]anthracene	56-55-3
Benzo[a]pyrene	50-32-8
Benzo[b]fluoranthene	205-99-2
Benzo[g,h,i]perylene	191-24-2
Benzo[k]fluoranthene	207-08-9
Bis(2-chloroethyl)ether	111-44-4
Chrysene	218-01-9
Dibenz(a,h)anthracene	53-70-3
Fluoranthene	206-44-0
Fluorene	86-73-7
Hexachlorobenzene	118-74-1
Indeno[1,2,3-cd]pyrene	193-39-5
Naphthalene	91-20-3
N-Nitrosodimethylamine	62-75-9
Pentachlorophenol	87-86-5
Phenanthrene	85-01-8
Pyrene	129-00-0

2.7 An isotope dilution selected ion monitoring (SIM) technique for the analysis of 1,4-dioxane in water at a reporting level of 0.4 ug/l is also described in this SOP. Using this technique 1,4-dioxane-d8 is added prior to sample extraction and is used as an internal standard to calculate the concentration of 1,4-dioxane present. Additionally, 1,4-dichorobenzene-d4 is added to the extract prior to analysis to monitor the recovery of 1,4-dioxane-d8.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of the Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Analysts must take steps to determine the source of the interference and take corrective action to eliminate the problem.

4.1.1 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce

carryover, the sample syringe is automatically rinsed with solvent between sample injections. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of a solvent blank to check for cross-contamination. Alternately, verify that the sample analyzed after the high concentration sample does not show any carryover through inspection of chromatogram and target results.

4.1.2 Contaminants from the extraction process detected in the method blank should be evaluated to determine the impact on the analysis. Interferences from any target analyte must not be present in the method blank above the reporting limit for that compound. If these types of interferences occur, corrective action is required. The source should be identified and corrective action initiated to eliminate the interference from the extraction process. Affected samples must be re-extracted and re-analyzed.

4.1.3 The analyst must take precautions to make sure that contaminants do not enter the analytical system. These precautions include systematic procedures designed to eliminate interferences.

4.2 Some compounds analyzed by this method are unstable or sensitive to extraction and/or instrument conditions:

- Benzidine is easily oxidized during extraction. Neutral extraction may enhance the recovery of this compound.
- Hexachlorocyclopentadiene breaks down photochemically and can decompose from high temperatures, particularly in the injection port of the GC. This compound can also react with acetone in solution.
- 1,2-Diphenylhydrazine is unstable even at room temperature and readily converts to azobenzene.
- Phenols are sensitive to active sites and can give a low response or exhibit poor chromatography by tailing. Therefore, it is important the GC is maintained in the best possible condition. See Section 10.1 for proper daily maintenance.
- N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.
- 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method. They are reported as 3 and 4-methylphenol.
- Pyridine may perform poorly at the GC injection port temperatures listed in this SOP. Lowering the injection port temperature may reduce the amount of degradation.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

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 MSDS 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 MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
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.
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.
Dimethyl-dichloro-silane	Flammable	none	Can be corrosive to the respiratory tract causing severe irritation and tissue damage. Harmful if absorbed through the skin. May cause severe irritation and systemic damage. Severely irritating to the skin and eyes. Harmful if swallowed. Can cause abdominal discomfort, nausea, vomiting, diarrhea, and irritation to the mouth, throat and stomach.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Gas chromatograph/mass spectrometer system

6.1.1 Gas chromatograph: An Agilent/HP 6890/7890/900 Intuvo (or equivalent) houses the capillary column. The GC provides a splitless injection port and allows the column to be directly coupled to the mass spectrometer. The oven is temperature programmable to meet the requirements of the method. An HP/Agilent 7673/7683/7963 autosampler (or equivalent) with a 10 ul syringe provides automatic injection of sample extracts while the instrument is unattended.

6.1.2 Analytical Column: 30m x 0.25mm ID, 0.25 um film thickness, Restek Rxi-5Sil MS, Catalog #13623

6.1.3 Mass spectrometer: Agilent (HP) 5972, 5973, 5975 or 5977A Mass Selective Detector (MSD) Capable of scanning from 35 to 500 amu every 1 sec or less, using 70 volts electron energy in the electron ionization mode. The mass spectrometer must be capable of producing a mass spectrum for 50 ng of decafluorotriphenylphosphine (DFTPP) which meets the criteria in Section 9.2.1 when 2 ul of the 25 ug/ml GC/MS tuning standard is injected through the GC.

6.1.4 GC/MS interface: Any GC-to-MS interface may be used that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria.

6.1.5 Data system: The data system is interfaced to the mass spectrometer and accommodates continuous acquisition and storage of GC/MS data throughout the duration of the chromatographic program. The data system consists of a Hewlett-Packard Chemstation equipped with Mustang software used for instrument control and data acquisition. This, in turn, is interfaced to TestAmerica's Chrom software for data processing. Data from sample extract analysis can be accessed in real-time, while sample data reports and library searches can be performed on data files from previously run samples. The software is also capable of searching any GC/MS data file for ions of a specific mass whose abundances can be plotted versus time or scan number which allows integration of abundances for any extracted ion between specified times or scan-number limits. Library searches utilize a NIST 02.1 Mass Spectral Library.

6.2 Bottles, glass with polytetrafluoroethylene (PTFE)-lined screw caps or crimp tops.

6.3 Injection port liners, splitless

6.4 Injection port septa

6.5 Injection port graphite seals

6.6 Pre-silanized glass wool (Supelco 2-0411 or equivalent)

6.7 Syringes, Assorted sizes 10ul - 1000ul; gas-tight

6.8 Bottles, 10 and 5ml amber screw cap with Teflon liner

6.9 Vials, 2ml amber screw cap with Teflon liner

6.10 Wheaton microvials 100ul (or equivalent)

6.11 Volumetric Flasks, Class A with ground glass stoppers (2ml - 100ml)

6.12 Analytical balance, ASP Model SP-180 (or equivalent), capable of accurately weighing to 0.0001 gr.

7.0 Reagents and Standards

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met. Please refer to the MSDS prior to the use of any reagent or standard.

The preparation of standards, surrogates and spiking solutions is documented in the TALS Reagent Module. Formulary reports can be generated upon request.

7.1. Reagents:

7.1.1. Methylene Chloride: J.T.Baker Resi-Analyzed, used for Organic Residue Analysis (P/N 9266-V8 or equivalent).

7.1.2. Methanol: J.T.Baker Purge and Trap Grade (P/N 9077-02 or equivalent).

7.1.3. Sylon-CT: Supelco (P/N 33065-U or equivalent). Sylon-CT is a highly reactive silanizing reagent consisting of 95% Toluene and 5% Dimethyldichlorosilane (DMDCS).

7.1.4. Each lot of solvent is screened for contaminants before being used for analysis as detailed in TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7.2. Standards:

7.2.1. **Calibration Standards (Full Scan Analysis)**: Stock analytical standard solutions are purchased mainly from Restek Corporation. Other standards are prepared in the laboratory as needed using neat compounds or prepared solutions purchased from Agilent, SPEX CertiPrep, Chem Service,

Accustandard, Supelco or other suppliers. Standards prep instructions are detailed for the following full scan analyte list options:

- Full Volume Aqueous Prep; and,
- Reduced Volume Aqueous Prep and Soils

Secondary dilutions are either made from purchased stock solutions as listed below or from prepared solutions as listed in the following table:

NOTE: Second sources (from certified separate lots) are used for ICV standards.

Table 3 –
Full Scan Stock Standards

Target Analyte Standard Name	Conc. (PPM)	Vendor	Catalog #
1,2,3,4-TCDD	50	SPEX	SVO-TANJ-12
Agilent Mix (contains compounds listed in Table 4 below)	2000 *	Agilent	Cus 0456
8270 List 1/ Std #1 Megamix	Varied	Restek	571995
8270 List 1/ Std#9	2000	Restek	569730
8270 List 1/ Std#11	2000	Restek	569732
8270 Surrogate Standard	5000*	Restek	567685
8270 Internal Standard	2000	Restek	567684
8270 List 1/ Std#10	2000	Restek	569731
Bisphenol-A	1000	Agilent	Cus-0457

*Agilent Mix, 8270 list1/std#9 and 8270 Surrogate standard are diluted to 100ppm prior to the preparation of the 1.0ppm and 0.5ppm standards.

Table 4
Agilent Mix
Catalog No. Cus-0456

Analyte	Concentration (PPM)
Pentachloronitrobenzene	2000
2 -tert-butyl-4-Methylphenol	2000
2,6-Di-tert-butyl-4-Methylphenol	2000
Coumarin	2000
Phenyl ether	2000
N,N'-Dimethylaniline	2000
N-Methylaniline	2000
Carbamazepine	2000
Benzonitrile	2000
1,3-Dimethylnaphthalene	2000

7.2.1.1. Individual calibration standards for full scan analysis are prepared in one of several ways depending upon the technique (full volume aqueous prep, soils prep, reduced volume prep with LVI) as well as the target analyte list. The following tables detail the preparation of calibration standard solutions for each of these techniques and analyte lists. Prepare by combining the indicated volumes of each stock solution using volumetric flask. Dilute to the volume marker with methylene chloride.

Table 5
Full Volume Aqueous Prep and Soils
Working Standards Preparation

Solution Name	120 PPM	80 PPM	50 PPM	20 PPM	10 PPM	5 PPM	2 PPM	1 PPM	0.5 PPM
8270 List 1/ Std #1 Megamix	3000 ul	2000 ul	2500 ul	500ul	250ul	250ul	100ul	50ul	25ul
8270 List 1/ Std #9	1500 ul	1000 ul	1250 ul	250ul	125ul	125ul	50ul	500ul*	250ul *
8270 List 1/ Std #10	1500 ul	1000 ul	1250 ul	250ul	125ul	125ul	-	-	-
Agilent custom Mix	1500 ul	1000 ul	1250 ul	250ul	125ul	125ul	50ul	500ul*	250ul *
1,2,3,4-TCDD	-	-	500ul	-	-	-	-	-	-
8270 Surrogate Standard	600ul	400ul	500ul	100ul	50ul	50ul	20ul	500ul*	250ul *
8270 Internal Standard	500ul	500ul	1000 ul	500ul	500ul	1000 ul	1000 ul	1000 ul	1000 ul
Bisphenol-A	3000 ul	2000 ul	2500 ul	500ul	250ul	250ul	100ul	-	-
8270 List 1/ Std #11	400ul	300ul	500ul	200ul	125ul	125ul	50ul	25ul	-
Final Volume (ml)	**25**	**25**	**50**	**25**	**25**	**50**	**50**	**50**	**50**

Note: The 1.0ppm and 0.5pmm standards (above) are prepared using the 100ug/ml standard for Agilent custom Mix, 8270 List1/std#9 and 8270 Surrogate Standard.

Table 6
Reduced Volume Extraction/LVI
Working Standards Preparation

Solution Name	24 PPM	16 PPM	10 PPM	4 PPM	2 PPM	1 PPM	0.4 PPM	0.2 PPM	0.1 PPM
120 ppm (see Table 5)	2.0mL								
80 ppm (see Table 5)		2.0 mL							
50 ppm (see Table 5)			2.0 mL						
20 ppm (see Table 5)				2.0 mL					

Table 6 Reduced Volume Extraction/LVI Working Standards Preparation									
Solution Name	**24 PPM**	**16 PPM**	**10 PPM**	**4 PPM**	**2 PPM**	**1 PPM**	**0.4 PPM**	**0.2 PPM**	**0.1 PPM**
10 ppm (see Table 5)					2.0 mL				
5.0 ppm (see Table 5)						2.0 mL			
2.0 ppm (see Table 5)							2.0mL		
1.0 ppm (see Table 5)								2.0 mL	
0.5 ppm (see Table 5)									2.0mL
Final Volume (ml)	**10**	**10**	**10**	**10**	**10**	**10**	**10**	**10**	**10**

7.2.1.2. **Initial Calibration Verification (full scan)**: Second source ICVs for full scan analysis are prepared in one of several ways depending upon the technique (full volume aqueous prep, soils prep, reduced volume prep with LVI) as well as the target analyte list. The following tables detail the preparation of ICVs for each of these techniques and analyte lists. Prepare by combining the indicated volumes of each stock solution using volumetric flask. Dilute to the volume marker with methylene chloride.

Table 7 8270/625 ICV Working Standards Preparation	
Solution Name	**25 PPM**
8270 List 1/ Std #1 Megamix (2nd Lot)	250ul
8270 List 1/ Std #9 (2nd Lot)	125ul
8270 List 1/ Std #10 (2nd Lot)	125ul
Agilent custom Mix (2nd Lot)	125ul
8270 Internal Standard	200ul
8270 List 1/ Std#11	125ul
Bisphenol-A (2nd Lot)	250ul
Final Volume (ml)	**10**

7.2.1.3. **Surrogate Standards (Full Scan Analysis):** A 5000ppm Surrogate Standard is purchased from Restek for use in spiking blanks, samples and associated QC prior to extraction (reference the applicable sample prep SOPs for spiking instructions).

Table 8 Full Scan Surrogate Standards Solution Restek Catalog No. 567685	
Surrogate Standard Compounds	**Concentration (PPM)**
Nitrobenzene-d5	5000
p-Terphenyl-d14	5000
2,4,6-Tribromophenol	5000
Phenol-d5	5000
2-Fluorobiphenyl	5000
2-Fluorophenol	5000

7.2.1.4. **Internal Standards** (**Full Scan Analysis**): The Internal Standards Solution at 2000ppm is purchased from Restek (Catalog # 567684). The Internal Standard solution is stored in 10ml amber screw cap bottles with Teflon liners in the dark at 4°C. The Internal standard solution is used in preparing all analytical standards. Inject 20ul of this solution (2000ppm) per ml of sample extract prior to analysis resulting in a concentration of 40ppm (ug/ml) in the extract.

Table 9 Full Scan Internal Standards Solution Restek Catalog No. 567684	
Internal Standard Compounds	**Concentration (PPM)**
1,4-Dichlorobenzene-d4	2000
Phenanthrene-d10	2000
Naphthalene-d8	2000
Chrysene-d12	2000
Acenaphthene-d10	2000
Perylene-d12	2000

7.2.2. **Calibration Standards (SIM analysis)**: The Edison lab currently analyzes only a select list of compounds by 8270D/8270E SIM (see Sections 1.0 and 2.0). Stock analytical SIM standard solutions are purchased mainly from Accustandard. Working standards are prepared from these solutions as listed in the tables in Section 7.2.2.1:

Table 10 Stock SIM Standards			
Standard Name	**Concentration**	**Vendor**	**Catalog #**
Pentachlorophenol	100ppm	Accustandard	App-9-176
n-Nitrosodimethylamine	100ppm	Accustandard	APP-9-149
Hexachlorobenzene	100ppm*	Accustandard	APP-9-112
PAH Mix	100ppm	Accustandard	M-610
Bis(2-chloroethyl)ether	100ppm*	Accustandard	App-9-027
4,6-Dinitro-2-methylphenol	100ppm	Accustandard	P-3845
1,4-Dioxane	1000ppm**	Accustandard	APP-9-096

*Hexachlorobenzene and Bis(2-chloroethyl)ether are diluted to 10ppm prior to SIM Standards prep

** 1,4-Dioxane is diluted (10x) to 100ppm prior to SIM Standards prep

NOTE: Second sources (from separate lots are used for ICV standards).

7.2.2.1 Individual calibration standards for SIM analysis are prepared in one of two ways depending upon the technique (full volume aqueous prep or reduced volume prep with LVI) as well as the target analyte list. The following tables detail the preparation of calibration standard solutions for each of these techniques and analyte lists. Prepare by combining the indicated volumes of each stock solution using volumetric flask. Dilute to the volume marker with methylene chloride.

Table 11
Full Volume Aqueous Prep – SIM
Working Standards Preparation

	0.025 PPM	0.05 PPM	0.1 PPM	0.5 PPM	1.0 PPM	5.0 PPM
Pentachlorophenol	10uL	25uL	50uL	50uL	100uL	250uL
n-Nitrosodimethylamine	10uL	25uL	50uL	50uL	100uL	250uL
PAH mix	2.5uL	5uL	100uL	25uL	50uL	100uL
Hexachlorobenzene	10uL	25uL	100uL	500uL	1000uL	2500uL
Bis(2-chloroethyl)ether	10uL	25uL	100uL	500uL	1000uL	250uL*
4,6-dinitro-2-methylphenol	50ul	100ul	200ul	200ul	250ul	500ul
1,4-Dioxane	20ul	50ul	100ul	100ul	200ul	500ul
ISTD	200uL	200uL	200uL	100uL	100uL	100uL
Final Volume (ml)	**10**	**10**	**10**	**5**	**5**	**5**

*For Bis(2-chloroethyl)ether the 5.0 ppm level is prepared using the 100ppm standard.

Table 12
Reduced Volume Extraction/LVI – SIM
Working Standards Preparation

	0.005 PPM	0.01 PPM	0.02 PPM	0.10 PPM	0.20 PPM	1.0 PPM
0.025 PPM Std (see Table 11)	1.0 mL					
0.05 PPM Std (see Table 11)		1.0 mL				
0.1 PPM Std (see Table 11)			1.0 mL			
0.5 PPM Std (see Table 11)				1.0 mL		
1.0 PPM Std (see Table 11)					1.0 mL	
5.0 PPM Std (see Table 11)						1.0 mL
Final Volume (ml)	**5**	**5**	**5**	**5**	**5**	**5**

7.2.2.2 Initial Calibration Verification (SIM): A 0.1 ppm separate lot SIM ICV is prepared as detailed in Table 13 using the stock standards detailed in Section 7.2.2 (above)

Table 13 0.1ppm SIM ICV preparation	
Pentachlorophenol	25uL
n-Nitrosodimethylamine	25uL
PAH mix	5uL
Hexachlorobenzene	5uL
1,4-Dioxane	5ul
4,6-Dinitro-2-methylphenol	100ul
ISTD	**100uL**
Final Volume	**5 ml**

7.2.2.3 Internal Standard solution (SIM): A 50 ppm Internal Standard solution for SIM analysis is prepared by adding 125ul of the 2000ppm stock ISTD (see Section 7.2.1.4) and bringing to volume with Methylene Chloride in a 5ml volumetric flask.

7.2.2.3.1 For SIM analysis inject 20ul of this solution (50ppm) per ml of sample extract prior to analysis resulting in a concentration of 1ppm (ug/ml) in the extract.

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7.2.3. Calibration Standards (Isotope Dilution SIM – 1,4-Dioxane): The Edison lab currently analyzes only for 1,4-dioxane by 8270D/8270E isotope dilution SIM (see Sections 1.0 and 2.0). Stock analytical isotope dilution SIM standard solutions are purchased mainly from Accustandard and Restek. Working standards are prepared from these solutions as listed in the tables below.

Table 14 - Stock 1,4-Dioxane Isotope Dilution SIM Standards			
Standard Name	**Concentration**	**Vendor**	**Catalog #**
1,4-Dioxane	1000ppm*	Accustandard	APP-9-096

* 1,4-Dioxane is diluted (10x) to 100ppm prior to SIM Standards prep

Table 15 - Stock Labeled 1,4-Dioxane SIM Surrogate/Internal Standard (added at prep)			
Standard Name	**Concentration**	**Vendor**	**Catalog #**
1,4-Dioxane-d8	2000ppm	Restek	30614

Table 16 - Stock 1,4-Dioxane Isotope Dilution SIM Internal Standard (added to extract)			
Standard Name	**Concentration**	**Vendor**	**Catalog #**
1,4-Dichlorobenzene-d4	2000ppm	Accustandard	AZ-014J-3

7.2.3.1 Individual calibration standards for 1,4-dioxane isotope dilution SIM analysis are prepared at the concentrations detailed in the following tables. Prepare by combining the appropriate volumes of each stock solution using volumetric flask. Dilute to the volume marker with methylene chloride.

Table 17
Reduced Volume Extraction/LVI – 1,4-Dioxane Isotope Dilution SIM
ICAL Standard Concentrations (ug/ml)

	Lev 1	Lev 2	Lev 3	Lev 4	Lev 5	Lev 6	Lev 7	Lev 8	ICV*
1,4-Dioxane	0.02	0.04	0.1	0.2	0.5	1	2	10	0.2
1,4-Dioxane-d8	4	4	4	4	4	4	4	4	4
1,4-Dichlorobenzene-d4	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2

***:** The ICV is prepared from the second source stock in Table 13.

7.2.4. GC/MS Instrument Performance Check (DFTPP): The DFTPP standard is prepared by is prepared at 25 ppm by adding 2.5ml of EPA 8270 GC/MS Tuning Solution II (Restek Catalog # 31615) to a 100ml volumetric flask and bringing to volume with Methylene Chloride.

7.2.5. Information on prepared standard solutions must be recorded in the TALS Reagent Module. Information such as standard supplier, lot number, original concentration, a description of how the standard was made, are required along with the laboratory lot number, analyst's initials, date prepared, expiration date and verification signature. Standards must be remade every 6 months, or sooner, if the standards expire or begin to show signs of unacceptable degradation. Class "A" volumetric must be used at all times and syringes, preferably gas-tight syringes when available, should be checked for accuracy using an analytical balance. Class "A" pipettes should also be used if volumes permit.

7.2.6. Please refer to TestAmerica Edison SOP No. ED-GEN-008, Standard Operating Procedure for Preparation, Purity and storage of Reagents and Standards.

- Shelf Life of Standard: 1 year after preparation or stock standard manufacture expiration, whichever comes first;
- Storage Requirements: Stock standards are stored at 4℃ and Working Standards stored at -10℃ to -20℃.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 All samples must be stored at 4ºC (± 2ºC) upon receipt.

8.2 Sample Extract Storage. Samples extracts must be protected from light and refrigerated at 4ºC (± 2ºC) from time of extraction until analysis.

8.3 Sample Extract Holding Time. All sample extracts must be analyzed within 40 days of extraction.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Amber glass, 1L	1000 ml or 250 ml (1)	Cool 4 ± 2°C	7 days to extraction; Analyze within 40 days of extraction	EPA Method SW846 8270D/8270E
Solids	Wide mouth glass, 8 or 16 oz.	50g	Cool 4 ± 2°C	14 days to extraction; Analyze within 40 days of extraction	EPA Method SW846 8270D/8270E

(1) : Reduced volume extraction (RVE) LVI option

9.0 Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]
Internal Standards	Every sample	Response within -50% to +100% of CCV

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD, Method Blank)
[4] Statistical control limits are determined annually and are updated into TALS limit group..

9.1.1. **Method blanks** are extracted with every sample batch on each day that samples are extracted. To be considered acceptable, the method blank must contain less than the reporting limit of all target compounds except for phthalates, which can be present at up to 5x the MDL. For method 8270E

the method blank is generally acceptable if target analyte concentrations are less than the one half the reporting limit.

If method blanks are unacceptably contaminated with target compounds that are also present in field samples, all affected samples must be re-extracted and re-analyzed. Corrective action must be taken to identify and eliminate the contamination source. Demonstrate that acceptable blanks can be obtained before continuing with sample extraction and analysis. Method blanks must be analyzed on each instrument on which the associated samples are analyzed.

9.1.1.1. Surrogate recoveries for the method blank are compared to laboratory generated limits. If two or more surrogates for any one fraction (base-neutral or acid) are outside of recovery limits or if any one surrogate recovers at <10%, the sample must be re-extracted and re-analyzed to confirm matrix interference.. If any surrogate is still outside limits, all samples and QC samples associated with that method blank must be re-extracted (volume permitting).

9.1.2. Matrix Spike (MS)/Matrix Spike Duplicate (MSD): A matrix spike/matrix spike duplicate (MS/MSD) pair is extracted and analyzed with every 20 environmental samples of a specific matrix (defined as a sample batch). Full compound list spiking is employed for MS/MSDs and LCSs. These spikes are prepared and extracted concurrent with sample preparation. MS and MSD recoveries are calculated and compared to lab generated acceptance criteria. See the current active TALS 8270 Method Limit Group for QC limits. The MS/MSD spiking solution should the same as used for the calibration standards.

9.1.2.1 A Laboratory Control Sample Duplicate (LCSD) is extracted and analyzed only when insufficient client sample is available for preparation of an MS/MSD pair. The LCS/LSCD is evaluated in the same manner as the MS/MSD (see Section 9.1.2)

9.1.2.2 An LCS/LCSD may be substituted for the MS/MSD if insufficient sample volume is available.

9.1.3. Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD): A Laboratory Control Sample (LCS) (aka blank spike) must be extracted and analyzed with each batch of 20 environmental samples. The LCS data is used to assess method performance if the MS/MSD recoveries fall outside of the lab generated limits (See the current active TALS 8270 Method Limit Group for QC limits). If the LCS recovery is within the current lab generated limits, the MS/MSD recoveries are attributed to matrix interference.

9.1.3.1 A Laboratory Control Sample Duplicate (LCSD) is extracted and analyzed only when insufficient client sample is available for preparation of an MS/MSD pair. The LCS/LSCD is evaluated in the same manner as the MS/MSD (see Section 9.1.2)

9.1.3.2 Spike recovery limits are lab generated and are updated annually. Certain state regulatory programs have defined recovery limits which, where applicable, are used for spike recovery evaluations. The TALS Method Limit Groups detail these regulatory program criteria.

9.1.4. Surrogate Standards: All full scan samples, blanks and QC samples are spiked with a six (6) component surrogate standard mix (see Section 7.2.1.3). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (See the current active TALS 8270 Method Limit Group for QC limits*)*.

If any two or more surrogates for any one fraction (base-neutral or acid) are outside of recovery limits or if any one surrogate recovers at <10%, the sample must be re-extracted and re-analyzed to confirm matrix interference. If a surrogate is diluted to a concentration below that of the lowest calibration standard, no corrective action is necessary.

9.1.4.1 Surrogate recovery limits are lab generated and are updated annually. Certain state regulatory programs have defined recovery limits which, where applicable, are used for spike recovery evaluations. The TALS Method Limit Groups detail these regulatory program criteria.

9.1.5. Internal Standards: The response (area count) of each internal standard in the sample must be within -50 +100% of its corresponding internal standard in the CCV or, the ICAL midpoint for samples analyzed under the initial calibration range. Failure to meet these criteria is indicative of sample matrix effects. All samples failing these criteria must be reanalyzed to confirm matrix effects.

9.2. Instrument QC

9.2.1 GC/MS Instrument Performance Check (DFTPP): (**Note**: the DFTPP performance check applies only to full scan analyses and is not evaluated for SIM analysis). The GC/MS system is tuned using Perfluortributylamine (PFTBA) such that an injection of 50ng of Decafluorotriphenylphosphine (DFTPP) meet the abundance criteria listed in the table below. Prior to the analysis of any calibration standards or samples, the GC/MS system must meet all DFTPP key ion abundance criteria. This analysis will verify proper tuning of the system for a period of 12 hours post-injection. After 12 hours, the instrument performance must again be verified prior to the analysis of standards, QC or samples. Daily tune verification is not required for 8270E CCV.

DFTPP Key Ions and Abundance Criteria	
Mass	**Ion Abundance Criteria**
51	30-60% of mass 198
68	<2% of mass 69
69	reference only
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base Peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	present but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

9.2.1.1. Evaluate DFTPP using three scan averaging and background subtraction techniques. Select the scan at the peak apex, add +1 scan from the apex and -1 scans from the apex.

9.2.1.2. The mass spectrum of DFTPP may be background subtracted to eliminate column bleed or instrument background ions. Background subtract DFTPP by selecting a scan for subtraction ≤20 scans before the apex scan of DFTPP.

9.2.1.3. Check column performance using pentachlorophenol and the benzidine peaks (these compounds are included in the DFTPP solution). Benzidine & Pentachlorophenol should respond normally without significant peak tailing (Tailing Factor should be <2 measured at 10% peak height). If responses are poor and excessive peak tailing is present, corrective action for the GC/MS instrument may be required. Corrective actions may include:

9.2.1.3.1 Retune the GC/MS;
9.2.1.3.2 Clip the injector end of the GC column;
9.2.1.3.3 Replace the septum and injection port liner;
9.2.1.3.4 Change the injection port seal;
9.2.1.3.5 Replace the GC column;
9.2.1.3.6 Clean the injection port with MeCl2
9.2.1.3.7 Clean the MS ion source;
9.2.1.3.8 Place a service call.

9.2.1.4. The breakdown of 4, 4-DDT into 4,4-DDD and 4,4'DDE may also be used to assess GC column performance and injection port inertness. If so evaluated the breakdown must be <20%.

9.2.1.5. DFTPP parameter settings are stored in a tune file, which will be used in all subsequent analysis of standards and sample extracts.

9.2.2 Initial Calibration Range and Initial Calibration Verification

9.2.2.1. Initial Calibration: The initial calibration range consists of a minimum of five concentration levels of analytical standards (six for second order regression) prepared as described in Section 7.2. and analyzed once the DFTPP instrument performance check has met the criteria in Section 9.2.1. .

9.2.2.2. Initial Calibration Verification (ICV): An Initial Calibration Verification (ICV) standard is analyzed immediately after the Initial Calibration Range and before any samples are analyzed. The ICV is prepared as detailed in Section 7.2. The ICV must be from a source (or lot) separate from the standards used in the Initial Calibration Range.

9.2.3 Continuing Calibration Verification (CCV) and Low Level Continuing Calibration Verification (LLCCV): A mid-point Continuing Calibration Verification (CCV) must be analyzed every 12 hours after the DFTPP instrument performance check (when applicable).. The CCV is prepared as detailed in Section 7.2. (typically, 50 ug/ml for full volume aqueous and soils, 10 ug/ml for LV, 0.02 ug/ml for LVI SIM) and 0.2 for isotope dilution SIM). Additionally a Low Level Continuing Calibration Verification (LLCCV) is analyzed after the CCV for full scan analysis. The LLCCV is the same as the lowest calibration level analyzed with the initial calibration range (See Section 7.2).

9.2.4 Calibration Acceptance Summary

9.2.4.1. Retention Time Windows: Retention time windows must be established to compensate for minor shifts in absolute retention times as a result of sample loading and normal chromatographic variability. Obtain the retention time for all compounds from the analysis of the midpoint standard for the calibration curve. Establish the center of the retention time window by using the absolute retention time for each analyte, internal standard and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration. For qualitative identification to be acceptable the retention time of the relative retention time (automatically calculated in Chrom) must be within 0.8 - 1.2 RRT units of its assigned internal standard. The relative retention times of each compound in the five calibration standards must agree within .06 relative retention time units.

9.2.4.2. Initial Calibration Range: Internal standard calibration is employed for this method. After the initial calibration range has been analyzed the relative response factor (RRF) for each target/surrogate compound at each concentration level is determined using the following equation.

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:
A_x = Area characteristic ion (see Table 21) for the compound
Ais = Area characteristic ion (see Table 21) of associated internal standard
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

9.2.4.2.1. Determine the mean RRF for each compound. Minimum response factors must be met for each of the compounds listed in Table 18 (below). Any compound that fails the minimum response factor must be reported as estimated for detects and must have a demonstration of sensitivity in the analytical batch to report non-detects. To demonstrate adequate sensitivity for out of criterion compounds analyze the low level point of the calibration (LLCCV) in the analytical sequence. The criterion for the LLCCV is detection only but the standard qualitative identification criteria in the method must be met.

Table 18: Minimum Response Factors

Compound	Minimum Response Factor
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl) ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2-Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-Methylphenol	0.600
N-Nitroso-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700

Table 18: Minimum Response Factors	
Compound	**Minimum Response Factor**
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800
2-Nitroaniline	0.010
Dimethyl phthalene	0.010
2,6-Dinitrotoluene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Diethyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010
4-chlorophenyl-phenyl ether	0.400
Fluorene	0.900
4-Nitroanailine	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenyl ether	0.100
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butyl phthalene	0.010
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3'-Dichlorobenzidine	0.010
Benzo(a)anthracene	0.800
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700

Table 18: Minimum Response Factors	
Compound	**Minimum Response Factor**
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010
Pentachloronitrobenzene	0.050

9.2.4.2.2. Calculate the Standard Deviation (SD) and Percent Relative Standard Deviation (% RSD) of the response factors for each compound:

$$\% \text{ RSD} = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

9.2.4.2.3. The % RSD of the RRF's must be ≤20% for each target analyte listed in Table 18. The % RSD of each target analytes must be ≤20% in order for the calibration range to be acceptable. Additionally for 8270E, the calculated concentration or amount of each analyte of interest in the CCV standard should fall within ±20%. If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit or do not meet the minimum correlation coefficient (0.99) for alternate fits (see below) then appropriate corrective maintenance action must be performed. If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit **AND** do not meet the minimum correlation coefficient (0.99) then recalibration is necessary.

9.2.4.2.4. If the above listed criteria is met, the system can be assumed to be linear and sample analysis may begin and the average RF from the initial calibration range is used to quantitate all samples.

9.2.4.2.4.1 Certain state regulatory programs have defined calibration acceptance limits which, where applicable, are used for calibration evaluations. The TALS ICAL Limit Groups detail these regulatory program criteria.

9.2.4.2.5. An alternative calibration technique may be employed for those any compounds exceeding the 20% RSD criteria:

9.2.4.2.5.1 Calculate the first order linear regression for any compound which did not meet the 20% criteria. First order linear regression calibration may be employed if alternative average response calibration procedures were not applicable. The r value (Correlation Coefficient) of the equation must be ≥0.99 for the calibration to be employed.

9.2.4.2.5.2 Second order regression calibration can be used for any compound that has an established history as a non-linear performer.

9.2.4.2.5.3 If second order regression calibration is used a minimum of six (6) calibration levels must be analyzed.

9.2.4.2.5.4 If second order regression calibration is used, the r^2 (Correlation Coefficient) value must be $\geq$ 0.99

9.2.4.2.5.5 Any compound that fails to meet the 20% RSD or or 0.99 correlation coefficient criteria must be flagged as estimated for detects (or must be noted in the narrative). If there are non-detects the compounds may be reported if there is adequate sensitivity to detect at the quantitation limit. To demonstrate adequate sensitivity analyze the low level point of the initial calibration in each analytical batch (LLCCV) The criteria for demonstrating adequate sensitivity is detection in the LLCCV using the standard qualitative identification criteria.

9.2.4.2.5.6. When calculating the calibration curve using the linear calibration model a minimum quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the low concentration back into the curve. The recalculated concentration of the low calibration point should be within ±50% of the standard's concentration. This evaluation can be checked using the Initial Calibration %Drift Report in Chrom. Any detects for analytes calibrated using the linear model and failing this readback criterion must be flagged as estimated or detailed in the narrative.

9.2.4.3. Calibration Point Read-back Criteria: Whichever calibration model above is selected, it should be subjected to an additional check to establish the representativeness of the data that were used to produce it. This check is the refitting of each calibration

point response back to the calibration model, or the comparison of the calculated amount of the standard against the expected amount.

- CHROM software provides an Initial Calibration %Drift report which shows the % Error for each calibration point. This report must be reviewed in addition to the %RSD / Linear Response Factor.

- The absolute value of the % Error for each calibration point should be < 30%. For the lowest calibration point, the % Error may be <50%.

- See Section 11.8 for the Calculation of Percent (%) Error.

9.2.4.4. **Initial Calibration Verification (ICV):**.Once the initial calibration has been analyzed and has met the above criteria, a second source Initial Calibration Verification (ICV) (as prepared in Section 7.2) must be analyzed and evaluated. The ICV must meet the criteria of 70-130% recovery for all compounds with the exception of the poor performing compounds listed in Attachment 1 which are allowed to be within 50-150% : An NCM must be initiated to denote any ICV non-conformances.

9.2.4.5. The ICV must meet the criteria of 70-130% recovery for all compounds however up to 10% of the compounds are allowed to exceed these criteria as long as their recoveries are within 65-135%. For the poor performers (see Attachment 1) the range is 50-150%. If the criterion is not met, a second ICV may be analyzed after corrective measures are taken. If a second ICV analysis fails to meet criteria proceed with corrective action and the analysis of a new initial calibration range. Flagging: If the ICV limits are outside of criteria (high) for an analyte and that analyte is undetected in the sample, no flagging or narration is required. If the ICV limits are outside of criteria (low) for an analyte and that analyte is undetected in a sample, narrate the non-conformance in an NCM. When that out of spec analyte is detected in a sample, describe the issue in the narrative, or flag as estimated.

9.2.4.6. **Continuing Calibration Verification (CCV):** A CCV consisting of a standard at or near the midpoint of the Initial Calibration Range is analyzed every 12 hours of instrument operation or at the beginning of an analytical sequence to verify the initial calibration. The calibration verification consists of a DFTPP instrument performance check (not required for 8270E), and analysis of a calibration verification standard. **Note:** Certain state regulatory programs have defined calibration acceptance limits which, where applicable, are used for calibration

evaluations. The TALS ICAL Limit Groups detail these regulatory program criteria.

9.2.4.5.1 Tune Verification: Follow the procedure for verifying the instrument tune described in section 9.2.1 using a 50 ng injection of DFTPP. If the tune cannot be verified, analysis must be stopped, corrective action taken and a return to "control" demonstrated before continuing with the calibration verification process. For 8270E analysis only, tune verification is required just prior to ICAL.

9.2.4.5.2 Calibration Verification: Analyze the calibration verification standard immediately after a DFTPP that meets criteria. Daily analysis of the DFTPP is not required as part of the CCV for 8270E analysis. .When samples are analyzed after an ICAL the last ICAL standard may be used as the starting time reference for evaluation. Use the mid point calibration standard (approximately 50ug/l). NOTE: The calibration standard contains internal standards; Dichlorobenzene d_4, Naphthalene d_8, Acenaphthene d_{10}, Phenanthrene d_{10}, Chrysene d_{12}, and Perylene d_{12} at 40ug/l (0.1ug/L for SIM). The calibration check standard must also include all the target analytes from the original calibration.

9.2.4.5.3 The RFs must meet the criteria for the compounds in Table 18. Any compound that fails the minimum response factor must be reported as estimated for detects and must have a demonstration of sensitivity to report non-detects. To demonstrate adequate sensitivity for out of criterion compounds analyze the low level point of the calibration (LLCCV) in the analytical sequence. The criterion for the LLCCV is detection only but the standard qualitative identification criteria in the method must be met

9.2.4.5.4 The percent difference (when using average response factor) or percent drift (when using linear regression) of the compounds in Table 18 must be ≤20% for at least 80% of the total analyte list. If more than 20% of the compound list fail to 20% difference or drift criterion then appropriate corrective action must be taken prior to the analysis of the samples. Any individual compound that fails must be reported as estimated for detects and must have a demonstration of sensitivity to report non-detects. To demonstrate adequate sensitivity for out of criterion compounds analyze the low level point of the calibration (LLCCV) in the analytical sequence. The

criterion for the LLCCV is detection only (%D criteria are not applied) but the standard qualitative identification criteria in the method must be met.

9.2.4.5.5 **CCV Poor Performers**: Refer to Attachment 1 for the identification of poor and/or erratic performing analytes. These analytes are allowed a %D >20% but must be <50 %D to be acceptable. If there are poor performers that exceed 50%D, the data may be reported provided results are noted as estimated. An NCM must be initiated to denote this situation.

9.2.4.5.6 The retention times of the internal standards from the calibration check must be within ±30 seconds of the internal standards from the mid point standard of the original calibration. If the retention time for any internal standard changes by more than 30 seconds from the latest daily (12 hour) calibration standard, the chromatographic system is inspected for malfunctions, and corrections made as required. If corrective action does not result in the retention time criteria being achieved, the system must be re-calibrated using four additional standards.

9.2.4.5.7 The response (area count) of each internal standard in the calibration verification standard must be within 50 - 100% of its corresponding internal standard in the mid-level calibration standard of the active calibration curve. If the EICP area for any internal standard changes by more than a factor of two (-50% +100%), the mass spectrometer system must be inspected for malfunction and corrections made as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning is required.

9.2.4.5.8 The relative retention times of each compound in the calibration verification standard must agree within .06 relative retention time units of its value in the initial calibration.

9.2.4.5.9 Use the average response factors from the original five-point calibration for quantitative analysis of target analytes identified in field samples.

9.2.4.5.10 Prepare a calibration summary or list indicating which compounds did not meet the 20% average percent difference criteria. Record this information in that run log.

9.2.4.7. **Low Level Continuing Calibration Verification (LLCCV):** An LLCCV consisting of the low level standard from the initial

calibration range is analyzed every 12 hours of instrument operation after the CCV. The purpose and evaluation of the LLCCV is described in Section 9.2.4.4.4.

10.0 Procedure

10.1. Gas Chromatograph/Mass Spectrometer Operation

10.1.1. The sequence of events for GC/MS analysis involves many steps. First the injection system and column performance and calibration must be verified. Maintenance operations are performed as needed.

10.1.2. Preparation of the Injection Port Liner and Installation Procedure: Prior to the start of initial calibration and each daily analysis of sample extracts, a new liner for the injection port must be prepared. Once a liner has been used it is no longer inert and will cause serious chromatography problems with phenols and other compounds. When preparing the liner, proper laboratory protection must be worn and the liner must be prepared in a well-ventilated hood. When the procedure is completed all traces of toluene, Sylon-Ct and methanol will be removed immediately so that extraction solvents and preparation of sample extracts will not come into contact with these solvents and become contaminated.

10.1.2.1 Remove one liner from a 40ml VOA bottle containing other liners immersed in Sylon-Ct solution. Rinse off the liner with Toluene and wipe dry. Insert 1cm of pre-silanized glass wool partially into one end of the liner and trim neatly. Push the glass wool into the center of the liner so that it is 1 1/4" from the bottom. Do not use glass wool or solvents that are dirty (i.e. suspended particles) or use liners which are chipped on the ends, deformed or fractured. Inspect the glass wool for cleanliness after it has been inserted.

10.1.2.2 Using a Pasteur pipette flush out the interior of the liner containing the glass wool with Sylon-Ct. Rest the liner horizontally on a small beaker and allow the Sylon-Ct to re-deactivate the interior surfaces and the glass wool. There should be no air bubbles caught in the glass wool. After several minutes flush out the Sylon-Ct with toluene and finally with methanol. Dry the outer surface of the liner and rest it on the injection port housing until the remaining methanol is boiled off

10.1.2.3 Insert the liner with the newly silanized glass wool plug into the injection port. Verify that the column extends up into the injection port and is perpendicular. Inspect the graphite seal and replace it if the edges are knife-shaped.

10.1.2.4 The septum is always replaced daily. Bake out the column at 300^{O}C for 15 minutes after the vacuum in the analyzer has returned to normal.

10.1.2.5 Performance may enhanced by clipping a small portion of the column at the injection port end. Document this activity in the maintenance record.

10.1.3. Prior to calibration or sample analysis always verify that the analyzer is under sufficient vacuum and that the column has proper carrier gas flow.

10.1.4. Establish the following GC/MS operating conditions:

10.1.4.1 Full Scan Operating Mode

Full Scan Mode – Standard Injection Volume
Mass Range: 35 to 500amu
Scan Time: 1 sec/scan
Transfer Line Temperature: 300°C
Source Temperature: Preset by H.P. at 280°C
Scan start time: 1.0 minutes
Initial Column Temperature and Hold Time: 45°C for 0.5 minutes
Column Temperature Program: 20°C /min to 100°C 25°C/min to 270°C 10° C/min to 310°C
Final Column Temperature Hold: 310°C for 5 minutes
Carrier Gas: Ultra High Purity Grade Helium at 1.3ml/min
Injector Temperature: 275°C
Injector: Grob-type, pulse, splitless
Injection Volume: 1ul
Splitless Valve Time: 0.3 minutes

Full Scan Mode – Large Volume Injection (LVI)
Mass Range: 35 to 500amu
Scan Time: 1 sec/scan
Transfer Line Temperature: 300°C
Source Temperature: Preset by H.P. at 280°C
Scan start time: 1.0 minutes
Initial Column Temperature and Hold Time: 45°C for 0.5 minutes
Column Temperature Program: 20°C /min to 100°C

25°C/min to 270°C 10° C/min to 310°C
Final Column Temperature Hold: 310°C for 5 minutes
Carrier Gas: Ultra High Purity Grade Helium at 1.3ml/min
Injector Temperature: 275°C
Injector: Grob-type, pulse, splitless
Injection Volume: 5ul
Splitless Valve Time: 0.3 minutes

10.1.4.2 SIM Operating Mode

SIM Mode
Mass Range: 35 to 500amu
Scan Time: 1 sec/scan
Transfer Line Temperature: 300°C
Source Temperature: Preset by H.P. at 280°C
Scan start time: 1.5 minutes
Initial Column Temperature and Hold Time: 40°C for 0.5 minutes
Column Temperature Program: 20°C /min to 100°C 25°C/min to 270°C 10° C/min to 310°C
Final Column Temperature Hold: 310°C for 3 minutes
Carrier Gas: Ultra High Purity Grade Helium at 1.3ml/min
Injector Temperature: 275°C
Injector: Grob-type, pulse splitless
Injection Volume: 1ul
Splitless Valve Time: 0.3 minutes

10.1.4.3 Isotope Dilution Selected Ion Monitoring Mode :

SIM Parameters

Group 1
Plot 1 Ion: 74.0

Ions/Dwell in Group	(Mass Dwell)	(Mass Dwell)	(Mass Dwell)
	42.0 50	43. 0 50	68.0 50
	74.0 50	128.0 50	129.0 50
	136.0 50	150.0 50	152.0 50
	93.0 50	66.0 50	
	58.0 50		
	88.0 50		

Group 2
Group Start Time: 6.00
Plot 1 Ion: 152.0

Ions/Dwell in Group	(Mass Dwell)	(Mass Dwell)	(Mass Dwell)
	151.0 50	152.0 50	153.0 50
	154.0 50	162.0 50	164.0 50
	165.0 50	166.0 50	

Group 3
Group Start Time: 7.80
Plot 1 Ion: 188.0

Ions/Dwell in Group	(Mass Dwell)	(Mass Dwell)	(Mass Dwell)
	94.0 50	101.0 50	142.0 50
	178.0 50	179.0 50	188.0 50
	202.0 50	264.0 50	266.0 50
	284.0 50		

Group 4
Group Start Time: 10.50
Plot 1 Ion: 228

Ions/Dwell in Group	(Mass Dwell)	(Mass Dwell)	(Mass Dwell)
	120.0 50	228.0 50	229.0 50
	240.0 50		

Group 5
Group Start Time: 12.00
Plot 1 Ion: 252.0

Ions/Dwell in Group	(Mass Dwell)	(Mass Dwell)	(Mass Dwell)
	138.0 50	139.0 50	252.0 50
	253.0 50	260.0 50	264.0 50
	267.0 50	276.0 50	278.0 50

Table 19: Target Compound - Primary and Monitoring Ions

Compound	1	2	3
1,4-Dioxane-d8	96	64	62
1,4-Dioxane	88	58	57
1,4-Dichlorobenzene-d4	152	150	

10.1.5. The above listed instrument conditions are used for all analytical standards for calibration and for all sample extracts analyzed by this method.

10.1.5.1 The column conditions, scan start time, and splitless valve time for analysis of DFTPP only are as follows are as follows:

Initial Column Temperature and Hold Time: 140^{o}C for 0.5 minutes
Column Temperature Program: 140^{o} to 320^{o}C at 22^{o}C/minute
Final Column Temperature Hold: 320C for 0.5 minutes
Scan Start Time: approx. 5 minutes
Splitless Valve Time: 0.3 minutes
Injection Volume: 2 ul

10.2. Analytical Sequence

10.2.1. Dilutions are made based on initial GC/MS analysis. Dilutions are made in 1-ml vials using microsyringes. Calculate the dilution factor using the equation below:

DF= Ph / 5 x Is

Where:

DF = Dilution Factor
Ph = Sample Peak Height
Is = Internal Standard Peak Height

When DF >1 but <2, combine 500ul of sample extract with 500ul methylene chloride in a 1 ml amber vial, add20 ul internal standard and crimp seal

Use **Table 20** to determine dilution and internal standard amount.

Table 20
Dilution Factor Calculations

DF Value	Volume of Sample (ul)	Volume of Methylene Chloride (ul)	Volume of ISTD (ul)
<1	1,000	None	None
>1, <2	500	500	10
>4, <5	200	800	16
>10, <20	100	900	36
>20	500*	500	10

*Prepare this dilution by serially diluting the >10, <20 dilution

10.2.2. Instrument Performance and Calibration Sequence

10.2.2.1. Once the GC/MS instrument has been setup and maintained as detailed in Section 10.1, the first operations to be performed are the performance checks and calibration standards.

10.2.2.2. Analyze the Instrument Performance Check Standard (DFTPP) as discussed in Section 9.2.1.

10.2.2.3. Initially and as required, analyze the Initial Calibration Range (minimum 5 points, six points for second order regression) as detailed in Sections 7.2.1 and 9.2.4.2. Evaluate the acceptability of the Initial Calibration Range as detailed in Section 9.2.4.2.

10.2.2.4. Immediately after the Initial Calibration Range only, analyze the Initial Calibration Verification (ICV) as detailed in Sections 7.2. and 9.2.4.3. Evaluate the acceptability of the ICV as detailed in Section 9.2.4.3.

10.2.2.5. Every 12 hours, reanalyze and evaluate the Instrument Performance Check Standard (DFTPP), not required for 8270E followed by the Continuing Calibration Verification (CCV) and Low Level Continuing Calibration Verification (LLCCV) as detailed in Section 9.2.3, 9.2.4.4 and 9.2.4.5. Evaluate the acceptability of the CCV and LLCCV as detailed in Section 9.2.4.4

10.2.2.6. Client samples and QC samples are analyzed (as detailed in Section 10.2.3) after acceptable Instrument Performance and Calibration Checks and until the 12 hour clock expires. Repeat the sequence as required. The automation of GC/MS runs is accomplished via the “SEQUENCE” macro of the ChemStation.

10.2.3. Sample Analysis Sequence

10.2.3.1. Sample extracts are normally prepared on the same day as analysis. The GC/MS operator will prepare the extracts that will be run on his or her instrument. Volume adjustments to the extracts will be made at the discretion of the supervisor.

10.2.3.2. Prior to the start of sample analysis the GC/MS operator will generate a sequence program containing the list of the sample extracts to be analyzed, the position on the autosampler tray, and the proper acquisition and tune methods that are to be used. This sequence program contains all the necessary information on the samples to be analyzed and how the GC/MS system is to analyze them. The sample extracts are loaded onto the autosampler (ALS) tray. Their position is verified by checking them against the ALS number on the sequence. This batch analysis will be performed automatically over the 12-hour period.

10.2.3.3. The analytical run log is printed as a record of samples analyzed. The analyst will annotate the run log with any required information regarding anomalies or unusual events. The run log must be signed by the analyst and a reviewed and signed by a trained peer or manager

10.3. Data Processing

10.3.1. Prior to processing any standards or samples, target compound lists and sublists must be assembled. Chrom's auto-processing system queries TALS (LIMS) for each sample's processing parameters (including target compounds lists) and downloads the required processing methods from LIMS to analyze data. These lists are required for processing of all data files including calibration files. The data includes compound names, retention time data, quantitation ions, qualitative identification ions, and the assigned internal standard for qualitative and quantitative identification.

10.3.2. Key data is manually entered the first time a compound list is used for data processing. Processing data using a compound list automatically generates response factor data and updates retention information.

10.3.3. The characteristic ions for target compounds, surrogate compounds, and internal standards which can be determined using SW8270D and 8270E are listed in Table 21..

10.4. Interpretation and Qualitative Identification: Qualitative identification of target compounds is based on retention time and mass spectral comparison with characteristic ions in the target compound list. The reference mass spectrum is taken from a standard of the target compound analyzed by this method. The characteristic ions are the three ions of greatest relative intensity or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met:

10.4.1 Target Analytes: Qualitative identification of target compounds is based on retention time and mass spectral comparison with characteristic ions in the target compound list. The reference mass spectrum is taken from a standard of the target compound analyzed by this method. The characteristic ions are the three ions of greatest relative intensity or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met:

10.4.1.1. Once the GC/MS instrument has been setup and maintained as detailed in Section 10.1, the first operations to be performed are the performance checks and calibration standards.

10.4.1.2. The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other.

10.4.1.3. The relative retention time (RRT) of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

10.4.1.4. The most abundant ion in the standard target spectrum that equals 100% MUST also be present in the sample target spectrum.

10.4.1.5. All other ions that are greater than 10% in the standard target spectra should also be present in the sample.

10.4.1.6. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

10.4.1.7. If the compound does not meet all of the criteria listed above, but is deemed a match in the technical judgment of the mass spectral interpretation specialist, the compound will be positively identified and reported with documentation of the identification noted in the raw data record.

10.4.2 Non-Target Analytes: Upon client request a library search to identify non-target Tentatively Identified Compounds (TIC) is performed. The NIST/EPA/NIH mass spectral library is used to identify non-target compounds (not including internal standard and surrogate compounds) of greatest apparent concentration by a forward search of the library. The following guidelines are used by the analyst when making TIC identifications:

10.4.2.1. Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

10.4.2.2. The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

10.4.2.3. Molecular ions present in the reference spectrum should be present in the sample spectrum.

10.4.2.4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

10.4.2.5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

10.4.2.6. If, in the technical judgement of the mass spectral interpretation specialist, no tentative identification can be

made, the compound will be reported as 'Unknown'. If the compound can be further classified the analyst may do so (i.e, 'Unknown hydrocarbon', 'Unknown acid' , etc.).

10.5. Data Reporting

10.5.1. Final Report. The Chom data system automatically produces a data report consisting of hardcopy reports corresponding to specific data reporting requirements, which is uploaded to the TALS LIMS System for the report production group.

10.5.1.1. Total Ion Chromatogram. Full length chromatogram depicting the full length of the GC/MS acquisition.

10.5.1.2. Spectra of all detected target compounds. A page for each detected target compound spectra with a standard reference spectrum for comparison.

10.5.1.3. The calculations of the concentrations of each target compound in the sample, reported in units of ppb, ug/kg or ug/l.

10.5.1.4. Data summaries for each method blank indicating which samples were extracted with the indicated blank.

10.5.1.5. A copy of the initial calibration range together with the calibration verification report, and tune report.

10.5.1.6. Quality Control (QC) data report for each batch including surrogate recoveries, internal standard area summaries, LCS, MS/MSD and RPD summaries.

10.6. The low-level calibration standard establishes the reporting limit. All reported data must be at a concentration at or above the low concentration standard. Any quantitative values below the report limit must be qualified as estimated.

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11.0. Calculations/Data Reduction

11.1. Target Compounds: are quantitated using the internal standard method (see the formula in Section 11.3).

11.1.1. Identified target compounds are quantitated using the integrated abundance from the EICP of the primary characteristic ion. The internal standard used shall be the one nearest the retention time of the analyte).

11.1.2. The average response factor (RRF) from the initial calibration is used to calculate the target analyte concentration in client samples using the formula found in Section 11.3. See Section 9.2.4 for discussion of RRF.

11.1.3. Secondary ion quantitation is utilized only when there are sample interferences preventing use of the primary characteristic ion. If secondary ion quantitation is used an average relative response factor (RRF) must be calculated using that secondary ion.

11.2. Non-Target Compounds (Tentatively Identified Compounds): An estimated concentration for non-target (tentatively identified compounds) is calculated using the internal standard method (see formula in Section 11.3). For quantiation, the nearest eluting internal standard free of interferences is used. The procedure used for calculating the concentration of non-target compounds is the same as that used for target compounds (see Section 11.1) with the following revisions:

11.2.1. The total area count of the non-target compound is used for As (instead of the area of a characteristic ion).

11.2.2. The total area count of the chosen internal standard is used as Ais (instead of the area of a characteristic ion).

11.2.3. A RF on 1.0 is assumed.

11.2.4. The resulting concentration is qualified as estimated ('J') indicating the quantitative uncertainties of the reported concentration.

11.3. Internal Standard Calculation:

11.3.1. Aqueous Samples

$$\text{Concentration } (\mu g/L) = \frac{(As)(Cis)(D)}{(Ais)(RF)(Vs)\ (Vi)\ (1000)}$$

Where:

As	=	Area of the characteristic ion for the target analyte in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the characteristic for the associated internal standard
RF	=	Average response factor from the initial calibration.
Vs	=	Volume of sample extracted (ml)

The 1000 in the denominator represents the number of ul in 1 ml.

11.3.2. Solid Samples

$$\text{Concentration } (\mu g/KG) = \frac{(As)(Cis)(D)(Vt)}{(Ais)(RF)(Ws)\ (Vi)\ (1000)}$$

Where:

As	=	Area of the characteristic ion for the target analyte in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the characteristic for the associated internal standard
RF	=	Average response factor from the initial calibration.
Vt	=	Volume of concentrated extract (ul)
Ws	=	Weight of sample (g)

The 1000 in the denominator represents the number of ul in 1 ml.

11.4. Relative Response Factors

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

A_x = Area characteristic ion for the compound (see Table 21)
Ais = Area characteristic ion of associated internal std (See Table 21)
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

11.5. Percent Relative Standard Deviation (% RSD) : as discussed in Section 9.2.4.4 (Initial calibration):

$$\%\ RSD = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

11.6. Percent Difference (% D):as discussed in Section 9.2.4.4 (Continuing calibration):

$$\%\ D = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

Where: RRFc = RRF from continuing calibration

$\overline{RRF_i}$ = Mean RRF from current initial calibration

11.7. Percent Recovery (% R): Surrogates and Spikes

$$\text{Recovery (\%)} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}} \times 100$$

11.8. Calculation of Percent (%) Error

$$\%Error = \frac{x_i - x_i'}{x_i} x\,100$$

Where:

x_i ' = Measured amount of analyte at calibration level *i*, in mass or concentration units

x_i = True amount

11.9. Dry Weight Correction: All solid samples must be corrected for dry weight using the following formula for dry weight determination.

$$DW = \frac{Gd}{Gw} \times 100$$

Where:

DW	=	Percent % Dry Weight
Gd	=	Dry weight of selected sample aliquot
Gw	=	Wet weight of selected sample aliquot

Multiply the DW value times the wet weight of the sample extracted. NOTE: This calculation can also be performed automatically by the target system provided the DW value is available and entered into the system.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

A Method Detection Limit (MDL) study, as described in the TestAmerica corporate Detection and Quantitation Limits SOP, CA-Q-S-006, must be performed initially and whenever a significant change affecting sensitivity is made to the analytical system. The MDL must be re-evaluated from quarterly MDL points at least every 12 months.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Lower Limit of Quantitation Verification

The lowest calibration standard analyzed establishes the LLOQ or Reporting Limit. The capability to reliably detect this concentration through the preparation, clean-up and analytical procedure is verified through the annual analysis of a standard at the LLOQ/RL. The LLOQ verification shall also be performed whenever significant changes are made to the preparation and/or analytical procedure.

12.3.1 The LLOQ verification standard shall be prepared at a concentration 0.5-2 times the LLOQ/RL, and be taken through all of the same preparation and clean-up methods as client samples.

12.3.2 The LLOQ verification standard for aqueous matrix shall be prepared using laboratory deionized water and for the solid matrix using clean Ottawa sand. Other clean matrices may be used in addition, for project specific requirements.

12.3.3 The LLOQ shall be verified annually on each instrument used for client sample analysis.

12.3.4 Recovery of each analyte must meet the laboratory established LCS recovery limits + 20%. (For example, if the LCS recovery limits are 70-130%, the LLOQ verification must meet recovery limits of 50-150%.) Once sufficient points have been generated, LLOQ based statistical limits may be used in place of limits based on LCS recovery.

NOTE: The lower recovery limit for the LLOQ can be no lower than 10%.

12.4. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, (*Training*), for the laboratory's training program.

13.0. Pollution Control

13.1 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."

14.0. Waste Management

14.1 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 TestAmerica Edison SOPs Nos. ED-SPM-007 (*Disposal of Samples and*

Associated Laboratory Waste, current revision) and ED-SPM-008 (*Laboratory Waste Disposal Procedures, current revision).* The following waste streams are produced when this method is carried out:

- Auto sampler vials and expired standards: These vials are collected in satellite accumulation within the instrument laboratory. The vials are then placed into a 55 steel open top drum in the waste room. When the drums are full, the drum will be collected by the waste vendor for disposal. This waste is treated for incineration.

  Teris Profile Number: 50016652
  Onyx Profile WIP Number: 282493

- Mixed Solvent Waste: Mixed solvent waste is collected in a small beaker inside the bench top hood. This waste is then transferred into the satellite accumulation container in the Organic Prep. Lab. on a daily basis. This material is transferred into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.

  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

14.1. Pollution Prevention

14.2.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

14.2.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

15.0. References / Cross-References

15.1. United States Environmental Protection Agency, “Method SW8270D, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Laboratory Manual, Physical/Chemical Methods, Revision 5, July 2014..

15.2. United States Environmental Protection Agency, "Method SW8270E, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry", Test Methods for Evaluating Solid Wastes, SW846 Update IV, Laboratory Manual, Physical/Chemical Methods, Revision 6, June 2018.

15.3. United States Environmental Protection Agency, "Method SW8000D: Determinative Chromatographic Separations", Test Methods for Evaluating Solid Wastes, SW846, Laboratory Manual, Physical/Chemical Methods, Update V, Revision 4, October 2012..

15.4. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, current revision.

15.5. TestAmerica Edison SOP No. ED-ORP-002, *SW846 Method 3510C-Extraction of Semi-Volatile Organic Compounds in Water by Separatory Funnel,* current revision.

15.6. TestAmerica Edison SOP No. ED-ORP-043, *SW846 Method 3580A - Waste Dilution Prep for Analysis of BNAs by SW846 Method 8270,* current revision.

15.7. TestAmerica Edison SOP No. ED-ORP-044, *Procedure for the Microwave Extraction of Solids, SW3546, current revision.*

15.8. TestAmerica Document No. CW-E-M-001, Corporate Environmental Health and *Safety Manual,* current revision.

15.9. TestAmerica Corporate Quality SOP No. CA-Q-S-001, *Solvent & Acid Lot Testing & Approval*, current revision.

15.10. TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*), current revision.

15.11. TestAmerica Edison SOP No. ED-GCS-001, *Preparation and Screening of Semivolatile Organic Extracts for GC/MS Analysis*, current revision.

15.12. TestAmerica Edison Work Instruction Document No. EDS-WI-012, *Client Complaint/Corrective Action Form,* current revision.

15.13. TestAmerica Edison SOP No. ED-GEN-003, *Standard Operating Procedure for Control of Non-Conformances and Corrective Action,* current revision.

15.14. TestAmerica Edison SOP No. ED-ORP-001, *Extraction of Semivolatile Organic Compounds in Water, EPA Method 625.1,* current revision.

15.15. TestAmerica Edison SOP No. ED-GEN-022, *Training,* current revision.

15.16. TestAmerica Corporate Quality Memorandum, CA-Q-QM-002, *GC/MS Tuning Policy*, current revision.

15.17. TestAmerica Corporate Quality SOP No. CA-Q-S-006, *Detection and Quantitation Limits,* current revision.

16.0. Method Modifications:

Method 8270E requires the DFTPP tune standard to be analyzed once prior to an ICAL and not daily prior to sample analysis. Until such time as 8270D is removed from lab capabilities and in order to satisfy both 8270D and 8270E The laboratory will analyze the DFTPP tune daily, prior to QC and sample analysis. The laboratory will use the tighter criteria from Methods 8270C/8270D for tune evaluation, rather than the criteria suggested in Table 3 of Method 8270E.

17.0. Attachments

Attachment 1 Poor Performing Analytes

18.0. Revision History

- Revision 8, date 06/29/2020
    - Updated to Eurofins branding.
    - Updated throughout to include 8270E requirements.
    - Removed references to SW846 3550B/C prep methods (no longer in use for this method at Edison lab).
    - Update equipment listed in Section 6.0.Updated analytical column in Section 6.1.2.
    - Updated, deleted and renumbered tables as required.
    - Made extensive updates to Standards (sources and preparation) in Section 7.2.
    - Removed all references to Aromatic Amines. Deleted all tables specific to Aromatic Amine analysis. Renumbered remaining tables in document and updated text references.
    - Throughout document clarified tune requirements for 8270E.
    - Following added to Section 9.1.1: For method 8270E the method blank is generally acceptable if target analyte concentrations are less than the one half the reporting limit.
    - Calibration Point Read-back Criteria was added to Section 9.2.4.3. The calculation for percent error was added to Section 11.8.
    - Section 9.2.4.2.3: added following for 8270E: the calculated concentration or amount of each analyte of interest in the CCV standard should fall within ±20%.
    - Section 9.2.4.2.5.6: added ‘The recalculated concentration of the low calibration point should be within ±50% of the standard’s concentration.”
    - Section 12.1 revised to reflect the updated MDL procedure.
    - Added Section 12.3: annual Lower Limit of Quantitation Verification
    - Added Corporate SOP CA-Q-S-006, Detection and Quantitation Limits to references.
    - Section 16.0: added a Method Modification regarding tuning check requirements.
- Revision 7, date 06/08/2018
    - Section 2.3: revised to clarify that RVE/LVI is lab standard procedure.
    - Section 9.1.3: removed statement regarding allowance for up to five analytes to recover outside of lab acceptance limits in LCS/LCSD.
    - Section 9.2.4.3: Replace table ‘ICV Poor Performers (50-150% Recovery) with

expanded list of 'Poor Performing Analytes' in Attachment 1.
  - Added Section 9.2.4.4.5: CCV Poor Performers
  - Corrected number in section 9.2.4.5
  - Added Attachment 1 – Poor Performing Analytes

- Revision 6, date 01/12/2018:

  - Section 7.2.5 included to specify reagent and standard storage conditions.
  - Revised Section 9.1.3 to clarify requirements for specific LCS/LCSD evaluation criteria regarding the # of out of criteria analytes.
  - Revised Section 9.2.4.3 to add 2,4-Dimethylphenol as a poor performing analyte, increased the range for the poor performers to 50-150 and also expanded the guidelines for flagging the ICV outliers.

- Revision 5, dated 09/29/2017:
  - Revised Section 9.1.1 to clarify requirements for surrogate recovery in method blanks.

- Revision 4, dated 08/21/2017:
  - Updated throughout to add a procedure for the analysis of 1,4-dioxane by isotope dilution selected ion monitoring (SIM)
  - Added tables for isotope dilution SIM standards. Renumbered all tables as necessary.
  - Section 7.2.1: added a list of full scan calibration list options.
  - Table 3: Renamed 'Full Scan Stock Standards'.
  - Section 9.2.1: noted that DFTTP applies only to full scan analysis.
  - Section 9.2.3: updated CCV concentrations
  - Added reference to GC/MS Tuning Policy in Section 15.16.

- Revision 3, dated 01/07/2016:
  - Tables 1 and 2: added SIM as option for 1,4-Dioxane.
  - Section 2.3: removed SW3541 (Soxtherm) as option for soils prep (lab has discontinued use of this method). Also removed SW3541 SOP reference from Section 15.0.
  - Tables 19 and 20: added source and prep instructions for 1,4-Dioxane SIM standard. Updated source and prep instructions for 4,6-Dinitro-2-methylphenol.
  - Table 22: added prep instructions for 1,4-Dioxane and 4,6-Dinitro-2-methylphenol SIM ICV standard.
  - Corrected the information in the 'DFTPP Key Ions and Abundance Criteria' table in Section 9.2.1 to match the info found in SW846 8270C.
  - Section 10.1.4.2: updated "SIM Parameters" to included ion masses/dwell times for 1,4-Dioxane.

- Revision 2, dated 01/28/2015:
  - Extensively reformatted the SOP. Placed tables that had been in rear of document into the body of the text. Renumbered tables as applicable and fixed text references to tables.
  - Section 1.1, Table 1: Revised table to include all current analytes. Also footnoted those compounds which are currently analyzed by SIM.

- Section 2.3: added options for extraction of solids by SW846 3456 (Microwave Extraction) and by SW3580A (Waste Dilution) and added SOP references. Deleted reference to SOP ED-ORP-005 (SW3550B – Low Level); Updated Section 15 (References).
- Section 2.5: added text detailing the RVE/LVI options.
- Section 2.6: added table which includes all analytes routinely analyzed by SIM.
- Section 6: updated to include newer GC, MS and autosampler models currently in use.
- Section 6.1.3: added Zebron ZB column as an option.
- Section 7.2: extensively revised standards information to reflect switch to Restek standards.
- Table 3:Added Custom Aromatic Amine Surrogate Standard and revised Table 8 to include initial calibration prep instructions for the Aromatic Amine surrogates.
- Throughout document: removed references to Target and replaced with Chrom.
- Section 7.2.1: Added reference to section 10.2.1.2 for LVI.
- Added Section 7.2.1.3.1 and Table 17A both of which discuss use of Aromatic Amine surrogates.
- Section 7.2.1.2: Added reference to Tables 9,10 and 11 (ICV Preparation)
- Section 8.0: Added Sample container and minimum sample size (250 ml) for Reduced volume extraction.
- Sections 9.1.2, 9.1.3, 9.1.4 and 9.2.4: added statement that certain state regulatory programs have defined recovery limits which, where applicable, are used for spike and calibration evaluations.
- Section 9.1.2: Deleted sentence “A minimum of 16 spiked analytes are reported to in client reports (the full list is reported at least once during each 2 year period because we employ full spiking list.
- Section 9.1.4: Added note regarding use of Aromatic Amine Surrogates.
- Section 9.2.2.2: Added reference to ICV Preparation tables in Section 7.2.
- Section 9.2.3: added more specific info as to the concentration of the CCVs for all techniques.
- Section 9.2.4.2.1: Changed to reflect that each analyte should meet minimum RF’s, not the average across the calibration. Added LLCCV requirement.
- Section 10.3.1: added explanation of Chrom’s interaction with TALS. Removed references to Target.
- Section 9.2.4.2.5.5: Added: (or can be noted in the narrative)
- Section 9.2.4.2.5.6: Revised last sentence to read: “This evaluation can be checked using the Initial Calibration %Drift Report in Chrom.”
- Section 9.2.4.3: Removed 65-135% criteria and added “poor performing” analyte list and associated criteria of 60-140%.
- Section 9.2.4.4.3: Added LLCCV criterion for RFs
- Section 9.2.4.4.4: Added LLCCV criterion for %D
- Section 10.1.4: Updated GC/MS operating conditions for full scan, SIM and DFTPP.
- Section 10.1.4.1: added a table detailing operating conditions for LVI option.
- Table 2: Added 2-ethylaniline, 2,4-dimethylaniline, 3,4-dimethylaniline, 2,3-dimethylaniline, 2,4,5-trimethylaniline and 4-chloro-o-toluidine to Working Standards preparation information.
- Table 25: updated to include all current analytis/surrogates/internal standards and associated ions.
- Throughout document: updated LQM section references as appropriate as some have changed with the latest LQM revision.

Title: SW846 Method 8082A, Analysis of Polychlorinated Biphenyls by Gas Chromatography

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Catalina Dalangin SVOA GC Manager	04/01/20 Date	Dan Helfrich Health & Safety Manager	04/01/20 Date
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Sylvanus Klusey Operations Manager - Organics	04/01/20 Date		

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1.0 Scope and Application

This method is used to quantify specific polychlorinated biphenyls (PCBs) as Aroclors (see Table 1 below) in extracts from aqueous, soil, sludge, leachate, wipe or oil matrices by direct injection dual capillary column gas chromatography using SW846 Method 8082A. An electron capture detector (ECD) is employed for detection.

1.1 Analytes, Matrix(s), and Reporting Limits

The specific analytes determined by this method are identified in Table 1.

Table 1
Polychlorinated Biphenyls

Compound Name	CAS Registry No.
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5
Aroclor 1262	37324-23-5
Aroclor 1268	11100-14-4

The routine TestAmerica Edison reporting limits (RLs) by analyte and matrix are summarized in Table 2 (below).

Table 2
Reporting Limits by Matrix

Parameter	Soil	Soil	Water	Leachate	Oil	Wipe
	Reporting Limits (ug/kg) LOW Level	Reporting Limits (ug/kg) MED Level	Reporting Limits (ug/L)	Reporting Limits (mg/L)	Reporting Limits (ug/kg)	Reporting Limits (ug/wipe)
Aroclor-1016	67	500	0.50	0.0050	1000	0.40
Aroclor-1221	67	500	0.50	0.0050	1000	0.40
Aroclor-1232	67	500	0.50	0.0050	1000	0.40
Aroclor-1242	67	500	0.50	0.0050	1000	0.40
Aroclor-1248	67	500	0.50	0.0050	1000	0.40
Aroclor-1254	67	500	0.50	0.0050	1000	0.40
Aroclor-1260	67	500	0.50	0.0050	1000	0.40
Aroclor-1262	67	500	0.50	0.0050	1000	------
Aroclor-1268	67	500	0.50	0.0050	1000	------

The most current MDLs and RLs for this method can be found in the active TestAmerica LIMS (TALS) SW846 8082A Method Limit Group (MLG) database.

1.2 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work Request*) and Section 19 (*Test Methods and Method Validation)* of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1. Samples undergo a preparation step prior to analysis by SW846 Method 8082A. A measured volume or weight of sample (15 g for soil, 1 g for oil, 250 ml for water and TCLP/SPLP/ASTM leachates) is extracted using the appropriate matrix-specific sample extraction technique (reference the applicable Organic Sample Prep SOPs listed below). The extract is exchanged into hexane and concentrated to a final volume between 1 and 20 ml depending upon the prep technique used.

2.1.1. Aqueous and leachate samples are extracted at a neutral pH using SW846 Method 3510C (SOP No. ED-ORP-014: *Extraction of Pesticides and PCBs in Water by Separatory Funnel, SW846 Method 3510C).*

2.1.2. Wipe samples are extracted using SW846 Method 3550B: Sonication (*SOP No. ED-ORP-018: Extraction of Pesticides/PCBs in Soil Using Low-Level Extraction, SW846 Method 3550B*)

2.1.3. Solid samples are extracted using SW846 Method 3546 (*SOP No. ED-ORP-0044: Procedure for the Microwave Extraction of Solids,SW846 Method 3546*).

2.1.4. Organic liquids are prepared using SW846 Method 3580A (*SOP No. ED-ORP-019: Waste Dilution for Pesticides and PCBs, SW846 Method 3580A*).

2.1.5. Extract cleanup steps are employed as need depending on the nature of the matrix interferences encountered. Suggested cleanups include SW846 Method 3620B (*SOP No. ED-ORP-020: Florisil Cleanup for Pesticide/PCB Sample Extracts*), SW846 Method 3660B (*SOP No. ED-ORP-021: The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts*) and SW846 Method 3665A (*SOP No. ED-ORP-022, Sulfuric Acid Cleanup for PCB Extracts, SW846 Method 3665A*) for heavy organic interferences.

2.2. After cleanup, the extract is analyzed by injecting a known volume of sample into a gas chromatograph equipped with a dual wide-bore fused silica capillary columns and dual electron capture detectors (GC/ECD). The GC is temperature programmed to separate and detect the analytes recovered during the extraction step. Quantitation is accomplished by comparing the area response of each target analyte relative to an internal standard established through a five-point initial calibration (six points for second order regression). Specific calibration and quality control steps are detailed in this SOP and meet the specification of SW846 Method 8082A.

2.3. Samples are analyzed only after all the necessary calibration and QC checks have been performed.

2.4. Acquired data from sample analysis is manually reviewed. Secondary column confirmation of target compounds and quantitation are conducted by the analyst as required.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Interferences from phthalate esters introduced during sample preparation can pose major difficulties for PCB determinations.

4.1.1. Interferences from phthalate esters can be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

4.2. The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting PCBs. Sulfur contamination should be expected with sediment samples. Employ SW846 Method 3660B (*SOP No. ED-ORP-021: The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts*) for removal of sulfur.

4.3. Co-eluting chlorophenols are eliminated by using SW846 Method 3620B (*SOP No. ED-ORP-020: Florisil Cleanup for Pesticide/PCB Sample Extracts*),

4.4. Interferences from other organic compounds can effectively be removed using a sulfuric acid treatment, SW846 Method 3665A (*SOP No. ED-ORP-022, Sulfuric Acid Cleanup for PCB Extracts, SW846 Method 3665A*). This destructive technique can be employed only when the sample extract is being analyzed solely for PCBs (i.e., it is not to be used prior to analysis for pesticides).

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum

5.1. Specific Safety Concerns or Requirements

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

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 MSDS 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 MSDS for each material before using it for the first time or when there are major changes to the MSDS.

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.
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.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-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.			

6.0 Equipment and Supplies

6.1 Gas Chromatograph:

6.1.1 Agilent Technologies (Avondale, PA) model 5890/6890 Gas Chromatograph (GC), equipped for simultaneous quantitation and confirmation columns using two separate detector channels on dual megabore capillary columns that are suitable for the analysis of organochlorine pesticides and PCB's. All operations are as automated as possible with the equipment utilized.

6.1.2 Injection system: Sample injection is accomplished by a single auto injector. The auto injector is serviced by a robot arm that shuttles a single sample between the sample tray and the injector turret.

6.1.2.1 The sample is injected into a split/splitless injection port equipped with electronic pressure control (EPC). The injection port is normally operated in splitless mode during injection. The EPC is operated in the ramp pressure mode.

6.1.3 Liners: The injection port is each fitted with a replaceable, heavy-walled glass double gooseneck liner. The liner contains a plug of silanized glass wool approximately 1 cm in length. The glass wool is positioned in the liner between the double gooseneck. The liner is replaced on a regular maintenance schedule.

6.1.4 Oven and Columns: Temperature programmable gas chromatograph ovens are required, capable of integrated temperature control between 35^{O}C and 350^{O}C.

6.1.4.1 Two dissimilar columns are used for analysis. A Restek StxCLPesticides, 30m x 0.53mm ID x 0.5um film thickness column (or equivalent) is used for sample quantitation. The secondary column is a Restek StxCLPesticides II, 30m x 0.53mm ID x 0.42um film thickness column (or equivalent).

6.1.5 Detectors: Sample detection is by electron capture. The GC is equipped with dual Electron Capture Detectors (ECD), one for each column.

6.1.5.1 Each detector is supplemented with make-up gas to provide sufficient detector flow for maintaining the electron plasma. This is in addition to the gas exiting the column. The make-up gas is fed from a supply other than the injection port.

6.2 Data System:

6.2.1 The data systems consist of Agilent Technologies GC Chemstation Revision A.08.02 and Agilent Technologies Enviroquant Chemstation G1701AA Version A.03.00 upgraded to A.03.02 which is used for

acquisition and TestAmerica Chrom (chromatography data processing software).

7. Reagents and Standards

7.1. Reagents

7.1.1. Gases: Ultra high purity (99.999%) Hydrogen is used as the carrier and injection port purge gas. It is introduced to the GC at the injection port. Ultra high purity (99.999%) Nitrogen is used as make-up gas. (Alternatively, ultra-high purity Helium with P-5 make-up gas may be used). Make-up gas is introduced to the GC via the make-up gas adapter at the end of the capillary column. Gases are supplied at tank pressures of 2000-2400 psig for a 300 cft tank. The tank pressure is regulated to an outlet pressure of 70 psig. Each tank is used until the tank pressure drops to less than 500 psig.

7.1.1.1. The gas streams are polished using three traps or filters before introduction to the G.C. The traps are as follows:

- Hydrocarbon trap
- H_2O (moisture) trap
- O_2 scrubber

7.1.1.2. Both the moisture trap and the Oxygen scrubber are of the indicating type. They require either replacement or reconditioning upon color change of the active agents. Refer to the instructions for the individual traps to determine if it is still active. The hydrocarbon trap is a simple activated carbon trap. With high quality gas, it should last for an extended period of time (1-yr. minimum).

7.1.2. Solvents used in the extraction, clean up procedures and dilutions include Hexane, Methylene Chloride, and Acetone that are exchanged to Hexane prior to analysis. All solvents must be pesticide quality or equivalent. Each lot of solvent is screened for contaminants before being used for analysis.

7.2. Standards

7.2.1. Standards are purchased as concentrated solutions. Standard compounds or mixtures for this analysis include an Aroclor 1016/1260 mix, Aroclor 1221, 1232, 1242, 1248, 1254, 1262, 1268 and the surrogate compound Decachlorobiphenyl (DCB) (packaged with the Tetrachloro-m-xylene (TCMX), a surrogate used in pesticide analysis).

NOTE: Two independent sources are used for quantitation standards and spiking standards

7.2.1.1. Most stock solutions are diluted (in volumetric glassware) to working concentration using hexane as the diluent.

7.2.2. Standards mixes and sources: *

Standard Name	Source	Concentration
TCMX/DCB Surrogate Calibration Mix	Restek 32000	200 ug/ml
TCMX/DCB Surrogate Spike Mix	Supelco 861275	10 ug/ml
Aroclor 1016 Calibration Standard	Supelco 48097	1000 ug/ml
Aroclor 1221 Calibration Standard	Restek 32007	1000 ug/ml
Aroclor 1232 Calibration Standard	Restek 32008	1000 ug/ml
Aroclor 1242 Calibration Standard	Restek 32009	1000 ug/ml
Aroclor 1248 Calibration Standard	Restek 32010	1000 ug/ml
Aroclor 1254 Calibration Standard	Restek 32011	1000 ug/ml
Aroclor 1260 Calibration Standard		1000 ug/ml
Aroclor 1262 Calibration Standard	Restek 32409	1000 ug/ml
Aroclor 1268 Calibration Standard	Restek 32410	1000 ug/ml
Aroclor 1660 Mix (Arolcors 1016 & 1260)	Restek 32039	1000 ug/ml
Aroclor 1016/1260 Calibration Standard (Second Source)	Restek 32039.sec	1000 ug/ml
1-Bromo-2-nitrobenzene (internal standard)	Restek 32279	1000 ug/ml

*Suppliers with equivalent standards may be used.

7.2.3. Aroclor 1016/1260 & Surrogate Calibration Standard Solution Preparation

Five levels of calibration standards are prepared using the above referenced Aroclor 1660 Mix (Restek – 23039) and TCMX/DCB Surrogate Calibration standard mix (Restek 32000). They are prepared as follows:

Final Concentration of Aroclor 1016/1260 (Concentration of DCB)	Volume (ul) of Aroclor 1660 Mix (1000 ug/ml)	Volume (ul) of TCMX/DCB Surrogate Calibration Mix (200 ug/ml)	Final Volume in hexane (ml)
50 ppb (25 ppb DCB) (1)	5	6.25	100
500 ppb (50 ppb DCB)	50	25	100
1000 ppb (100 ppb DCB)	1000	500	1000
1500 ppb (150 ppb DCB)	150	75	100
2500 ppb (200 ppb DCB)	250	100	100

(1): The low level Aroclor 1016/1260 standard is 50 ppb and 12.5 ppb for the surrogate DCB (prepare by making a 2x dilution of the 100 ppb standard in hexane).

7.2.4. Surrogate Spiking Solution (soil and water)

A TCMX/DCB Surrogate Spike Mix is prepared by diluting 10 ml of Restek 32000 (see Table 1 above) to 200 ml of Acetone. Final solution concentration is 10 ug/ml. For reduced volume LVI preps a secondary dilution of this mix is utilized. This is prepared by diluting 20 ml of the 10 ug/ml solution to 100 ml acetone with a final solution concentration of 2 ug/ml.

7.2.5. Aroclor 1016/1260 Spiking Solution (soil, water and wipe)

An Aroclor 1660 Mix is prepared by diluting 10 ml of Restek 32039 (See Tables 1 and 2 above) to 100 ml acetone. Final solution concentration is 100 ug/ml. For reduced volume LVI preps a secondary dilution of this mix is utilized. This is prepared by diluting 20 ml of the 100 ug/ml solution to 200 ml with a final solution concentration of 20 ug/ml. used spiking soils and waters as received from Supelco without further dilution.

7.2.6. Individual Aroclor Calibration Solutions (1221, 1232, 1242, 1248, 1254, 1262 & 1268)

A 1000 ppb calibration standard is prepared for each remaining Aroclor from the stock standards detailed in Section 7.2.1. 200ul of 1000 ug/ml individual Aroclor solution and 100ul of 200 ug/ml TCMX/DCB is diluted to 200ml with Acetone. The final concentration of surrogates is 100 ppb.

7.2.7. Aroclor 1016/1260 Initial Calibration Verification (ICV) Standard Solution Preparation

A mid-point Aroclor 1016/1260 ICV standard is prepared using the second source Aroclor 1016/1260 Calibration Standard (Restek 32039.sec) detailed in Section 7.2.2. 1000 ul of 1000 ug/ml standard along with 500 ul of 200 ug/ml TCMX/DCB surrogate standard (Restek 32000) is diluted to 1000 ml with acetone for a final ICV concentration of 1 ug/ml (1000 ppb).

7.2.8. PCB Internal Standard Spike Mix (1 ug/ml)

The PCB 1 ug/ml internal standard spike mix is prepared by dilution 500ul of 1000 ug/ml of the 1-Bromo-2-Nitrobenzene standard (Restek 32279) in to 500 ml of Hexane. 20 ul of this solution is added to all standards, QC samples and field sample extracts prior to analysis.

8. Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Amber glass, 250 ml	250 ml	Cool 4 $\pm$ 2°C	7 days to extraction; Analyze within 40 days of extraction	SW846
Soils	Glass, 2 or 4 oz	100 g	Cool 4 $\pm$ 2°C	14 days to extraction; Analyze within 40 days of extraction	SW846

.

8.1. Extracts must be stored under refrigeration in the dark and analyzed within 40 days of extraction.

9. Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]
Internal Standard	every sample[3]	Response within -50% to +100% of most recent cal standard.

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into LIMS.

9.1.1. **Method Blanks** are extracted with each sample batch on each day that samples are extracted. The analytical results for the method blank must fall below the reporting limit for each compound of interest. If a target compound is detected in the blank at a concentration higher than the reporting limit, first the extract is reanalyzed for confirmation. If results are still outside of limits the entire batch of samples extracted with the affected blank must be re-extracted and reanalyzed.

9.1.2. **Laboratory Control Sample (LCS):** A Laboratory Control Sample (LCS) or blank spike must be extracted and analyzed for with each batch of 20 environmental samples. The LCS data is used to assess method performance and serves to determine whether the methodology is in control at the time of preparation and analysis. The recoveries of the LCS must fall within lab generated acceptance criteria. If the LCS recovery results are outside QC limits, the extract is reanalyzed. If upon reanalysis the recoveries remain outside of recovery limits the following evaluations are made:

- If LCS results fall outside the laboratory generated limits with low recoveries (refer to the current TALS Method Limit Group database), the LCS and all associated samples should be re-extracted and re-analyzed.

- If LCS results fall outside the laboratory generated limits with high recoveries (refer to the current TALS Method Limit Group database), the LCS and associated sample results may be reported with an Non-Conformance Memo (NCM) detailing the issue.

9.1.3. **Matrix Spike/Matrix Spike Duplicate (MS/MSD):** A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. MS/MSD recoveries are evaluated against lab generated limits (refer to the current TALS Method Limit Group database). If the MS/MSD recovery limits fall outside of lab limits the LCS recovery is evaluated. If the LCS recoveries meet criteria the data is reported and a Non-Conformance Memo (NCM) is written.

9.1.4. **Surrogate Standards:** All samples, blanks and QC samples are spiked with a 2 component surrogate standard mix containing TCMX & DCB (see Section 7.2). The percent recovery of the DCB surrogate standard is calculated and compared to lab generated limits (refer to the current TALS Method Limit Group database). (Note: the surrogate must pass CCV criteria to be reportable and must be reported from the same column as sample target analyte results). If the DCB recovery is outside of acceptance limits the sample extract is reanalyzed to confirm. If the recoveries are still outside of limits the sample must be re-extracted and reanalyzed or the data flagged as “estimated concentration”.

9.1.5. **Internal Standard:** The internal standard (1-bromo-2-nitrobenzne) must elute within 30 seconds of and have an area response of 50 to 100% as compared to the most recent preceding calibration standard.

9.2. Instrument QC

9.2.1. Initial Calibration Range and Initial Calibration Verification (ICV)

9.2.1.1. ***Initial Calibration Range*:** Aroclors 1016/1260 and the surrogate (DCB) are calibrated using a five-point calibration range using a minimum of five (5) peaks per Aroclor. The reporting limit (RL) is equal to the low point of the calibration range. The initial calibration block must include at least one level with Aroclor 1016 analyzed separately for pattern recognition purposes (note: this run does not need to be part of the actual calibration). If the 1016/1260 calibration meets the required average RF criteria all other Aroclors are then calibrated at the anticipated midpoint of the calibration range with a single point calibration using a minimum of five (5) peaks for each Arolcor (minimum of 3 peaks for Aroclor 1221). All peaks selected must be at least 25% the peak height of the largest peak in the Aroclor (except for Aroclor 1268 where the requirement is 10%). The following Aroclors can be analyzed

together: 1016/1260, 1221/1254, 1232/1262 and 1242/1268. Standards are prepared following the instructions in Section 7.2.

9.2.1.2. ***Initial Calibration Verification (ICV):*** An Initial Calibration Verification (ICV) standard is analyzed immediately after the Initial Calibration Range and before any samples are analyzed. The ICV is prepared as detailed in Section 7.2.7 and must be from a source separate from the standards used in the Initial Calibration Range.

9.2.2. Continuing Calibration Verification (CCV): A mid-point Continuing Calibration Verification (CCV) standard (typically Aroclors 1016/1260) must be analyzed after every 20 samples at minimum. If the samples being analyzed are being specifically targeted for a different Aroclor, that Aroclor may be analyzed as the CCV instead of 1016/1260. If an Aroclor other than Aroclors 1016/1260 is detected a CCV of the identified Arolcor must be analyzed within 12 hours of the sample. If that CCV does not meet criteria the sample must be reanalyzed under an acceptable CCV using the detected Arolcor. The response factors for the CCV must be within +/- 20 % of the initial calibration RF.

9.2.3. Calibration Acceptance Summary

9.2.3.1. Retention Time Windows: Retention time (RT) windows must be determined for all analytes.

9.2.3.1.1 Initial determination of RT windows.

9.2.4.1.1.1. The center of the retention time (RT) window shall be updated based on the middle level in the initial calibration of the first CCV in the daily sequence, whichever is most recent.

9.2.3.1. Initial Calibration Range. The internal standard calibration technique is employed for this method. The response factor (defined as the ratio of the area to the standard concentration) is calculated for each characteristic peak in the Aroclor 1016/1260 standard at each calibration concentration. The percent relative standard deviation (% RSD) of the response factors for each individual peak in the Aroclor 1016/1260 mix on each column is then determined (both columns must pass calibration criteria).

9.2.3.1.1. Calculate the percent Relative Standard Deviation of the response factors for each compound at each level:

% RSD = (RF Standard Deviation/RF Mean) X 100

RF = Response Factor

9.2.3.1.2. Linear Calibration: If the % RSD is less than 20% over its working range for at least five peaks in the Aroclor 1016/1260 mix, the linearity of the range is assumed for all Aroclors over the same analytical range. Each individual peak's response factor is used for quantitation of all the samples and verification standards. The average of the value calculated for each individual peak is used to report the concentration in the samples.

9.2.3.1.3. Linear Calibration Using Least Squares Regression: If the % RSD is >20% for any given compound, a first order linear regression can be applied to the data to calculate the calibration curve and determine sample concentration. If this method is employed, the r squared value must be > 0.99 for the calibration to be acceptable

9.2.3.2. Initial Calibration Verification (ICV):

9.2.3.2.1. After the initial calibration range has been analyzed, an Initial Calibration Verification standard is analyzed on each column to verify the validity of the initial calibration range. The ICV standard must be from a standard lot independent of the standards used in the initial calibration range. The verification standard for PCBs is the mid-range Aroclor 1016/1260 standard at 1000ppb. (See Section 7.2.7 for details on the preparation of the ICV).

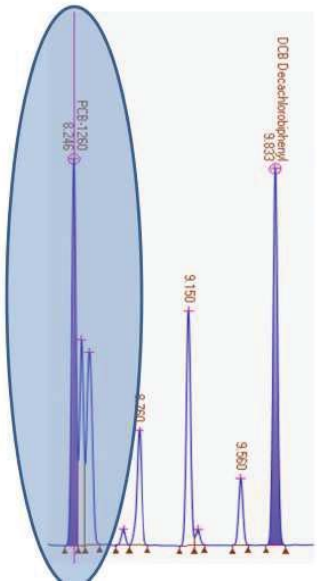
9.2.3.2.2. At least five characteristic peaks of each Aroclor 1016/1260 plus surrogates in the ICV must be checked to verify the Initial Calibration Verification. The calculated concentration of the ICV must be within ±20%D of the expected concentration. Should the %D exceed 20%, the analyst should take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the ICV. If the %D still exceeds 20% after a single ICV reinjection, a new Initial Calibration Range must be analyzed.

9.2.3.3. Continuing Calibration Verification (CCV):

9.2.3.3.1. A mid-point Continuing Calibration Verification (CCV) standard (typically Aroclors 1016/1260) must be analyzed after every 20 samples at minimum. If the samples being analyzed are being specifically targeted for a different Aroclor, that Aroclor may be analyzed as the CCV instead of 1016/1260.

9.2.3.3.2. If there are Aroclor hits in the associated samples then the calculated concentration of the CCV must be within ±20%D of the expected concentration on both columns. If there are no Aroclor hits in the associated samples one column may exceed the ±20%D criteria on the hight side (alternatively a low level Aroclor 1016/1260 standard may be analyzed to demonstrate adequate sensitivity. Should the foregoing criteria not be met the analyst must take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the CCV. If the %D still exceeds 20% after a single CCV reinjection, a new Initial Calibration Range must be analyzed. The surrogate (DCB) must also meet CCV criteria on both columns.

9.2.3.3.3. Resolution (Aroclor 1260): The CCV level Aroclor 1016/1260 standard must meet the following resolution critieria: Resolution (degree of overlap) for the triplet towards the end of the 1260 chromatogram must be <75% on <u>one</u> of the two columns used. The resolution requirement must be met for peak 1/2 and peak 2/3:



The circled triplet of peaks is observed towards the end of the 1260 pattern on columns such as CLP 1. Minimum resolution (degree of overlap) requirement between peak 1 / 2 and peak 2 / 3 is <75%. This chromatogram shows overlap of about 50% between peak 2 and 3, and 30% between peak 1 and 2.

Resolution (degree of overlap) is calculated as

[Height of the valley / (Sum of the two peak heights / 2)] x 100%

The acceptance criteria for the Initial Calibration Range, the ICV and the CCV are detailed in the table below.

Step	Standards	Type	Control Limit	Frequency
Method # 8082A				
Initial Calibration Range	50, 500, 1000, 1500 and 2500 ppb for Aroclor 1016/1260, 1000 ppb for all remaining Aroclors	*Average response factor or 1st order linear regression*	*For average RF: <20%RSD all analytes. For linear regression: r^2 ≥0.990*	*Initially and as required when ICV or CCV do not meet requirements*
ICV	*1000 ppb*	*Average*	*± 20%D*	*Once after each initial calibration*
CCV	*1000 ppb*	*Average*	*± 20%D (see Section 9.2.3.4.2 for exceptions); Acceptable Resolution*	*Every 20 samples*

10. Procedure

10.1. Gas Chromatograph (GC) Operation

10.1.1. The sequence of events for GC analysis involves many steps. First the injection system and column performance and calibration must be verified. Maintenance operations are performed as needed. Then samples must be run on the instrument. Chromatograms and reports must be evaluated for content, integration and concentration. Re-runs and dilutions must be made based on the calibrations that were in effect at the time the sample was run. Lastly, a detailed analysis and calculations must be performed to determine the concentration of all the parameters for which the sample was analyzed.

10.1.2. General Instrument Operating Conditions:

10.1.2.1. **Injection System**: A splitless injection port with electronic pressure control (EPC) is used. Seventy-five seconds after sample injection, the purge valve is turned on to facilitate the sweeping of any remaining residual solvent/sample from the injection port.

10.1.2.2. The EPC is used in the pressure Ramp mode. The ramp pressure program is as follows:

Initial Pressure	InitialTime	Rate	Final Pressure	Hold
25 psi	0.50 min	20psi/min	15 psi	2.00 min
		8 psi/min	12 psi	6.60 min
		10.0 min	16 psi	2.00 min

10.1.2.3. For PCB analysis the normal operating conditions of the injection port are as follows:

Injection Temperature:	250^{o}C
Injection Port Pressure: Column flow:	25ml/min 33.2 ml/minute
Split vent flow:	60.0 ml/minute
Purge vent flow:	1.2 ml/minute
EPC:	Ramp pressure mode

10.1.2.4. In addition to the EPC, the injection port is also equipped with a siltek-coated glass double goose neck liner that contains a 1 cm glass wool plug. This liner/glass wool combination provides many functions.

10.1.2.5. The glass wool serves as a heat sink rapidly vaporizing solvent and samples resulting in higher response factors. The liner also protects the column head from accumulation of high boiling residuals and particulates.

10.1.2.6. Regular maintenance is performed on the injection port. When the glass wool/liner is changed, the septa also must be changed. Injection port, oven and detector temperatures are lowered to ambient prior to "cracking" the system. This is so as to introduce a minimum of damaging oxygen molecules into the system.

10.1.2.6.1. After the system has cooled, the old liner is removed. The injection port should be checked for particulate residues and cleaned as needed. A flashlight is usually required for this. After a new liner has been prepared it is placed into the injection port. A graphite seal is placed around the liner. The edges of the seal must be flat, not knife-edged, and free of nicks or burrs. If any of these conditions are not met, the graphite seal must be replaced as well. The graphite seal is critical to proper operation of the injection port. If in doubt, replace it.

10.1.2.6.2. The locking ring on the top of the injection port should be turned, with the wrench, about 1/8 turn past finger tight. The septum nut should never be tightened more than finger tight. After the injection port is reassembled, all column nuts inside the oven should be checked for leaks using Snoop (Supelco) or another suitable leak tester.

10.1.2.6.3. The septa should be changed each time the injection port is opened. Another routine maintenance operation to improve column performance is the removal of the first 3 cm of the column.

10.1.2.6.4. Once the signal from both detectors has stabilized, it is time to re-heat the zones. The zones should be heated in the order of detectors, oven and then injectors. This is to ensure that volatilized contaminants do not condense on the column or detector.

10.1.2.7. Oven: With the megabore columns installed, temperature programming is employed to achieve higher resolution of compounds and shorter run times than could be accomplished using isothermal methods.

10.1.2.7.1. The oven program and pressure ramping for PCB analysis is employed for all columns as follows:

Initial Temp	Hold Time 1	Rate1	Temp1
164°C	0.0min	12°/min	234°C

Hold Time2	Rate2	FinalTemp	FinalTime
2.4 min	40°/min	325°C	1.5min

10.1.2.8. If the detectors are particularly contaminated, they must be sent to Agilent Technologies in Avondale, Pennsylvania for reconditioning. This should occur if the detector baseline is greater than 100 Hz. Detector reconditioning should be required at a maximum of biannually.

10.1.2.9. Chemstation: The Chemstation is utilized for automation of runs and acquisition. The system is dedicated to a single GC and does not multitask. Therefore, data manipulation cannot be done while sample analysis is in progress. The data system acquires and stores all chromatographic data.

10.1.2.10. TestAmerica Chrom data processing software is used for the processing of the chromatography data files. Calibrations, verification standards and samples are processed and reviewed using this database. Chrom is integral to TestAmerica LIMS (TALS) which is used to generate all reports.

10.2. Analytical Sequence

10.2.1. The instrument operating conditions should be set as detailed in Section 10.1.

10.2.2. Once instruments conditions have been established, the Initial Calibration Range, calibration verifications and retention time windows must be established Section 9.2.

10.2.3. The analytical sequence is established via the "SEQUENCE" macro of the Chemstation data system. The sequence file contains the name of Method file corresponding to the type of analysis to be performed, the range of samples to be run, and the number of injections per bottle. It is common practice to run the calibration and/or calibration verification standards, evaluate the instrument status, and, finally, (if all meet criteria) complete the Sample Table and Sequence File. If everything else is complete, the run is initiated using the START SEQUENCE soft-key of the SEQUENCE macro

10.2.4. An idealized analytical sequence including an Initial Calibration Range is presented in the table below.

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Idealized Analytical Sequence with Initial Calibration Range	
Injection Number	**Identification**
1	Hexane
2	Instrument Blank
3	Aroclor-1660 Level 1 Cal Std (50 ppb)
4	Aroclor-1660 Level 2 Cal Std (500 ppb)
5	Aroclor-1660 Level 3 Cal Std (1000ppb)
6	Aroclor-1660 Level 4 Cal Std (1500 ppb)
7	Aroclor-1660 Level 5 Cal Std (2500 ppb)
8	Aroclor-1221 Level 3 Cal Std (1000 ppb)
9	Aroclor-1232 Level 3 Cal Std (1000 ppb)
10	Aroclor-1242 Level 3 Cal Std (1000 ppb)
11	Aroclor-1248 Level 3 Cal Std (1000 ppb)
12	Aroclor-1254 Level 3 Cal Std (1000 ppb)
13	Aroclor-1262 Level 3 Cal Std (1000 ppb)
14	Aroclor-1268 Level 3 Cal Std (1000 ppb)
15	Initial Calibration Verification (Aroclor 1660)
16	Hexane
17	Continuing Calibration Verification (Aroclor1660)
18 thru 37	Client samples and QC Samples (MS/MSD, LCS, Method Blank)
19	Continuing Calibration Verification (Aroclor1660) (*every 20 samples*)

10.2.5. After each 20 samples a CCV standard mix must be analyzed. If this standard fails the criteria listed in Section 9.2.3.4, all samples analyzed during the previous period must be re-analyzed with a passing CCV.

10.2.6. PCB Data Reporting: The Chrom data system calculates the concentrations of the selected Aroclor Peaks. The reporting limit is based on the concentration of the lowest standard in the initial calibration, adjusted for the sample wt/vol, final volume, dilution factor and % moisture (No unqualified analytical results or non detects may be

reported which correspond to an extract concentration less than the lowest standard in the calibration range).

10.2.7.1. The quantitative values for all confirmed analytes must agree within 40% between the primary column and the confirmation column.

10.2.7.2. If the quantitative values do not agree within 40%, the discrepancy must be noted in the report with a qualification

10.3 Dual Column Approach

NOTE: Data generated under the NJDEP DKQP requires the reporting of the higher concentration in all cases unless it can be demonstrated that interfering compounds are the cause of the higher results in which case the lower value can be reported with a narrative explanation. Other programs may also require a dual column reporting approach different than the one described below in which case the lab will report as required by that program.

10.3.1. The laboratory designates the rear column as the primary column and the front column as the secondary column. Results are reported from the primary column unless the difference in concentration between the two columns results in ≥40% RPD in which case the lower concentration is reported (unless the client or program requirements dictate otherwise).

10.3.2. The values are calculated from the chromatographic peaks that fall within the daily retention time windows established from the most recent preceding calibration verification.

10.3.3. If the calculated values are greater than 40% RPD of each other, report the lower concentration regardless of whether that result is from the primary or secondary column. Report the result with a flag of P*.

10.3.4. If the surrogates on one column are very different (>40% RPD) compared to the other column, this may be indicative of a bad injection or columnar blockage. The sample should be reanalyzed. If similar results are obtained following reanalysis, report the lower of the two numbers and describe the circumstances in the job summary and report case narrative.

10.3.5. If one of the columns fails CCV criteria (but the CCV is between 15%-40% greater than expected value), the sample results shall be reported from the compliant column. If the recovery falls outside of acceptance criteria on the low side, reanalysis shall be performed.

10.3.6. If the CCV on one of the columns is more than 40% different from the correct value, it can be assumed that there has been significant drift on that column. The sample shall be reanalyzed against an acceptable calibration.

10.3.6.1. An exception to this requirement would be if the CCV recovery on one column fails on the high side and >40% RPD but the

associated samples were non-detect for all target analytes on both columns. In this case the non-detect results may be reported from the compliant column.

10.3.7. In some cases where the sample chromatography is complex and has largely varying peaks concentrations, the chromatographic separation may not be sufficient on the 0.53mm ID columns. In this case a confirmatory analysis on an instrument with 0.32 ID columns may be required. The supplemental data produced using analysis on the 0.32mm ID 'microbore' column may minimize overlapping and baseline interference difficulties, and better resolves potential positive identifications. Use of this alternative chromatographic technique shall be noted in the job summary and report case narrative.

10.3.8. In summary, the flow chart in Attachment 1 presents a recommended approach to selecting the better number to report for dual column data. It shall be noted that these recommendations may be overridden by project specific requirements and that they cannot cover all eventualities. The complexity of some data set will require the final decision to be made utilizing the judgment of experienced analysts. In some cases further cleanup steps to remove interferences may be appropriate.

10.4 Extract Cleanup

10.4.1. Cleanup methods are dictated by the original sample matrix and the parameters being determined.

10.4.2. Cleanup of all water samples, if needed, is performed using Sulfuric Acid Permanganate and/or TBA sulfite. Refer to TestAmerica Edison SOP No. ED-ORP-021: *The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts, SW846 Method 3660B,* most current revision and TestAmerica Edison SOP No. ED-ORP-022: *Sulfuric Acid Cleanup for PCB Extracts, SW846 Method 3665A*, *SW846 Method 3665A,* most current revision. Blanks must also undergo cleanup following the same procedures as samples..

10.4.3. Cleanup of all soil samples is conducted using TBA sulfite and Sulfuric Acid Permanganate. Blanks must also undergo cleanup following the same procedures as samples.

10.4.4. Cleanup using Sulfuric Acid Permanganate effectively destroys the majority of organic material in the sample extract and should be used only when PCB is the only analysis to be performed on the sample extract.

10.5 Documentation

10.5.1 Before the analysis sequence is initiated the GC Performance and Repairs logbook must be filled out. It should contain the following information: date, injector temp, oven temp, detector temp, column A

flow, column B flow, signal A, signal B, analysts initials, and notes for any necessary repairs.

10.5.2 After samples have been run, each standard and sample must be entered into the Instrument Run Log. The Instrument Run Log should contain the following information: run date, data file name, vial position, sample number, initial volume/weight, final volume, dilution factor method, job number, QA number, extraction date, lab prep batch, target batch signature of analyst at the bottom of each page, lot numbers for standards used, and result of run (O.K., dilution, non-inject, etc.).

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Calculation of Sample Amounts (Internal Standard Procedure)

11.3.1 Aqueous Samples

$$\text{Concentration (µg/L)} = \frac{(As)(Cis)(D)}{(Ais)(RF)(Vs)\,(Vi)\,(1000)}$$

Where:

As	=	Area of the target analyte peak in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the internal standard peak
RF	=	Average response factor from the initial calibration.
Vs	=	Volume of sample extracted (ml)

The 1000 in the denominator represents the number of ul in 1 ml.

11.3.2 Solid Samples

$$\text{Concentration } (\mu g/kg) = \frac{(As)(Cis)(D)(Vt)}{(Ais)(RF)(Ws)\,(Vi)\,(1000)}$$

Where:

As	=	Area of the target analyte peak in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the internal standard peak
RF	=	Average response factor from the initial calibration.
Vt	=	Volume of concentrated extract (ul)
Ws	=	Weight of sample (g)

The 1000 in the denominator represents the number of ul in 1 ml

11.3.3 Wipe Samples

$$\text{Concentration } (\mu g/kg) = \frac{(As)(Cis)(D)(Vt)}{(Ais)(RF)(W)\,(Vi)\,(1000)}$$

Where:

As	=	Area of the target analyte peak in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the internal standard peak
RF	=	Average response factor from the initial calibration.
Vt	=	Volume of concentrated extract (ul)
W	=	Wipe

The 1000 in the denominator represents the number of ul in 1 ml

11.4 Relative Response Factors

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

A_x = Area of target analyte peak
Ais = Area of internal standard peak
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

11.5 Percent Relative Standard Deviation (% RSD):

$$\% \text{ RSD} = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

11.6 Percent Difference (% D):

$$\overline{\%\ D} = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

Where: RRFc = RRF from continuing calibration

$\overline{RRF_i}$ = Mean RRF from current initial calibration

11.7 Percent Recovery (% R): Surrogates and Spikes

$$\text{Recovery (\%)} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}} \times 100$$

11.8 Dry Weight Correction: All solid samples must be corrected for dry weight using the following formula for dry weight determination

$$DW = \frac{Gd}{Gw} \times 100$$

Where:

DW	=	Percent % Dry Weight
Gd	=	Dry weight of selected sample aliquot
Gw	=	Wet weight of selected sample aliquot

Multiply the DW value times the wet weight of the sample extracted.

NOTE: All dry weight corrections are made in TALS at the time the final report is prepared.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, *Training*, for the laboratory's training program.

13.0. Pollution Control

13.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

13.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage

14.0. Waste Management

14.1. The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and

regulations. 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. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.2. The following waste streams are generated as a result of this analysis:

- Auto sampler vials and expired standards: These vials are collected in satellite accumulation within the instrument laboratory. The vials are then placed into a 55 steel open top drum in the waste room. When the drums are full, the drum will be collected by the waste vendor for disposal. This waste is treated for incineration.
  Teris Profile Number: 50016652
  Onyx Profile WIP Number: 282493

- Mixed Solvent Waste: Mixed solvent waste is collected in a small beaker inside the bench top hood. This waste is then transferred into the satellite accumulation container in the Organic Prep. Lab. on a daily basis. This material is transferred into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.
  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

  Teris Profile Number (incineration): 50016710
  Onyx Profile Number: (stabilization) 402535

15.0. References / Cross-References

15.1. United States Environmental Protection Agency, "Method SW8000C: Determinative Chromatographic Separations,", Test Methods for Evaluating Solid Wastes, SW846, Revision 3, March 2003.

15.2. United States Environmental Protection Agency, "Method 8082A, Organochlorine Pesticide by Gas Chromatography", Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 1, February 2007.

15.3. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4. TestAmerica Edison SOP No. ED-ORP-014: *Extraction of Pesticides and PCBs in Water by Separatory Funnel, SW846 Method 3510C,* most current revision.

15.5. TestAmerica Edison SOP No. ED-ORP-018*: Extraction of Pesticides/PCBs in Soil Using Low-Level Extraction, SW846 Method 3550B,* most current revision.

15.6. TestAmerica Edison SOP ED-ORP-044, *Procedure for the Microwave Extraction of Solids, SW846 3546*

15.7. TestAmerica Edison SOP No. ED-ORP-019*: Waste Dilution for Pesticides and PCBs, SW846 Method 3580A,* most current revision.

15.8. TestAmerica Edison SOP No. ED-ORP-020*: Florisil Cleanup for Pesticide/PCB Sample Extracts*, SW846 Method 3620B, most current revision.

15.9. TestAmerica Edison SOP No. ED-ORP-021: *The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts, SW846 Method 3660B,* most current revision.

15.10. TestAmerica Edison SOP No. ED-ORP-022: *Sulfuric Acid Cleanup for PCB Extracts, SW846 Method 3665A*, *SW846 Method 3665A,* most current revision.

15.11. TestAmerica Edison SOP No. ED-GEN-022, *Training,* most current revision.

15.12. TestAmerica Corporate Work Instruction No. CA-T-WI-003, PCB Minimum Requirements, most current revision.

16.0. Method Modifications:

None

17.0. Attachments

Attachment 1: Dual Column Approach

18.0. Revision History

Revision 5 dated 01 Apr 2020

- Updated to Eurofins branding
- Section 7.2.5: added wipe matrix
- Added Section 11.3.3 – calculation for wipe samples

Revision 4 dated 15 Dec 2016

- Section 7.2.8: clarified text to more accurately describe the PCB Internal Standard Mix.

Revision 3 dated 16 May 2016

- Section 2.1: removed SW846 3550 as a prep method for solids and added it as a prep method for wipe samples.
- Section 2.1.1 and 15.0: removed reference to SW846 Method 3520C (SOP No. ED-ORP-003: *Extraction of Semi-Volatile Organic Compounds in Water by Continuous Liquid-Liquid Extraction, SW846 Method 3520C).*
- Section 2.2: expanded to include reference to internal standard calibration.
- Section 7.1.1: revised to clarify that the primary carrier:make-up gas combo is hydrogen and nitrogen.
- Section 7.2.2: added 1-Bromo-2-nitrobenzene (internal standard) to the list of standards (Restek 32279). Components and concentration added to the table
- Added Section 7.2.8 which describes the preparation of the internal standard solution.
- Section 9.1: added internal standard to 'Sample QC' table
- Section 9.1.2: completely re-written to clarify LCS evaluation and corrective action.
- Section 9.1.4: clarified that surrogates must pass CCV criteria in order to be reportable and must be reported from the same column as target analyte results.
- Added Section 9.1.5 which describes acceptance criteria for internal standards (retention time and response)
- Section 9.2.1.1: added following sentence for clarity and compliance with TestAmerica PCB Minimum Requirements document (WI No. CA-T-WI-003): "All other Aroclors are calibrated using a single point calibration up to 8 peaks at the anticipated midpoint of the calibration range if the 1016/1260 calibration meets the required average RF criteria." Added language detailing which Aroclor standards can be analyzed together.
- Section 9.2.1.1: added this sentence: "The initial calibration block must include at least one level with 1016 analyzed separately for pattern recognition purposes (note: this run does not need to be part of the actual calibration)."
- Section 9.2.1.1: clarified that a minimum of five peaks are required for quantitation of each Aroclor (except for Aroclor 1221 which requires a minimum of three peaks). Added requirement that selected peaks must be at least 25% of height of largest peak in Aroclor (except Aroclor 1268 where the requirement is 10%).
- Section 9.2.2: Revised first sentence to read: "A mid-point Continuing Calibration Verification (CCV) standard must be analyzed after every 20 samples at minimum," (i.e., removed the closing CCV requirement and 12 hour requirement).
- Section 9.2.2: clarified that the CCV typically consists of a 1016/1260 standard. Also added the following sentence: "If the samples being analyzed are being specifically targeted for a different Aroclor, that Aroclor may be analyzed as the CCV instead of 1016/1260."
- Section 9.2.2: added requirement to analyze a CCV for each detected Aroclor within 12 hours of detection.
- Table 2: updated with the expected RT acceptance criteria from Minimum Requirements document (0.03 minutes).
- Section 9.2.3.2: Revised 'External standard' to 'Internal Standard'. Emphasized that both columns must pass calibration criteria.

- Section 9.2.3.2.2: revised to require that 5 peaks must be selected (was 3) and evaluated for initial calibration (per corporate Minimum Requirements).
- Section 9.3.3.3.1: corrected section reference for preparation of ICV (was Section 7.2.9; now Section 7.2.7);
- Section 9.2.3.3.2: revised first sentence to read: “At least five characteristic peaks of each Aroclor 1016/1260 plus surrogates in the ICV must be checked to verify the Initial Calibration Verification.” (it had previously required three peaks).
- Section 9.2.4.3.1: updated text reflect corporate Minimum Requirements document.
- Added Section 9.2.3.4.2: more fully describes CCV acceptance criteria and corrective actions. Updated table with calibration criteria.
- Added Section 9.2.3.4.3: details resolution requirements per the corporate Minimum Requirements document.
- Section 10.2.5: revised to remove 12 hour requirement (replaced solely with 20 sample requirement) and bracketing requirement. Corrected section reference for CCV acceptance criteria.
- Section 11: completely re-written to include internal standard result calculations and other required QC calculations.
- Section 15 (References): added following document reference: TestAmerica Corporate Work Instruction No. CA-T-WI-003, PCB Minimum Requirements, most current revision.
- Attachment 1: revised completely.

- Revision 2, dated 08 Jun 2015

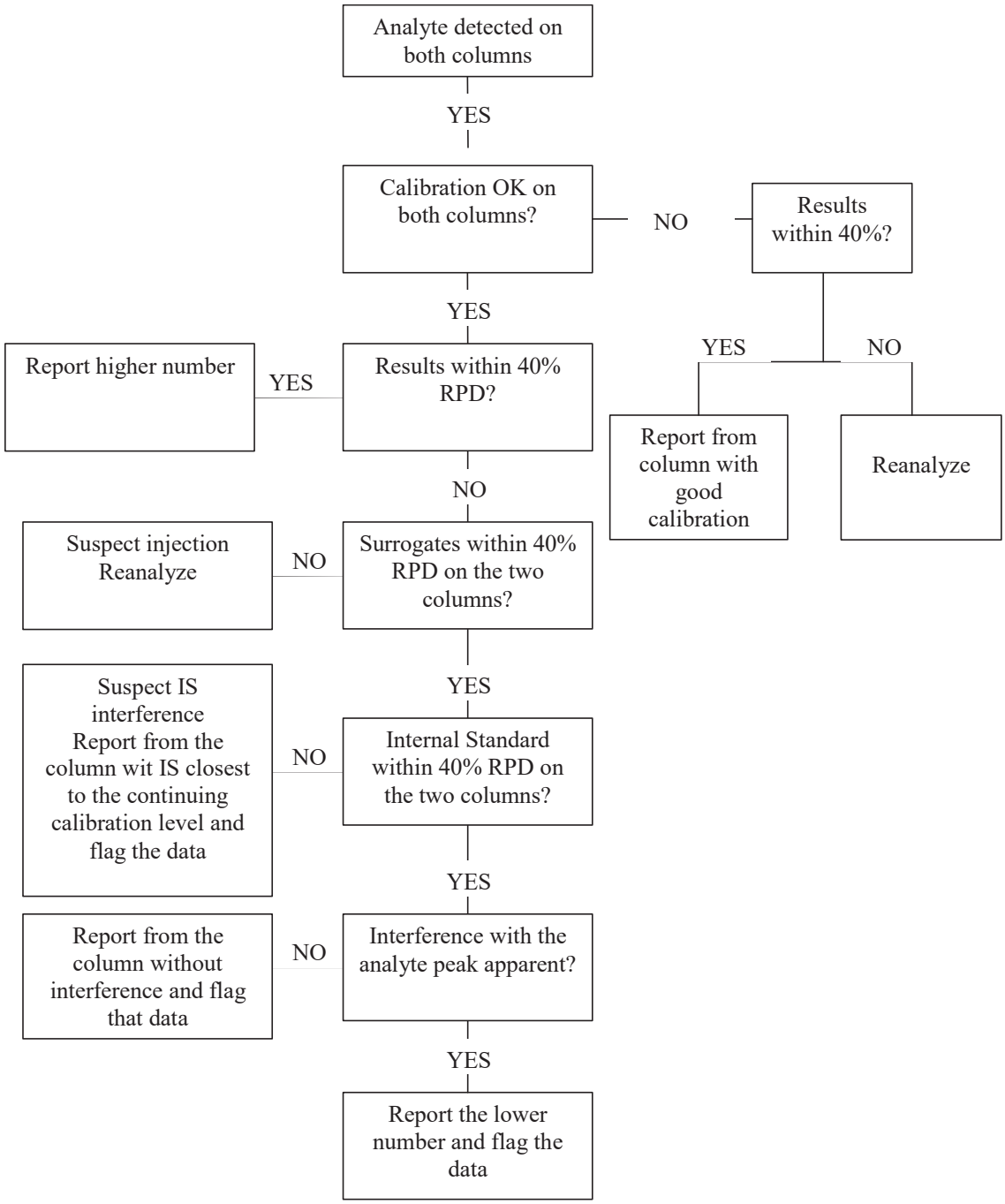
  - Section 1.1: Corrected Method Limit Group (MLG) reference to 8082 (was incorrectly listed as 8081B,
  - Table 2: updated RLs for leachates from 0.0050 mg/L to 0.00050 mg/L.
  - Throughout document: updated the default initial volumes for aqueous preps (250 ml) and leachate (TCLP/SPLP/ASTM) preps (250 ml).
  - Throughout document: removed any notes referencing option for ‘reduced volume’ extractions since this is now standard as defined in SOP.
  - Section 2.2.2: replaced SW846 3541 (Soxtherm) prep with SW846 3546 (Microwave) prep.
  - Section 6.2.1 (and throughout document): replaced Target software references with references to TestAmerica’s Chrom chromatography data processing software.
  - Section 7.2.2 and throughout document: updated source of standards from Supelco to Restek.
  - Section 7.2.3 and throughout document revised concentration of low calibration standard to 50 ppb.
  - Section 7.29: updated ICV prep instructions using Restek standards.
  - Section 8: updated sample container from 1000ml to 250ml; updated minimum sample size to 250ml.
  - Section 10.3: added note explaining NJDEP DKQP requirement to report the higher concentration in all cases.
  - Section 10.3 and Attachment 1: revised to reflect current TestAmerica dual column reporting rules.
  - Section 15: removed outdated references.

- Revision1, dated 11 October 2012

    - Throughout document: Revised LQM section references to reflect the most current LQM revision
    - Revised Table 2 to reflect RLs for reduced initial/final volume prep option.
    - Section 2.1.1: added description of reduced initial volume (125ml)/final volume (1ml) extraction option.
    - Section 2.1.2 and Section 15.0: Added reference to TestAmerica Edison SOP ED-ORP-044, *Procedure for the Microwave Extraction of Solids, SW846 3546.*
    - Section 7.2.3: added procedure for prep and analysis of a lower level Aroclor 1016/1260 standard (50 ppb) and for the surrogate DCB (12.5 ppb) when analyzing samples prepped using the reduced initial/final volume procedure.
    - Section 9.2.3.4.1: Added reference to 50 ppb 1016/1260 ICAL standard for the reduced initial/final volume method.
    - Section 10.2.4: Added 50 ppb 1016/1260 ICAL standard to the sequence for the reduced initial/final volume method

- Revision 0, dated 02/16/2011: NEW

Attachment 1
Dual Column Approach


Analyte detected on both columns
YES
Calibration OK on both columns?
NO
Results within 40%?
YES
NO
YES
Report higher number
YES
Results within 40% RPD?
Report from column with good calibration
Reanalyze
NO
Suspect injection Reanalyze
NO
Surrogates within 40% RPD on the two columns?
YES
Suspect IS interference Report from the column wit IS closest to the continuing calibration level and flag the data
NO
Internal Standard within 40% RPD on the two columns?
YES
Report from the column without interference and flag that data
NO
Interference with the analyte peak apparent?
YES
Report the lower number and flag the data

- Revision 1, dated 11/07/2011

    - Section 1.1, Table 1: Added Pentachloronitrobenzene and associated CAS# to the analyte list.
    - Section 7.2.1: Added Pentachloronitrobenzene standard information.
    - Table 2: Added Pentachloronitrobenzene to Working Standards preparation information.
    - Table 4: Added Pentachloronitrobenzene and associated minimum RF.
    - Table 8: Added Pentachloronitrobenzene and associated ions.

- Revision 0, dated 02/22/2011: NEW

Table 21
Characteristic Ions Of Semi-Volatile Organic Compounds

Compound	Primary Ion	Secondary Ion(s)
1,1'-Biphenyl	154	153,76
1,2,4,5-Tetrachlorobenzene	216	214, 179
1,2,4-Trichlorobenzene	180	182, 145
1,2-Dichlorobenzene	146	148, 111
1,2-Diphenylhydrazine	77	105, 182
1,3-Dichlorobenzene	146	148, 111
1,3-Dimethylnaphthalene	156	141, 115
1,4-Dichlorobenzene	146	148, 111
1,4-Dichlorobenzene d4 **(ISTD)**	152	150, 115
1,4-Dioxane	88	58, 43
1-Methylnaphthalene	142	141, 115
1-Naphthylamine	143	115, 116
2,2'-oxybis[1-chloropropane]	45	77, 121
2,3,4,6-Tetrachlorophenol	232	131, 230
2,3,7,8-TCDD (screen)	320	322, 324
2,3-Dihydroindene		
2,3-Dimethylaniline	106	129
2,4,5-Trichlorophenol	196	198, 200
2,4,5-Trimethylaniline	102	55, 56
2,4,6-Tribromophenol **(Surrogate)**	330	132, 141
2,4,6-Trichlorophenol	196	198, 200
2,4-Dichlorophenol	162	164, 98
2,4-Xylidine	121	120, 106
2,4-Dimethylphenol	122	107, 121
2,4-Dinitrophenol	184	63, 154
2,4-Dinitrotoluene	165	63, 89
2,6-Dinitrotoluene	165	63, 89
2-Chloronaphthalene	162	127, 164
2-Chlorophenol	128	64, 130

Table 21 Characteristic Ions Of Semi-Volatile Organic Compounds		
Compound	**Primary Ion**	**Secondary Ion(s)**
2-Ethylaniline	106	122,104
2-Fluorobiphenyl **(Surrogate)**	172	171
2-Fluorophenol **(Surrogate)**	112	64
2-Methylnaphthalene	142	141
2-Methylphenol	108	107
2-Naphthylamine	143	115, 116
2-Nitroaniline	65	108, 138
2-Nitrophenol	139	109, 65
2-tert-butyl-4-Methylphenol	149	121, 91
2-Toluidine	107	106, 77
3,3'-Dichlorobenzidine	252	254, 126
3,4-Dimethylaniline	106	129, 127
3,5-Di-tert-butyl-4-Hydroxytol	205	220, 145
3-Nitroaniline	138	108, 65
4,6-Dinitro-2-methylphenol	198	51, 105
4-Bromophenyl phenyl ether	248	250, 141
4-chloro-2-methylaniline	106	144, 142
4-Chloro-3-methylphenol	107	144, 142
4-Chloroaniline	127	129
4-Chloroaniline-d4 **(Surrogate)**	131	133
4-Chlorophenyl phenyl ether	204	206, 141
4-Methylphenol	108	107
4-Nitroaniline	138	108, 65
4-Nitrophenol	139	109, 65
Acenaphthene	154	153, 152
Acenaphthene d10 **(ISTD)**	164	162, 160
Acenaphthylene	152	151, 153
Acetophenone	105	77, 51
Aniline	93	66
Aniline-d5 (**Surrogate**)	98	71,42
Anthracene	178	176, 179
Atrazine	200	173,215
Benzaldehyde	77	105,106
Benzidine	184	92, 185
Benzo(a)anthracene	228	229, 226
Benzo(a)pyrene	252	253, 125
Benzo(b)fluoranthene	252	253, 125
Benzo(g,h,i)perylene	276	138, 277
Benzo(k)fluoranthene	252	253, 125
Benzoic Acid	122	105, 77
Benzyl Alcohol	108	79, 77
Bis(2-chloroethoxy)methane	93	95, 123
Bis(2-chloroethyl)ether	93	63, 95
Bis(2-ethylhexyl)phthalate	149	167, 279
Bisphenol-A	213	228, 119
Butyl benzyl phthalate	149	91, 206

Table 21 Characteristic Ions Of Semi-Volatile Organic Compounds		
Compound	**Primary Ion**	**Secondary Ion(s)**
Caprolactam	113	55,56
Carbamazepine	193	236, 135
Carbazole	167	166, 139
Chrysene	228	226, 229
Chrysene d12 **(ISTD)**	240	120, 136
Coumarin	146	118, 63
Dibenz(a,h)anthracene	278	139, 279
Dibenzofuran	168	139
Diethylphthalate	149	177, 150
Dimethylphthalate	163	194, 164
Di-n-butylphthalate	149	150, 104
Di-n-octylphthalate	149	167, 43
Fluoranthene	202	101, 203
Fluorene	166	165, 167
Hexachlorobenzene	284	142, 249
Hexachlorobutadiene	225	223, 227
Hexachlorocyclopentadiene	237	235, 272
Hexachloroethane	117	201, 199
Indeno(1,2,3-cd)pyrene	276	138, 227
Isophorone	82	95,138
Kepone	272	237, 355
N,N-Dimethylaniline	120	122, 104
Naphthalene	128	129, 127
Naphthalene d8 **(ISTD)**	136	68
n-decane	43	57
Nitrobenzene	77	123, 65
Nitrobenzene-d5 **(Surrogate)**	82	128, 54
N-Nitrosodimethylamine	42	74, 44
N-Nitroso-di-n-propylamine	170	42,101,130
N-Nitrosodiphenylamine	169	168, 167
n-Octadecane	57	43, 85
o-Toluidine-d9 **(Surrogate)**	114	112, 42
Pentachloronitrobenzene	237	214,295
Pentachlorophenol	266	264, 268
Perylene d12 **(ISTD)**	264	260, 265
Phenanthrene	178	179, 176
Phenanthrene d10 **(ISTD)**	188	94, 80
Phenol	94	65, 66
Phenol-d5 **(Surrogate)**	99	42, 71
Phenyl ether	170	77, 115
Pyrene	202	200, 203
Pyridine	79	52, 51
Terphenyl-d14 **(Surrogate)**	244	122, 212

Attachment 1
Poor Performing Compounds

1,2,4,5-Tetrachlorobenzene
1,4-Dioxane
1-Naphthylamine
2,3,4,6-Tetrachlorophenol
2,4-Dimethylphenol
2,4-Dinitrophenol
2-Chloroaniline
2-Naphthylamine
3&4-Methylphenol
3'3-Dichlorobenzidine
4,6-Dinitro-2-methyl- phenol
4-Chloroaniline
4-Nitrophenol
Aniline
Atrazine
Benzaldehyde
Benzidine
Benzoic Acid
Benzyl Alcohol
Biphenyl
Caprolactam
Diphenylamine
Hexachlorocyclopentadiene
Hexachloroethane
n-Decane
n-Nitrosodimethylamine
o,o,o-Triethylphosphorothioate
o-Toluidine
Pentachloronitrobenzene
Pentachlorophenol
Phenol
Pyridine

These analytes are exempt from the ICV and CCV criteria as detailed in this SOP

Title: Mercury Analysis for Water and Wastewater using EPA 245.1 and SW846 7470A; Mercury in Drinking Water using EPA 245.1; Leeman Mercury Analyzer (Cold Vapor Technique)

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

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	08/02/20		
Dan Helfrich Health & Safety Manager	Date		

1.0 Scope and Application

1.1. Analytes, Matrix(s), and Reporting Limits

EPA Method 245.1 and SW846 Method 7470A are applicable to the determination of mercury in water matrices. Mercury may be found in water in both inorganic and organic forms. Organomercury compounds must first be broken down to respond to the cold vapor atomic absorption technique.

The typical detection limit using a 30 ml sample size is 0.2ug/L Hg.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

A digested sample is analyzed using cold vapor atomic absorption. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 The addition of potassium persulfate during the digestion step can eliminate the possible interference from sulfide in the sample without affecting the recovery of inorganic mercury.

4.2 Copper may also be a potential interference although no effect has been observed for samples containing up to 10 mg/l total copper.

4.3 Samples that contain high levels of chloride have a potential to interfere due to a reaction that takes place during the oxidation step. During this step chloride is converted to free chlorine which absorbs light at 253.7 nm. The analyst must not allow the chlorine to be swept into the optical cell. The possibility of the chlorine interfering with the analysis can be minimized by using an excess of up to 7.5 ml hydroxylamine sulfate.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

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 MSDS 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 MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
a)Mercury b)(1,000 PPM in Reagent)	Oxidizer Corrosive Poison	0.1 mg/M^3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	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.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	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.
Potassium Permanganate	Oxidizer	5 Mg/M^3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1. Leeman Laboratories Inc. Hydra II AA Automated Hg Analyzer

6.1.2. Computer and Monitor with Leeman Envoy software

6.1.3. Block digestor (Environmental Express or SCP Science): Adjustable and capable of maintaining a temperature of 90 -95°C.

6.2 Supplies

6.2.1. 50 ml Hot Block Digestion Cups

6.2.2. 100 ml graduated cylinder

6.2.3. Eppendorf Pipettes and tips in various sizes

6.2.4. 100 ml volumetric flasks

6.2.5. 15 ml sample cups

6.2.6. 10 liter carboy container

6.2.7. Pump tubing:

- Sample, viton, blue tab
- Reductant, red tab
- Drain, blue tab
- Rinse, black tab

6.2.8. Drying Tube – Purchased pre-packed with Magnesium Perchlorate from Leeman Labs. Located prior to the optical cell.

6.2.9. Nitrogen or Argon supply - capable of producing 80 PSI.

7. Reagents and Standards

7.1. Reagents

Storage requirements: store at room temperature

Life of Reagent: - Concentrated acids: refer to manufacturer's instructions
- Laboratory prepared reagents and diluted acids: one year

Document prepared reagents in the Reagent module located in the TestAmerica Laboratory System (TALS).

7.1.1 Sulfuric acid - Concentrated (Trace Grade or Equivalent); store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.2 Nitric acid - Concentrated (Trace Grade or Equivalent)

7.1.3 Hydrochloric acid-Concentrated (Trace Grade or Equivalent)

7.1.4 Potassium Permanganate (ACS Grade); for stability information, refer to manufacturer's instructions.

7.1.5 Sodium Chloride (analytical reagent grade); for stability information, refer to manufacturer's instructions.

7.1.6 Hydroxylamine Hydrochloride (ACS Grade); for stability information, refer to manufacturer's instructions.

7.1.7 Stannous Chloride (ACS Grade); for stability information, refer to manufacturer's instructions.

7.1.8 Potassium Persulfate (ACS Grade); for stability information, refer to manufacturer's instructions.

7.1.9 Deionized water - 18 megohm minimum

7.1.10 10% Hydrochloric Acid- Add approximately 5 liters of deionized water into an 8 liter carboy container. Cautiously add 800 ml of concentrated HCl and bring the final volume up to 8 liters with deionized water.

7.1.11 10% Stannous chloride solution - Add 50 g of $SnCl_2$ to 500 ml 10% HCl solution.

7.1.12 Sodium chloride/Hydroxylamine Hydrochloride solution - Dissolve 120 g of NaCl and 120 g of hydroxylamine hydrochloride in deionized water and dilute to 1 liter using deionized water.

7.1.13 Potassium Permanganate ($KMnO_4$) 5% solution w/v - Dissolve 100 g of $KMnO_4$ in deionized water and dilute to 2 liters using deionized water.

7.1.14 0.15% Nitric Acid- Add approximately 5 liters of deionized water into an 8 liter carboy container. Cautiously add 12mL of concentrated HNO_3 and bring the final volume up to 8 liters with deionized water.

7.1.15 Potassium Persulfate ($K_2O_8S_2$) 5% solution w/v: Dissolve 50 g of potassium persulfate in 1 liter of deionized water.

7.2 Standards

Storage requirements: all standards are stored at room temperature
Shelf-life: Stock standards – refer to manufacturer's instructions
Intermediate standards – made fresh daily
Working standards – made fresh daily

(Note: expiration date must not go beyond the expiration date of the source stock).

Concentration: see Attachment 1 for example certificates of analysis (COA) for all of the standards mixes listed below. The COA lists the manufacturer's part number, certified concentration and shelf life.

Document standard preparation in TALS (see Section 11.5.2).

7.2.1 Stock Mercury Calibration (10 ppm Hg) - Purchase from SCP Science; store at room temperature; for stability information, refer to manufacturer's instructions.

7.2.2. Stock Mercury Quality Control Standard (10 ppm Hg) - Purchase from Inorganic Ventures; store at room temperature; for stability information, refer to manufacturer's instructions. This stock standard must be purchased from a second source vendor.

7.2.3. Intermediate Calibration Standard (DCAL-Int): Dilute 1 ml of Stock Mercury Calibration (Sec 7.2.1) to 100 ml with 0.15% HNO_3. The resulting solution will contain 100ppb Hg.

7.2.4. Intermediate Quality Control Standard (DQCS-Int): Dilute 1 ml of Stock Mercury Calibration Verification Standard (Sec 7.2.2) solution to 100 ml with 0.15% HNO_3. The resulting solution will contain 100ppb Hg.

7.2.5. Calibration Standard Preparation: Use six 50 ml hot block digestion cups to prepare the standards. Add small portion of 0.15% HNO_3 to each cup. Working in increasing order, spike the appropriate cup with 0.0, 0.06, 0.3, 0.6, 1.5, and 3.0 ml of working solution DCAL-Int (Sec 7.2.3). Bring to final volume of 30 ml and mix thoroughly. The corresponding concentrations are 0.0ppb, 0.2ppb, 1.0ppb, 2.0ppb, 5.0ppb, and 10.0ppb mercury respectively. For drinking water analysis, the 2.0ppb standard is also analyzed as the Maximum Contaminant Level (MCL) standard

7.2.6. Quality Control Standard (QCS) Preparation: Add a small portion of 0.15% HNO_3 to a 30 ml hot block digestion cup and spike 1.5 ml of DQCS-Int (Sec 7.2.4). Bring up to final volume and mix thoroughly. The resulting solution will contain 5.0 ppb of Hg.

7.2.7. Initial Calibration Verification (ICV) standard: Add a small portion of 0.15% HNO3 to a 30 ml hot block digestion cup and spike 1.5 ml of DQCS-Int (Sec 7.2.4). Bring up to final volume and mix thoroughly. The resulting solution will contain 5.0 ppb of Hg.

7.2.8. Continuing Calibration Verification (CCV) standard 5.0ppb: The 5.0 ppb

prepared from the calibration standards (Sec 7.2.5) is used as the Continuing Calibration Verification standard (CCV).

7.2.9. Maximum Contaminant Level (MCL) standard 2.0 ppb *for drinking water analysis:* The 2.0ppb standard prepared from the calibration standards (Sec 7.2.5) is analyzed as the Maximum Contaminant Level (MCL) standard.

7.2.10. Reporting limit check standard (CRI) 0.2 ppb: Use the 0.2 ppb prepared from the calibration standards (Sec 7.2.5) as the reporting limit check standard (CRI).

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [2]	Reference
Waters	P. FP, G[1]	250 ml	HNO_3, pH < 2 prior to shipment; if not, acidify upon receipt in lab; Cool 4±2°C	28 Days	40 CFR Part 136.3

[1] Polyethylene, fluoropolymer, glass
[2] Inclusive of digestion and analysis
[3] Acid preservation may be omitted for shipping; however, acid must be added upon receipt in the lab. Following acidification, mix the sample and hold for at least 16 hours for method 245.1 drinking water and 24 hours for non-potable water. Just prior to digestion or direct analysis, verify pH<2. If pH≥2, repeat steps (i.e., add acid, hold for 24hrs, verify ph<2).

9.0 Quality Control

9.1. Sample QC - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	**245.1**: < RL or; <10%sample concentration (whichever is greater) **7470A**: < RL; **or;** < 5% of the reg limit; **or,** < 5% of the measured sample concentration (whichever is greater)
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	**245.1**: 85-115% **7470A**: 80-120%
Matrix Duplicate (DUP)[1]	1 in 20 or fewer samples	20% RPD
Matrix Spike (MS)[1]	**245.1**: 1, if 10 samples or less; 2 if 11-20 samples **7470A**: 1 in 20 or fewer samples	**245.1**: 70-130% **7470A**: 75-125%
Matrix Spike Duplicate (MSD)	When requested by client	**245.1**: 70-130% **7470A**: 75-125% **For both 245.1 & 7470A**: If MS and MSD are both ≥ 5X CRDL, then 20% RPD. If MS and MSD are less than the CRDL, the RPD is not calculated; otherwise ±CRDL.
Serial Dilution (SD) for 7470A	1 in 20 or fewer samples	±10%

[1] The sample for DUP and MS are randomly selected, unless specifically requested by a client. Use the same environmental sample for the matrix spike and matrix duplicate sample whenever possible. If insufficient sample amount is available, another environmental sample may be used for the duplicate sample.

9.1.1 **Method Blank:** One laboratory method/preparation blank will be analyzed with each batch of samples prepared together (not to exceed 20 samples). Preparation blank is used to identify possible contamination during acid digestion. If the Mercury concentration in the MB is above the specified control limit, the batch must be prepared again and the samples reanalyzed.

- For 7470A, results must be less than RL, 5% of the regulatory limit for that analyte, or 5% of the measured concentration in the sample, whichever is greater.
- For 245.1, results must be less than RL or less than 10% of the determined Mercury concentration in the sample, whichever is greater.

9.1.2 **Laboratory Control Sample (LCS):** A laboratory control sample must be analyzed with each batch of samples digested. A blank is spiked with 0.1 ug of mercury (0.30 ml of standard DCAL-Int). This is equivalent to 1.0 ppb Hg if a 30 ml portion of sample is digested. Results of the aqueous LCS must fall within ±15% of the true value for Method 245.1 and ±20% for Method 7470A.

9.1.3 **Matrix Duplicate (DUP):** A duplicate is analyzed for each batch of samples digested. If original sample and duplicate are both ≥ CRDL, then 20% RPD. If original sample and duplicate are less than the CRDL, the RPD is not calculated; otherwise, ±CRDL.

9.1.4 **Matrix Spike (MS):** A matrix spike is prepared and analyzed for each batch of samples. A portion of sample is spiked with 0.1 ug of mercury (0.30 ml of standard DCAL-Int). This is equivalent to 1.0 ppb Hg if a 30 ml portion of sample is digested. A recovery of 70-130% for Method 245.1 and 75-125% for Method 7470A is required.

9.1.5 **Matrix Spike Duplicate (MSD):** When requested by the client, a matrix spike duplicate is prepared and analyzed for each batch of samples. A portion of sample is spiked with 0.1 ug of mercury (0.30 ml of standard DCAL-Int). This is equivalent to 1.0 ppb Hg if a 30 ml portion of sample is digested. A recovery of 70-130% for Method 245.1 and 75-125% for Method 7470A is required. For both 245.1 & 7470A: if original sample and duplicate are both ≥ CRDL, then 20% RPD. If original sample and duplicate are less than the CRDL, the RPD is not calculated; otherwise, ±CRDL.

9.1.6 **Serial Dilution (SD):** For method 7470A, a five fold serial dilution must be performed on one sample per batch. The sample should contain a sufficiently high concentration; minimally a factor of 25 times above the estimated detection limit. Dilute the sample by a minimum of five fold (1+4) and reanalyze. Results must agree within 10% of the original determination. If not, a chemical or physical effect should be suspected.

9.2 Instrument QC

9.2.1. **Initial Calibration Verification (ICV):** Initial calibration is verified after calibration. The ICV solution should be prepared using a second source vendor; see section 7.2.7 for preparation instructions. Use a concentration of mercury at the midpoint of the calibration range (5.0ppb). The Cal standard containing 5ppb is analyzed as the ICV; see Sec 7.2.7 for preparation instructions. For 245.1, the results must not differ from the true value by more than 5%. For 7470A, the results must be within 10% of the true value. If it's outside of the acceptable limits, terminate the analysis, correct the problem and recalibrate the instrument.

9.2.2. **Continuing Calibration Verification (CCV):** Calibration verification is performed after the calibration, after every 10 samples, and at the end of the run. The CCV solution should be prepared from the same CAL standard as used to prepare the calibration solutions. Use a concentration of mercury at the midpoint of the calibration range (5.0ppb). The Cal standard containing 5ppb is analyzed as the CCV.

For 245.1 and 7470A, CCV must not differ from the true value by more than 10%. If it does, stop the analysis and recalibrate. Re-analyze the previous ten samples following the last good calibration verification.

9.2.3. **Initial and Continuing Calibration Blank (ICB/CCB):** ICB and CCB must be analyzed after the calibration curve, every 10 samples and at the end of the analytical run. For methods 245.1 and 7470A, the absolute value of the calibration verification blank must not exceed the reporting limit. If it does, terminate the analysis, correct the problem, recalibrate and reanalyze the samples following the last good CCB. The calibration verification blank is the same blank solution as used for the calibration blank.

9.2.4. **Maximum Contaminant Level (MCL):** For drinking water analysis, one MCL standard shall be analyzed per calibration. The 2.0ppb standard prepared from the calibration standards (Sec 7.2.5) is analyzed as the Maximum Contaminant Level (MCL) standard. The result must be within 50% of the true value. If it's outside of the acceptable limit, terminate the analysis, correct the problem, and recalibrate the instrument.

9.2.5. **Quality Control Standard (QCS):** The calibration is verified after calibration using a second source vendor; see Sec 7.2.6 for preparation instructions. For 245.1 and 7470A, the results must not differ from the true value by more than 10%. If it's outside of the acceptable limits, terminate the analysis, correct the problem and recalibrate the instrument.

9.2.6. **Reporting limit check standard (CRI):** At the beginning of the analysis, verify the accuracy at the reporting limit (RL) by analyzing a solution at the RL level. RL check solution is analyzed to demonstrate that the mercury analyzer is capable of detecting the target analyte at the reporting limit (RL). Laboratory limits are 50-150% of the true value.

10.0 Procedure

10.1 Sample Preparation

10.1.1 Filtration Procedure for Dissolved Mercury not filtered in the Field

10.1.1.1 The unpreserved sample must be filtered through a 0.45um filter unit as soon as practical after collection.

10.1.1.2 Collect the required volume of filtrate by using a 0.45um filter unit and a vacuum pump.

10.1.1.3 Acidify the filtrate with 1:1 HNO_3 to a pH of <2.

10.1.1.4 The method blank (MB) must be filtered and digested under the same conditions as the lab filtered samples.

10.1.2 If sample is preserved in laboratory, hold sample for 16 hours for method 245.1 drinking water and 24 hours for wastewater following acidification. Verify and record if sample pH is < 2.0 after 16 or 24 hours (depending on the analysis- drinking water or wastewater) and prior to digestion/analysis. If pH is > 2.0, add HNO3, hold for 24 hours and verify pH.

10.1.3 Transfer 30 ml sample (DI water for MB and LCS) or standard, or an aliquot diluted to 30 ml, to an appropriately identified 50 ml hot block digestion cup. For QA samples, transfer 3 aliquots of 30 ml sample to three digestion cups labeled as SAMPLE, DUP and MS. Spike LCSW and MS with 0.1 µg of mercury (0.3 ml of DCAL-INT standard).

10.1.3.1. Due to insufficient sample volume or an unusual matrix (e.g., samples with potentially high interferences), it may be necessary to perform a pre-digestion dilution. For example, a 10X pre-digestion dilution can be performed by transferring 3 mL of sample to 50 mL hotblock cup. Add 27 ml of deionized water. Record 3 mL initial volume and 30 mL final volume in TALS (see Sec 11.5.2). Continue to section 10.1.3.

10.1.4 Add 1.5 ml concentrated H_2SO_4 and 0.75 ml concentrated HNO_3 mixing well after each addition.

10.1.5 Add 4.5 ml of potassium permanganate solution to each bottle. Mix well and let stand for 15 minutes (minimum); if the color has disappeared, add additional $KMnO_4$ until the purple color persists for at least 15 minutes (document in the Sample preparation log any additional amount of $KMnO_4$ added). The same amount of $KMnO_4$ must be added to the standards and samples.

10.1.6 Add 2.4 ml potassium persulfate solution to each bottle.

10.1.7 Heat for 2 hours in a 95^{o} C hot block digestor. Remove from block digestor and cool.

10.1.8 Add 1.8 ml Sodium chloride - Hydroxylamine hydrochloride solution to reduce the excess permanganate. Mix well; solution should become colorless. If necessary additional Sodium chloride - Hydroxylamine HCl solution may be added.

10.1.9 Due to differing rates of evaporation, the final volume of the standards/samples maybe different. <u>The standards and samples, including all QC, must be at the same final volume before analysis;</u> if not, find the

sample or standard with the highest final volume (e.g., 41 mL) and add deionized water to any sample or standard to bring them to the same final volume . Mix well. Wait at least 30 seconds after decolorization before analyzing.

10.2 Calibration

10.2.1. The instrument must be calibrated daily or once every 24 hours and each time the instrument is set up. The instrument is calibrated according to the manufacturer's specifications and must contain at least four standards and a blank. The laboratory currently uses five standards and a blank. The correlation coefficient of the calibration curve must be ≥0.995. If it does not, the problem must be corrected, and the instrument must be recalibrated. Standard preparations must be documented in TALS (see Sec 11.5.2).

10.2.2. Prepare five calibration standards, blank and calibration verification standard as detailed in sections 7.2.5 and 7.2.6.

10.2.3. Calibration Curve Read-Back:

10.2.1.1 Low-Level Readback (at the RL) – evaluate the 0.2 ug/L calibration standard. The %RE (relative error) must be +/- 20% (see Sec 11.4). If %RE is outside of criteria limits, stop the analysis and recalibrate.

10.2.1.2 Mid- Level Readback – evaluate the 5.0 ug/L calibration standard (the mid-level calibration standard). The %RE (relative error) must be +/- 10% (see Sec 11.4). If %RE is outside of criteria limits, stop the analysis and recalibrate.

10.3 Sample Analysis

10.3.1 Following a sample digestion procedure, the samples are ready for instrumental analysis. It is advisable to investigate each matrix for any complexities, which might adversely affect the acquisition of valid data.

10.3.2 The following analytical run sequence is currently used:

Instrument Calibration (Blank and five standards)

ICV
ICB
CRI
QCS
MCL (for drinking water analysis)
CCV

CCB
10 Samples
CCV
CCB
Repeat until run is completed
CCV
CCB

10.3.3 Instrument Operation:

10.3.3.1. Turn on Computer, Monitor and the Hg analyzer (Hydra IIAA).

10.3.3.2. Plumbing the Reagent Lines:

10.3.3.2.1. One at a time, feed each of the pump tubes into a pump cassette, sliding the tube through the plastic clips at the bottom until the plastic tab is secure. Then, holding the tube taut, slide the loaded cassette onto the pump head and click the clamp, lever up. The tab end of the tube should be located at the front of the pump head.

10.3.3.2.2. Reductant (Red): Connect tab end of tube to the reductant tubing that is connected to the reductant bottle and the other end to the mixing tee.

10.3.3.2.3. Sample (Blue): Connect tab end of tube to the autosampler probe and the other end to the mixing tee.

10.3.3.2.4. Drain (Blue): Connect the tab end of tube to the sample discharge tube connected on the Liquid/Gas separator and the other end to the waste line.

10.3.3.2.5. Rinse (Black): Connect tab end of tube to rinse tubing that is connected to the rinse bottle. Connect the other end to the rinse tubing leading to the rinse cup.

10.3.3.3. Preparation of Reagents:

10.3.3.3.1. Pour the 10% $SnCl_2$ solution into the reductant bottle.

10.3.3.3.2. Pour the 10% HCl solution into the rinse reservoir bottle.

10.3.3.4. Start the Program:

10.3.3.4.1. Click the Envoy icon on the computer desktop

10.3.3.4.2. Click Method, select 245.1_7470A, and select OK
10.3.3.4.3. On the main screen, click the StartUp icon. Wait 15 minutes before analyzing calibrating.
10.3.3.4.4. Click Sequence Tab on bottom of screen
10.3.3.4.5. Click Sequence on top of screen
10.3.3.4.6. Select Open, then select New
10.3.3.4.7. Using the Prep Batch Sheet and hand scanner, enter the sample barcodes into the Sample ID column. Include the Serial Dilution and all needed sample dilutions
10.3.3.4.8. Pour out the digested calibration standards and samples into the proper locations on the autosampler.
10.3.3.4.9. Click the Run Sequence icon to begin calibration and to run samples.

10.3.3.4.10. To shut down the instrument, click the Stop icon on the main screen.

11.0 Calculations / Data Reduction

11.1 Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2 Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Final results calculation in aqueous samples :

$$\text{Concentration = mg/ L} = \frac{C \times V1 \times D}{V2}$$

Where:
C= Element concentration from instrument (ppb)
V1= Final volume of sample digested (in liters)
D= Dilution performed on sample

V2= Initial volume of sample digested
(in liters).

11.4. Relative Error (%RE):

% Relative Error = (100) (MC-TC)/TC

MC = Measured Concentration of the calibration standard
TC = True Concentration for the calibration standard

11.5. Data Processing:

11.5.1. All data is recorded directly in TALS' Analyst Desktop II program.

11.5.2. Record standard/sample preparations in the Analyst Desktop II program located in the TestAmerica Laboratory System (TALS) Reagent module. The analyst must enter the following information: Source standard, Initial and final sample volume, spike name and amount used, all reagents and their corresponding lot numbers, creation and expiration dates.

11.5.3. Record the following reagents and the volume used for sample preparation in the batch information page under "batch comments" in TALS Analyst Desktop II: concentrated sulfuric acid, concentrated nitric acid, potassium permanganate, potassium persulfate and sodium chloride-hydroxylamine hydrochloride. Record reagents in TALS by opening the prep batch, click on "edit" and then right click to choose "view batch information." Enter the information in the "batch comments" section.

11.5.4. Import Data to TALS

Click the Analysis tab

11.5.4.1. Select the analytical run that needs to be imported
11.5.4.2. Select Statistics
11.5.4.3. Click Load and select TALS Import, click OK
11.5.4.4. Click Report, click CSV File
11.5.4.5. Name the import file (e.g., batch name, today's date)
11.5.4.6. The newly created import file is in the Import Folder on the desktop Send to TALS Import Folder

11.5.5. Creating the Raw Data PDF:

11.5.5.1. Click the Analysis Tab, select Detailed

11.5.5.2. Click Load, select Use This To Make PDF

11.5.5.3. Click Report, Click Printer

11.5.5.4. Include the calibration curve graph:

11.5.5.4.1. Go to Methods Tab and click Calibration, click Print

11.5.5.5. Combine both documents using the PDF Creator program. The new document is located in the Documents folder on the C:\ drive. Add this document to the "Doc's" location in the analytical batch.

11.5.6. Complete the Data Review Checker (DRC) in TALS: Prior to data submission (i.e., prior to 1st leveling data in the Analyst Desktop II module in TALS), the analyst must execute the DRC (Data Review Checker) program in the applicable analytical batch.

11.5.6.1. Open the analytical batch and click on the Edit tab above to enter the Edit Mode.

11.5.6.2. Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on 'Run checklist.'

11.5.6.3. Acknowledge by filling in responses to all unacknowledged findings.

11.5.6.3.1. Highlight the checklist with findings, then highlight the associated list of findings; right click and choose 'Acknowledge Item.'

11.5.6.3.2. Fill in appropriate comments in the response box, then hit 'OK.'

11.5.6.3.3. Acknowledge all Finding items in the 'Manual Batch Checklist' except for the *"2nd Level review complete?"* this is to be completed by the 2nd level reviewer

12. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Instrument Detection Limit:

The IDL for each analyte must be determined for each wavelength used on each instrument. The IDL must be determined annually or if the instrument is adjusted in any way that may affect the IDL. For 245.1, the IDL is determined by multiplying the average of the standard deviations obtained from the analysis of 10 reagent blanks by 3. For 7470A, the IDL is determined by multiplying the average of the standard deviations obtained from the analysis of 7 reagent blanks by 3.14

12.3 Linear Dynamic Range (LDR)

The upper limit of the LDR must be established. It must be determined from a linear calibration prepared from a minimum of three different concentration standards, one of which is close to the upper limit of the linear range. The LDR should be determined by analyzing succeedingly higher standard concentrations of mercury until the observed analyte concentration is no more than 10% below the stated concentration of the standard. The determined LDR must be documented and kept on file. The LDR which may be used for the analysis of samples should be judged by the analyst from the resulting data. Determined sample analyte concentrations that are greater than 90% of the determined upper LDR limit must be diluted and reanalyzed. The LDR should be verified annually or whenever, in the judgement of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be redetermined.

12.4 Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.5 Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13. 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. Waste Management

14.1. 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 TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out.

- Digested Samples: Corrosive Acid- Materials that are not above regulatory limits will be submitted for elementary neutralization with 50% sodium hydroxide solution (Siedler Chemical SC-1824-03). Major concern is heat generated from the neutralization process. Initial volume of acid waste to be neutralized should be no more than 15 gallons. Finished neutralization with sodium bicarbonate (Siedler Chemical SC-0219-25) to a pH of 6 – 9 in the primary tank. Once pH has been established the primary tank is transferred through filter housing to a secondary tank. The pH is rechecked. If the pH is within specifications, the secondary tank is released to the municipal sewer system.

- Samples above regulatory limits and expired RCRA metals standards (Waste Corrosive Liquid, Acidic, Inorganic, n.o.s.) are collected in satellite accumulation and sent off site through a Waste disposal vendor.
  Onyx Profile WIP Number: 590598
  Teris Profile Number 50016653

15. References / Cross-References

15.1. Determination of Mercury in Water by Cold Atomic Absorption Spectrometry, EMSL-Cincinnati, EPA/600/R-94/11, May 1994; Method 245.1 Revision 3.0.

15.2. Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996.

15.3. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.5. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.6. Corporate Environmental Health and Safety Manual CW-E-M-001, most current revision.

15.7. Leeman Hydra II AA Operating Manual

16. Method Modifications:

Item	Method No.	Modification

Sample Preparation	SW 7470A	Stannous Chloride is automatically added via the instrument versus the manual addition of Stannous Chloride as stated in the method. This is an instrument manufacturer's improvement that will reduce error due to loss of Mercury.
Sample Preparation	SW 7470A EPA 245.1	The hotblock has replaced the hot-water bath for digestion. This modification has been made to reduce cross-contamination (the hotblock tubes are disposable).
Sample Preparation	SW 7470A EPA 245.1	The typical prep sample size is reduced to 30ml (previously 100ml). This modification was made to allow for limited available sample volumes. Reagent volumes were adjusted to maintain sample to reagent volume ratio
Sample Preparation	EPA 245.1	The Initial Calibration Verification (ICV) will be prepared from a secondary source (as required in Method SW7470A), rather than the calibration standard source. Method 7470A and 245.1 will be prepared and analyzed in the same batch which improves efficiency and reduces waste. CCV will be prepared using the same source as the calibration standards.

17. Attachments

Attachment 1: Example Certificate of analysis (10 ppm Hg)

18. Revision History

- Revision 12, dated 03 August 2020
  - Updated header with Eurofins logo.
  - Sec 6.1.1: Replaced Hydra AA with Hydra IIAA
  - Sec 6.1.2: Updated Hg software from WinHg to Envoy.
  - Sec 8.0: Revised the required sample volume from 500ml to 250ml.
  - Sec 9.1: Revised MB control limits for 245.1 and 7470A to <RL; added MSD to the QC table.
  - Sec 9.1.1: Updated the control criteria for 245.1 and 7470A from <MDL to <RL.
  - Sec 9.1.3: Updated the criteria for the matrix duplicate to reflect current laboratory practices.
  - Sec 9.1.5: Added MSD to the list of Sample QC.
  - Sec 9.2.2: Revised the CCV recovery limit for method 7470A to 10%.
  - Sec 10.2.3: Added calibration curve read-back criteria.
  - Sec 10.3.3.4: Updated instructions for the instrument/software operation.
  - Sec 11.4: Added Relative % error calculation.
  - Sec 11.5.2: Added reagent/standard information to document in TALS ADII.
  - Sec 11.5.3.: Added requirements to record reagent volumes in the batch information page of TALS ADII; subsequent sections adjusted accordingly.
  - Sec 11.5.4 & 11.5.5: Updated instructions for data import and raw data pdf generation.
  - Sec 11.5.6: Added Data Review Checker (DRC) instructions.
  - Sec 15.7 & 15.8: Deleted work instructions EDS-WI-007 (TestAmerica Edison Metals Data Review Checklist) and WI EDS-WI-125 (TestAmerica Edison Metals

Initial-Calibration Data Review Mercury checklist), not applicable.

- Revision 11, dated 25 April 2018
  - Sec 8: Added footnote 3 to clarify instructions for metals sample preservation (verify pH <2.0 prior to digestion).
  - Sec 10.1.2: Added procedure for samples which pH are >2.0 and acid preservation is performed in laboratory.
  - Sec 12.3: Added procedure for the determination of linear dynamic range.

- Revision 10, dated 05 February 2018
  - Sec 7.2.10: Added CRI to the list of standards.
  - Sec 9.2.6: Added CRI to the list of instrument QC.
  - Sec 10.3.2: Revised the instrument sequence list to include CRI.

- Revision 9, dated 09 December 2015
  - Sec 7.2.7: Revised preparation procedure for ICV, standard solution should be obtained from a source different from the calibration standard. Removed reference to CCV.
  - Sec 7.2.8: Added CCV preparation procedure; standard solution same source as the calibration standard. Subsequent sections adjusted accordingly.
  - Sect 9.2.1: Revised - ICV solution should be prepared from a separate source.
  - Section 16: Added Method Modifications for using the secondary source when preparing the ICV standard for method EPA 245.1.

- Revision 8, dated 13 January 2014
  - Sec 7.1.8: Added the stock standard Potassium Persulfate to the list of reagents; subsequent sections adjusted accordingly.
  - Sect 7.1.15: Added preparation instructions for the 5% Potassium Persulfate solution.
  - Sec 7.1: Revised to include recording of reagent prep information in TALS and discontinue the use of Mercury Prep Logbook.
  - Sec 7.2.5 & 7.2.6: Revised the spiking amount to add when preparing calibration standards in 30 ml final volume; also replaced the 100 ml flasks with 50 ml digestion cups.
  - Sec 9.0: The matrix spike (MS) frequency for Method 245.1 has been revised to 1 per 10 samples to comply with the method.
  - Sec 11.4.4: Added the Mercury calibration checklist (EDS-WI-125).
  - Sec 15.8: Added WI# ED-WI-125 on the list of references; subsequent sections adjusted accordingly.
  - Sec 17: Updated the attached COA for the Hg standards.

- Revision 7, dated 02 December 2011
  - Sec 1.1: Revised detection limit to 0.2 ug/L Hg to reflect actual laboratory limits.
  - Sec 1 & 12: Revised the LQM reference for DOC and Test Methods and Method Validation to Section 19.
  - Sec 3: Revised LQM reference for the definitions.
  - Sec 5: Revised in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002, Writing a Standard Operating Procedure (SOP), most current revision.

- Sec 7.1: Added requirements that all prepared reagents are recorded in TestAmerica Edison Mercury Std Prep Log.
- Sec 7.2 & 10.2.1: Revised the documentation of standard preparation from logbook to TALS.
- Sec 7.2.5: Replaced cal std 0.1 ug/L (Hg) with 0.2 ug/L.
- Sec 9.1 and 9.1.1: Expanded MB control limit for 245.1 to reflect method's criteria.
- Replaced PB (preparation blank) with MB (method blank) in various sections where applicable.
- Sec 10.1.1: Added procedure for filtering dissolved samples in the lab.
- Sec 10.1.2.1: Added procedure for pre-digestion dilutions.
- Sec 10.1.8: Added procedure for visually checking and adjusting final volumes for all standards and samples.
- Sec 10.3.3.11.7: Added procedure for post-digestion dilutions.

- Revision 6, dated 03 September 2009

  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Combined SOP ED-MT-014 and ED-MT-015 with SOP ED-MT-017; retired SOP ED-MT-014 and ED-MT-015 at the effective date of this SOP.
  - Sec 6.1.3: Replaced water bath with block digestor.
  - Sec 6.2.1: Replaced 300 ml BOD bottles with 50 ml hot block digestion cups
  - Sec 6.2.3. Added 'Rinse Black' and changed Drain 'Black' to Drain 'Blue' in the list of Reagent lines
  - Sec 7.1: Added 0.15% HNO3 to list of reagents. Deleted Potassium Persulfate, Magnesium Perchlorate, 0.5N H2SO4 & 5% Potassium Persulfate solution to list of reagents; Reagents deleted are not applicable to this method.
  - Sec 7.2 Standards: Revised the Hg stock standard concentrations and preparation of standards.
  - Sec 7.1.9 & 7.1.13: Revised preparation procedure to reflect actual lab practices.
  - Sec 7.2.6: Renamed the second source standard 'Initial Calibration Verification standard' to 'Quality control standard.' QCS is added in the Instrument QC and analytical run sequence.
  - Section 8: Updated the section into Table format and have included the sample container, sample size requirements and method reference.
  - Changed the verification standard concentration of ICV (3ppb) to 5ppb; CCV concentration remains 5ppb.
  - Revised control limits to comply with Method 245.1
  - Sec 9.1.1: Clarified QC limits for the Preparation Blank
  - Sec 9.1.2: Revised the LCS limits for wastewater samples analyzed via Method 245.1 from ±20% to ±15%.
  - Sec 9.1.4: Revised the MS limits for wastewater samples analyzed via Method 245.1 from ±20% to ±30%.
  - Sec 9.1.5: Added Serial Dilution (L) in Sample QC
  - Sec 9.2.1: Clarified the recovery limits of ICV for method 245.1; % Rec limits were revised from 10% to 5% to reflect actual laboratory practices.
  - Sec 10.3.2: Added MCL in the analytical run sequence.
  - Sec 10.3.3.2: Added 'Rinse Black' and changed Drain 'Black' to Drain 'Blue' in the

list of Reagent lines.

- Sample size reduced from 100 ml to 30ml; preparation of the LCS (Sec 9.1.2) and MS (Sec 9.1.4) were revised to reflect this change in sample volume.
- Sec 10.1: Adjusted reagent volume to maintain sample to reagent ratios.
- Sec 11: Updated data processing in accordance with the new TALS.
- Sec 15: Added applicable references.
- Sec 16: Described the elimination of stannous chloride in the sample preparation and replacement of hot water bath as a method modification.

Attachment 1

SCP SCIENCE
Providing Innovative Solutions to Analytical Chemists

ACCREDITED

Certificate of Analysis

1.0 DESCRIPTION : ***Plasma*** **CAL – Custom Standard**

Catalogue Number : **141-110-11X**
Lot Number : **S130418021**
Matrix: 10.0% HNO3
Expiration Date : **July 2014**

2691783
ID: ME_Hg_1st_00013
Exp: 07/01/14 Post DLE
Mercury Calibration Stand

2.0 CERTIFIED VALUES AND ASSOCIATED UNCERTAINTY:

Method of Analysis: Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES)

Traceability: Applicable NIST Standard Reference Materials (see list below):

3101a	Al	3109a	Ca	3117a	Eu	3126a	Fe	3134	Mo	3142a	Pr	3151	Ag	3159	Th	3167a	Y
3102a	Sb	3110	Ce	3118a	Gd	3127a	La	3135a	Nd	3143	Re	3152a	Na	3160a	Tm	3168a	Zn
3103a	As	3111a	Cs	3119a	Ga	3128	Pb	3136	Ni	3144	Rh	3153a	Sr	3161a	Sn	3169	Zr
3104a	Ba	3112a	Cr	3120a	Ge	3129a	Li	3137	Nb	3145a	Rb	3154	S	3162a	Ti		
3105a	Be	3113	Co	3121	Au	3130a	Lu	3138	Pd	3147a	Sm	3155	Ta	3163	W		
3106	Bi	3114	Cu	3122	Hf	3131a	Mg	3139a	P	3148a	Sc	3156	Te	3164	U		
3107	B	3115a	Dy	3123a	Ho	3132	Mn	3140	Pt	3149	Se	3157a	Tb	3165	V		
3108	Cd	3116a	Er	3124a	In	3133	Hg	3141a	K	3150	Si	3158	Tl	3166a	Yb		

Certified Concentrations:

Hg 9.96 ± 0.06 µg/ml

Note: The uncertainty of the certified value has been calculated from applicable uncertainty contributors (u_i) such as the SRM inherited uncertainty, weighing and dilution errors and instrument variability. The combined uncertainty ($uc = \sqrt{\Sigma u_i^2}$) has been multiplied by a coverage factor (k) of 2 to provide a 95% confidence interval.

3.0 REFERENCE VALUES:

Density: **1.051 g/ml** @ **20.9 °C**

4.0 APPROVAL AND DATE OF CERTIFICATION:

Certification Approval: Yaling Sui, Chemist Yaling Sui
Certification Date: **April 19, 2013**

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CERTIFICATE OF ANALYSIS

1.0 **INORGANIC VENTURES** is an ISO Guide 34 "General Requirements for the Competence of Reference Material Producers" and ISO 9001 registered manufacturer. Our manufacturing laboratory is accredited to ISO/IEC 17025 "General Requirements for the Competence of Testing and Calibration Laboratories."

ACCREDITED Testing Laboratory Certificate 863.01

ACCREDITED Reference Material Producer Certificate 863.02

2.0 **DESCRIPTION OF CRM** **10 µg/mL Mercury in 10% (v/v) HCL**

2035815
ID: ME_Hg_00019
Exp: 04/01/14 Prpd: CDC
Mercury Calibration Stand

Catalog Number:	MSHG-10PPM
Lot Number:	F2-HG02097
Starting Material:	Hg metal
Starting Material Purity (%):	100.0000
Starting Material Lot No:	R307HGA1
Matrix:	10% (v/v) HCL

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

Certified Concentration: 9.990 ± 0.074 µg/mL

Certified Density: 1.026 g/mL (measured at 20 ± 1°C)

The following equations are used in the calculation of the certified value and the uncertainty. Reported uncertainties represent expanded uncertainties expressed at approximately the 95% confidence level using a coverage factor of k = 2.

Certified Value $(\bar{x}) = \frac{\Sigma x_i}{n}$ ($\bar{x}$) = mean

x_i = individual results

n = number of measurements

Uncertainty (±) = $2 [\Sigma(s_i)^2]^{½}$ 2 = the coverage factor.

$[\Sigma(s_i)^2]^{½}$ = The square root of the sum of the squares of the most common errors (where 's' stands for the standard deviation) from instrumental measurement, density, NIST SRM uncertainty, weighing, dilution to volume, homogeneity, long term stability and short term stability.

4.0 **TRACEABILITY TO NIST AND VALUES OBTAINED BY INDEPENDENT METHODS**

- "Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties." (ISO VIM, 2nd ed.,

Title: Trace Metals Analysis for Water, Wastewater, Soil and Sediment Samples by ICP-MS EPA Method 200.8

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

This SOP describes the procedures used to determine the concentration of various elements in surface, drinking, and ground water, wastewater, by Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) using EPA Method 200.8.

The routine target analytes and reporting limits are as follows:

Table 1

Analyte List and Method Reporting Limits (RL)

Element	Drinking Water RL	Non-Drinking Water RL
	(ug/L)	(200.8 NPDES (ug/L)
Aluminum (Al)	25	40
Antimony (Sb)	2.5	2
Arsenic (As)	1	2
Boron (B)	20	80
Barium (Ba)	2.5	4
Beryllium (Be)	2.5	0.8
Cadmium (Cd)	2.5	2
Calcium (Ca)	250	200
Cobalt (Co)	2.5	4
Chromium (Cr)	2.5	4
Copper (Cu)	2.5	4
Iron (Fe)	250	120
Lead (Pb)	2.0	1.2
Magnesium (Mg)	250	200
Manganese (Mn)	25	8
Molybdenum (Mo)	2.5	4
Nickel (Ni)	2.5	4
Potassium (K)	250	200
Selenium (Se)	2.5	2.5
Silver (Ag)	2.5	2
Sodium (Na)	250	200
Strontium (Sr)	2.5	4
Thallium (Tl)	0.5	0.8
Tin (Sn)	4	16
Titanium (Ti)	2.5	4
Vanadium (V)	2.5	4
Zinc (Zn)	4	16

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

Prior to analysis by ICP-MS, the sample must be solubilized or digested using the sample preparation method appropriate to the matrix. Sample digestates are aspirated and nebulized into a spray chamber. A stream of argon gas carries the sample aerosol through the innermost of three concentric tubes and injects it into the middle of the donut-shaped plasma. The sample elements are dissociated, atomized, and excited to a higher energy level. The ions that are produced are entrained in the plasma and introduced, by means of an interface, into a mass spectrometer. The ions are sorted according to their mass to charge ratios and quantified with a channel mass spectrometer.

Aqueous samples are digested using Method 200.8 Drinking water samples with turbidity $\geq$ 1 NTU must undergo digestion prior to analysis. Refer to TestAmerica Edison SOP Nos. ED-MTP-001.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Isobaric elemental interferences are caused by isotopes of different elements which form singly or doubly charged ions of the same nominal mass-to-charge ratio and which cannot be resolved by the mass spectrometer in use. Isobaric polyatomic/molecular ion interferences are caused by ions consisting of more than one atom which have the same nominal mass-to-charge ratio as the isotope of interest and which cannot be resolved by the mass spectrometer in use. Most isobaric interferences that could affect the ICP-MS analysis for elements in this SOP have been identified. These can be managed by the selection of an alternate isotope, the use of the collision/reaction cell (used for non-potable samples only), or by the use of elemental interference equations when the collision/reaction cell is not in use.

4.2. For Drinking Water analysis (standards, samples, and relating QC) the collision/reaction cell can not be used; therefore, the following interference equations should be used to reduce interferences:

Mass	Equation
6:	(6)*1 - (7)*0.082
44:	(44)*1 - (88)*0.015
51:	(51)*1 – (53)*3 + (52)*0.34
66:	(66)*1 – (69)*0.00141
75:	(75)*1 – (77)*2.9 + (82)*2.23 – (83)*2.23
82:	(82)*1 – (83)*1
111:	(111)*1 – (108)*1.073 + (106)*0.712

115 (115)*1 – (118)*0.016
208: (208)*1 + (206)*1 + (207)*1 (used for both drinking and non-drinking water analysis)

The basic elemental interference equations are based on natural isotopic abundances. The most precise coefficients for an instrument must be determined from the ratio of the net isotope signals that are observed for a known standard solution at a concentration sufficient to produce suitable counting statistics.

4.3. Krypton affects the determination of both arsenic and selenium but can be greatly reduced with the use of high purity Krypton free argon. Krypton must be analyzed for every sample and standard.

4.4. Physical interferences are effects associated with the sample nebulization and transport processes as well as ion-transmission efficiencies. Changes in viscosity can cause significant inaccuracies, especially in samples containing high concentrations of dissolved solids or high acid concentrations. These changes in matrix can cause significant signal suppression or enhancement. Dissolved solids can deposit on nebulizer tips and interface cones (reducing the orifice size and the instrument's performance). Internal standards can be used to correct for physical interferences if they are carefully matched to the analyte so that both elements react similarly to the matrix changes.

4.5. Memory interferences can occur when analytes from a previous sample contribute to signals measured from subsequent samples. The memory effects can result from analyte deposition of sample on the sample tubing, joints, nebulizer, spray chamber, torch, and/or interface cones. Routine maintenance on the sample introduction system is necessary in order to minimize the memory interferences. The memory effects must be taken into account when setting up a suitable rinse times. The evaluation of a minimum of three replicate integrations will help to determine memory problems.

4.6. Contamination of the sample can occur when the preparation glassware and/or reagents contain the target elements. Reagent blanks (method blanks) must be analyzed as a check on contamination due to the sample digestion.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.

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 MSDS 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 MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Hydrochloric Acid	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.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

- Agilent 7800 and Agilent 7900: inductively coupled plasma mass spectrometer with data system (microprocessor, monitor, printer) and autosampler. Mass Range 2-260 amu. Vacuum purged spectrophotometer with an axial plasma torch. Collision/Reaction Cell.
- Heat exchanger –PolyScience Model 6106T Recirculating Chiller or equivalent
- Autosampler – Agilent SPS 4 Autosampler

6.2. Supplies

- Reagent Water -18 megohm Reagent grade Type II water
- Volumetric Flasks (Class A): 50 mLs, 100 mLs, 500mls & 1000mls
- Eppendorf & Fisher Pipettes, varying volumes
- Polypropylene tubes

7.0. Reagents and Standards

7.1. Reagents

7.1.1. Concentrated Nitric Acid (HNO3) - Trace Grade or Equivalent; store at room temperature; for stability information, refer to manufacturer's instructions. The assay sheet of each lot of acid received into the lab must be reviewed to ensure the quality of the acid is sufficient for trace analysis of metals.

7.1.2. Concentrated Hydrochloric Acid (HCL) - Trace Grade or Equivalent; store at room temperature; for stability information, refer to manufacturer's instructions. The assay sheet of each lot of acid received into the lab must be reviewed to ensure the quality of the acid is sufficient for trace analysis of metals.

7.1.3. Argon supply - 99.9% (Liquid)

7.1.4. Helium and Hydrogen supply – 99.999% (Gas)

7.1.5. 18 megohm Reagent grade Type II water.

7.1.6. Reagent water, 2% HNO3 + 0.5% HCl by volume: Add 400ml of concentrated HNO3 and 100ml of concentrated HCl to deionized water and bring to 20 liter volume with deionized water. For potable samples not requiring digestion, the reagent water is prepared without HCL. Note: Always add acid to water. Record preparation in the TALS Reagent Module Prepare every 12 months or refer to manufacturer's expiration date; store at room temperature.

7.1.7. 5% HNO3 + 5% HCl: Add 1 liter of concentrated HNO3 and 1 liter of concentrated HCl to deionized water and bring to 20 liter volume with deionized water. For potable samples not requiring digestion, the reagent water is prepared without HCl. Varying amounts of this reagent may be made (e.g., 10L) by proportionally adjusting the volume of acids used. Note: Always add acid to water. Record preparation in the TALS Reagent Module Prepare every 12 months or refer to manufacturer's expiration date; store at room temperature.

7.2. Standards

Calibration and spike solutions are prepared from either certified stock solutions or from stock solutions purchased from vendors. Certificates of analysis or purity must be received with all neat compounds or stock solutions. Certificate of analysis are filed in Metals managers office.

Storage requirements: all standards are stored at room temperature
Shelf-life: Stock standards – refer to manufacturer's instructions
Intermediate standards – 6 months
Working cal standards – two weeks
Working Initial calibration verification standard – quarterly
Interference Check Standards (A&B) - weekly
(Note: expiration date must not go beyond the expiration date of the source stock).
Concentration: Final concentrations for the calibration standards are given in Attachment 1. All standards must be prepared in reagent water (Sec. 7.1.6 & 7.1.7).

Standards must be prepared in accordance to the shelf life (Sec 7.2) "or sooner if needed or required." "If needed" means the standard has been exhausted; "if required" means that the standard does not meet the QC criteria.

All standards must be prepared in reagent water (Sec. 7.1.6 and 7.1.7). Final concentrations for the calibration standards are given in Attachment 1.

7.2.1. Calibration Standards: ICPMS-Cal (Custom Calibration Standard Stock Solution) purchased from Inorganic Ventures and from CPI International. Refer to manufacturer's instructions for stability and storage information.

TANJ-STD-1 contains: 20ug/mL B
10 ug/mL Mo, Sb, Sn and Ti

TANJ-STD-2 contains: 1000 ug/mL Ca, Fe, K, Mg, & Na
100 ug/mL Al & Mn
10 ug/mL Ag, As, Ba, Be, Cd, Co, Cr3, Cu, Ni, Pb, Se, Sr, V & Zn
2.0 ug/mL Tl

TA-88 contains:
10,000 ug/L Ca, K, Mg, Na
6,000 ug/L Fe
4,000 ug/L B
2,000 ug/L Al
800 ug/L Sn, Zn
500 ug/L Se
400ug/L Mn
200 ug/L Ba, Co, Cr, Cu, Mo, Ni, Sr, Ti, V
100 ug/L Ag, As, Cd, Sb
60 ug/L Pb
40 ug/L Be, Tl

STLNJ-STD-1 contains: 5000 ug/L Ca, K, Mg, Na
3,000 ug/L Fe

2,000 ug/L B
1,000 ug/L Al
400 ug/L Zn
200ug/L Mn
100 ug/L Ag, Ba, Co, Cr, Cu, Ni, Sr, V
50 ug/L As, Cd, Se
30 ug/L Pb
20 ug/L Be, Tl

STLNJ-STD-2 contains: 400 ug/L Sn
100 ug/L Mo, Ti
50 ug/L Sb

6020B RL Standard contains: 20 mg/L Ca, K, Mg, Na
12 mg/L Fe
8 mg/L B
4 mg/L Al
1.6 mg/L Sn, Zn
0.8 mg/L Mn
0.4 mg/L Ba, Co, Cr, Cu, Mo, Ni, Sr, Ti, V
0.25 mg/L Se
0.2 mg/L Ag, As, Cd, Sb,
0.12 mg/L Pb
0.08 mg/L Be, Tl

TA-CAL-1 contains: 1,000 mg/L Si
100 mg/L As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Mo, Ni, Pb, Sb, Se, Si, Sn, Sr, Ti, Tl, V

TA-CAL-2 contains: 1,000 mg/L Al, Ca, Fe, K, Mg, Na

Single Element Standard: Ag 1,000 mg/L

Single Element Standard: B 1,000 mg/L

Single Element Standard: Zn 1,000 mg/L

TA-72REV2 contains: 5000 ug/L Ca, Fe, K, Mg, Na
2500 ug/L Al, Mn,
2000 ug/L B
400 ug/L Sn, Zn,
250 ug/L Ag, Ba, Be, Cd, Co, Cr, Cu, Mo, Ni, Sb, Se, Sr, Ti, V
Pb 200 ug/L
As 100 ug/L
Tl 50 ug/L

7.2.2. Working Calibration Standards:

For Non-Drinking Waters:

CAL5: Add 100ml of 5% HNO3 / 5% HCl (Sec 7.1.7) to a clean 200ml flask. Add 0.4ml of TA-CAL-1 and 2ml of TA-CAL-2. Add 10 ml of MS-Cal-Int. Bring to volume with 5% HNO3 / 5% HCl.

CAL4/CCV: Add 100ml of 5% HNO3 / 5% HCl (Sec 7.1.7) to a clean 200ml flask. Add 0.2ml of TA-CAL-1 and 1ml of TA-CAL-2. Add 5 ml of MS-Cal-Int. Bring to volume with 5% HNO3 / 5% HCl.

CAL3: Add 100ml of 5% HNO3 / 5% HCl (Sec 7.1.7) to a clean 200ml flask. Add 0.1ml of TA-CAL-1 and 0.5ml of TA-CAL-2. Add 2.5 ml of MS-Cal-Int. Bring to volume with 5% HNO3 / 5% HCl.

CAL2: Add 100ml of 5% HNO3 / 5% HCl to a clean flask. Add 10ml of 6020B RL Standard. Bring to volume with 5% HNO3 / 5% HCl (Sec 7.1.7).

CAL1: Add 100ml of 5% HNO3 / 5% HCl to a clean flask. Add 2ml of 6020B RL Standard. Bring to volume with 5% HNO3 / 5% HCl (Sec 7.1.7).

MS-Cal-Int: Add 100ml of 5% HNO3 / 5% HCl (Sec 7.1.6) to a clean 200ml flask. Add 2ml each of B and Zn. Add 0.4ml of Ag. Bring to volume with 5% HNO3 / 5% HCl (Sec 7.1.7).

For Drinking Waters:

CAL4: Add 100ml of reagent water (Sec 7.1.6) to a clean 200ml flask. Add 2ml each of TANJ-STD-1 and TANJ-STD-2. Bring to volume with reagent water (Sec 7.1.6).

CAL3/CCV: Add 100ml of reagent water to a clean 200ml flask. Add 1 mL each of TANJ-STD-1 and TANJ-STD-2. Bring to volume with reagent water (Sec 7.1.6).

CAL2: Add 100ml of reagent water to a clean 200mL flask. Add 0.2ml each of TANJ-STD-1 and TANJ-STD-2. Bring to volume with reagent water (Sec 7.1.6).

CAL1: Add 100ml of reagent water to a clean 200mL flask. Add 2 mL of TA-72REV2. Bring to volume with reagent water (Sec 7.1.6). This standard is used as the Reporting Limit standard.

Note: see Attachment 1 for final elemental concentrations.

7.2.3. Initial Calibration Verification Stock standards: purchased from CPI International. Refer to manufacturer's instructions for stability and storage information. Standards must be from a different source than those used for

the calibration standards.

Standard	Element	Conc. µg/mL
ICV1ICPMS	Be	5
	Na	500
	Mg	500
	Al	50
	K	500
	Ca	500
	V	5
	Cr	5
	Mn	50
	Fe	500
	Co	5
	Ni	5
	Cu	5
	Zn	5
	As	5
	Se	5
	Sr	5
	Ag	5
	Cd	5
	Ba	5
	TL	1
	Pb	5
ICV2ICPMS	B	10
	Ti	5
	Mo	5
	Sn	5
	Sb	5

TA-CAL1-SS contains: 1,000 mg/L Si
100 mg/L As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Mo, Ni, Pb, Sb, Se, Si, Sn, Sr, Ti, Tl, V

TA-CAL2-SS contains: 1,000 mg/L Al, Ca, Fe, K, Mg, Na

2nd Source Single Element Standard: Ag 1,000 mg/L

2nd Source Single Element Standard: B 1,000 mg/L

2nd Source Single Element Standard: Zn 1,000 mg/L

7.2.4. ICV Working Standard:

7.2.4.1. ICV Potable: The ICV Working Standard must be at a concentration different from the concentrations used for the CCV working standard. Prepare by adding 300ml of reagent water (Sec 7.1.6) to a clean 500ml flask. Add 4ml each: ICV1ICPMS and ICV2ICPMS. Bring to volume with reagent water (Sec 7.1.6). The final concentrations of the various elements are listed in Attachment 1. This standard is prepared fresh quarterly or as needed and stored at room temperature.

7.2.4.2. ICV Non-Potable: The ICV Working Standard must be at a concentration different from the concentrations used for the CCV working standard. Prepare by adding 100ml of 5% HNO3 / 5% HCl (Sec 7.1.7) to a clean 200ml flask. Add 0.16ml of TA-CAL-1-SS and 0.8ml of TA-CAL-2-SS. Add 4 ml of MS-ICV-Int (see Sec 7.2.4.1). Bring to volume with 5% HNO3 / 5% HCl. The final concentrations of the various elements are listed in Attachment 1. This standard is prepared fresh quarterly or as needed and store at room temperature.

7.2.4.2.1. MS-ICV-Int: Add 100ml of 5% HNO3 / 5% HCl (Sec 7.1.6) to a clean 200ml flask. Add 2ml each of 2nd Source: B & Zn. Add 0.4ml of 2nd Source: Ag. Bring to volume with 5% HNO3 / 5% HCl (Sec 7.1.7).

7.2.5. Continuing Calibration Verification standard, Non-potable: see CAL4/CCV (Sec. 7.2.2).

7.2.6. Continuing Calibration Verification standard, Potable: see CAL3/CCV (Sec. 7.2.2).

7.2.7. Internal Standards and EPA Tune Check:

The following elements are used as the internal standards: Bi, In, Li6, Sc, Tb, and Ge.

7.2.7.1. Internal Standards Stock Solutions**:**

7.2.7.1.1. Testamerica-1 (Custom Calibration Standard Stock Solution) purchased from Inorganic Ventures. Refer to manufacturer's instructions for stability and storage information. Contains: 250 mg/L each: Li6, Sc; 50 mg/L each: Bi, In, Tb.

7.2.7.1.2. MSGE-100PPM (Custom Calibration Standard Stock Solution) purchased from CPI International. Refer to manufacturer's instructions for stability and storage information. Ge 100 mg/L.

7.2.7.1.3. Triton X-100 (Standard Stock Solution: Non-ionic Surfactant) purchased from Fisher Scientific, p/n BP151-

100. Refer to manufacturer's instructions for stability and storage information. According to the ICPMS manufacturer (Agilent Technologies), Triton-X 100 will reduce the 3 replicate %RSD during analysis.

7.2.7.2. Working Internal Standard Solution: add 100ml of reagent water to a clean 1000ml flask. Add 1 ml of Testamerica-1 and 1 ml of MSGE-100PPM, and 0.1 ml of Triton X-100. Bring to volume with reagent water.

The following are the recommended analysis masses, tune steps, and internal standards:

Element	IS
Be9	Li6
B11	Li6
Na23	Sc45
Mg24	Sc45
Al27	Sc45
K39	Sc45
Ca40	Sc45
Ti47	Sc45
V51	Sc45
Cr52	Sc45
Mn55	Sc45
Fe56	Sc45
Co59	Sc45
Ni60	Sc45
Cu63	Sc45
Zn 66	Sc45
As75	Sc45
Se78	Sc45
Sr88	In115
Kr83**	(none)
Mo95	In115
Ag107	In115
Cd111	In115
Sn118	In115
Sb121	In115
Ba137	Tb159
Tl205	Bi209
Pb 208*	Bi209

*Pb 208 = Pb 208 + Pb 207 + Pb 206
**Kr83 is used for monitoring Krpyton levels only.

Different masses and internal standards may be utilized, as matrix issues deem necessary. Non-drinking waters are analyzed using collision/reaction cell technology (helium/hydrogen gas mode). Typical non-drinking water Collision/Reaction Cell usage: Calcium, Iron, and Selenium are analyzed

in the Hydrogen reaction mode; Beryllium and Boron are analyzed in the No Gas mode; all other elements are analyzed in the helium collision mode. For drinking water analyses, collision cell technology is not allowed; therefore, the no-gas mode is utilized. Use masses Ca44, Fe57, Cu65, and Se82 instead of their respective masses listed above.

Tune check criteria:

Instrument tunes must be performed daily, before calibration. A solution at ~100ppb of Be, Mg, Co, In, and Pb is analyzed, and the precision, mass calibration, and resolution are checked. Resolution is checked by analyzing Magnesium isotopes 24, 25, 26 and Lead isotopes 206, 207, 208.

The following limits are used to evaluate mass calibration, resolution, and instrument stability:

The following limits are used to evaluate the tune:

Mass calibration: +/-0.1 amu
Resolution check: ≤0.9 amu at 10% peak height. Note: This also satisfies Method 200.8, 1.0 amu at 5% peak height)
Stability (5reps): <5%

7.2.7.3. EPA Tune Check Stock Standard: 2008TS purchased from Inorganic Ventures. Refer to manufacturer's instructions for stability and storage information. Contains: 10 ug/mL each: Be, Co, In, Mg, Pb.

7.2.7.4. Working EPA Tune Check Standard: add 100ml of reagent water to a clean 1000ml flask. Add 10 mL of 2008TS. Bring to volume with reagent water. This solution contains 100 ug/L of Be, Co, In, Mg, and Pb

7.2.8. Reporting Limit (CRI) Check standard:

For Non-Drinking Water: See Section 7.2.2, CAL1
For Drinking Water: See Section 7.2.2, CAL1.

7.2.9. ICP Interference Check Solutions: Stock solutions for ICSA and ICSAB, purchased from Inorganic Ventures (6020ICS-8A & 6020ICS-8B) and CPI International (TA-ICPMS-ICSA & TA-ICPMS-ICSAB), contains the following elemental concentrations:

6020ICS-8A contains: 18,000ppm Chloride, 3000ppm Ca, 2500ppm ea: Fe, Na, 2000ppm Carbon, 1000ppm ea: Al, K, Mg, P, S, 20 ppm ea: Mo, Ti

6020ICS-8B contains: 20ppm ea: Ag, Co, Cr3, Cu, Mn, Ni, V
10ppm ea: As, Cd, Se, Zn

TA-ICPMS-ICSA contains: 10000 mg/L Chloride; 2000 mg/L Carbon; 1000 mg/L ea: Al, Ca, Fe, K, Mg, Na, P, S; 20 mg/L ea: Mo, Ti

TA-ICPMS-ICSAB contains: 100 mg/L W; 10 mg/L ea: As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, Pb, Se, Si, Sn, Sr, V; 5 mg/L ea: Sb, Tl;

Single Element Standard: Ag 1,000 mg/L

Single Element Standard: Zn 1,000 mg/L

7.2.10. Working Interference Standards (ICSA/ICSAB):

For Non-Drinking Waters:

ICSA: Add 50ml of 5% HNO3 / 5% HCl (Sec 7.1.7) to a clean 100ml flask. Add 10ml of TA-ICPMS-ICSA stock solution. Bring to volume with 5% HNO3 / 5% HCl (Sec 7.1.6). This standard is made fresh weekly; store at room temperature. Refer to Attachment 2 for final elemental concentrations.

ICSAB: Add 50ml of 5% HNO3 / 5% HCl (Sec 7.1.7) to a clean 100ml flask. Add 10ml of TA-ICPMS-ICSA, 2 ml of TA-ICPMS-ICSAB, and 0.02 ml each of 1000 mg/L Ag and Zn. Bring to volume with 5% HNO3 / 5% HCl (Sec 7.1.6). This standard is made fresh weekly; store at room temperature. Refer to Attachment 2 for final elemental concentrations.

For Drinking Waters:

ICSA: Add 50ml of reagent water (Sec 7.1.7) to a clean 100ml flask. Add 2.5ml of 6020ICS-8A stock solution. Bring to volume with reagent water (Sec 7.1.6). This standard is made fresh weekly; store at room temperature. Refer to Attachment 2 for final elemental concentrations.

ICSAB: Add 50ml of reagent water (Sec 7.1.7) to a clean 100ml flask. Add 2.5ml of 6020ICS-8A and 0.5ml of 6020ICS-8B stock solutions. Bring to volume with reagent water (Sec 7.1.6). This standard is made fresh weekly; store at room temperature. Refer to Attachment 2 for final elemental concentrations.

7.2.11. ICP-MS Matrix Spiking Solution, ICPMS LCS/SPK: purchased from High-Purity Standards. Refer to manufacturer's instructions for stability and storage information. Refer to attachment 2 for final elemental concentrations. Stock solution contains the following elemental concentrations:

Element	Conc. Of Stock (mg/L)
Aluminum (Al)	500
Antimony (Sb)	5
Arsenic (As)	10
Barium (Ba)	10
Beryllium (Be)	5
Boron (B)	100
Cadmium (Cd)	5
Calcium (Ca)	500
Chromium (Cr)	10
Cobalt (Co)	5
Copper (Cu)	10
Iron (Fe)	500
Lead (Pb)	5
Magnesium(Mg)	500
Manganese (Mn)	50
Molybdenum (Mo)	10
Nickel (Ni)	10
Potassium (K)	500
Selenium (Se)	10
Silver (Ag)	5
Sodium (Na)	500
Strontium (Sr)	10
Thallium (Tl)	4
Tin (Sn)	10
Titanium (Ti)	10
Vanadium (V)	10
Zinc (Zn)	50

8.0. Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size[2]	Preservation	Holding Time[1]	Reference
Metals Waters except Hg	Polyethylene or Glass	250 mLs	HNO_3 to pH < 2 prior to shipment; if not, acidify upon receipt in lab [3,4]	6 months	40 CFR Part 136.3

[1] Inclusive of digestion and analysis.
[2] Drinking water samples may require 1000ml sample volume.

[3] Acid preservation may be omitted for shipping; however, acid must be added upon receipt in the lab. Following acidification, mix the sample and hold for at least 24 hours. Just prior to digestion or direct analysis, verify pH<2. If pH≥2, repeat steps (i.e., add acid, hold for 24 hrs, verify ph<2).
[4] Aqueous samples may be stored at room temperature.

Note: If drinking water samples are properly preserved with nitric acid to a pH<2 and the turbidity is <1 NTU, then acid digestion is not required. The laboratory must maintain documentation of the turbidity reading.

Samples for dissolved metals should be filtered in the field before acid is added to the sample. If the sample is to be filtered in the lab, no preservative is added to the sample until the sample is filtered.

9.0. Quality Control

Note: If a batch of samples requires digestion, then the relating QC samples must be carried through the entire digestion process.

9.1. Sample QC - The following quality control samples are prepared with each batch of samples.

Quality Control	Frequency	Control Limit
Preparation blank	One per batch of twenty samples or less	**200.8**: < RL or <10% sample concentration.
Lab Control Sample Water (LCSW)	One per batch of twenty samples or less	**200.8**: +/- 15%
Matrix Duplicate	One in 20 or fewer samples	20% RPD
Matrix Spike (200.8)[1]	One, if 10 or less samples in batch; two, if 11 or more samples	70-130%
Matrix Spike Duplicate	When requested by client	70-130%; If MS and MSD are both ≥ 5X CRDL, then 20% RPD. If MS and MSD are less than the CRDL, the RPD is not calculated; otherwise ±CRDL.
Post Digestion Spike	One per batch of twenty samples or less	See Section 9.1.6.4

[1] The sample for MS is randomly selected, unless specifically requested by a client.

9.1.1. Preparation Blank/Method Blank: One laboratory method/preparation blank will be analyzed with each batch of samples prepared together (not to exceed 20 samples). Preparation blank is used to identify possible contamination during acid digestion. results must be less than the reporting

limit (RL) or less than 10% of the determined analyte concentration for a sample. If any analyte concentration in the blank is above the control limit, the batch must be prepared again for the element in question and the samples reanalyzed.

9.1.2. Laboratory Control Sample Water (LCS): A laboratory control sample must be analyzed with each group of samples digested. Refer to Water Matrix Spike concentration in Attachment 1 for final elemental concentrations. Results must be within the acceptable control limits, if not, all samples prepared in association with the LCS must be redigested and reanalyzed.

- Method 200.8: wastewater samples - prepare the LCS by adding 0.25 ml of ICP-MS LCS/SPK (Sec. 7.2.11) in 50 ml deionized water. Results must be within ± 15% of the true value.
- Method 200.8: drinking water samples - prepare the LCS by adding 0.10 ml of ICP-MS LCS/SPK (Sec. 7.2.11) in 50 ml deionized water. Results must be within ± 15% of the true value.
- For dissolved aqueous samples not undergoing a preparation procedure, the CCV may serve as the LCS.

9.1.3. Matrix Duplicate: A duplicate is analyzed for each batch of samples digested. The relative percent difference between the results of the sample and the duplicate must fall within 20% RPD for samples greater than five times the reporting limit.

9.1.5. Matrix Spike (MS): A matrix spike is prepared and analyzed for each batch of samples digested.

- For aqueous MS sample, spike 0.25 ml of ICP-MS LCS/SPK (Sec 7.2.11) into 50 ml sample.
- For drinking water MS sample spike 0.10 ml of ICP-MS LCS/SPK into 50 ml sample.
- Method 200.8: a recovery of 70-130% is required.
  An exception to this occurs if the sample concentration exceeds the spike concentration by a factor of four or more. If the recovery is not within specified limits a post digestion spike is required to be analyzed at a concentration between 10 to 100 times the instrument detection limit. If the Post digestion spike recovery is not recovered within 85-115% for 200.8, a matrix effect should be suspected. See Attachment 1 for final Matrix spike concentration for water and soil.

9.1.6. Post-Digestion Spike: To check for possible matrix interference, analyze a post digestion spike on a representative sample (minimum of 1 per batch). Post-Digestion Spike sample should be evaluated if serial dilution fails.

9.1.6.1. Transfer 10mL of a digestate to a suitable vial.

9.1.6.2. Spike the sample with 0.01ml of ICP-MS LCS/SPK. Analyze the spiked aliquot and an un-spiked aliquot (the un-spiked may have been analyzed previously and does not need to be reanalyzed). The final concentration of the post digestion spike

is as follows:

Element	Post spike concentration (ug/L)
Aluminum (Al)	500
Antimony (Sb)	5
Arsenic (As)	10
Barium (Ba)	10
Beryllium (Be)	5
Boron (B)	20
Cadmium (Cd)	5
Calcium (Ca)	500
Chromium (Cr)	10
Cobalt (Co)	5
Copper (Cu)	10
Iron (Fe)	500
Lead (Pb)	5
Magnesium(Mg)	500
Manganese (Mn)	50
Mercury (Hg)	0.5
Molybdenum (Mo)	10
Nickel (Ni)	10
Potassium (K)	500
Selenium (Se)	10
Silver (Ag)	5
Sodium (Na)	500
Strontium (Sr)	10
Thallium (Tl)	4
Tin (Sn)	10
Titanium (Ti)	10
Vanadium (V)	10
Zinc (Zn)	10

9.1.6.3. Calculate the percent recovery of the post digestion spike as follows:

$$\%REC = \frac{Cps - Cs}{C2} \otimes 100$$

Where: Cps = concentration of post digestion spike (ug/L)
Cs = concentration of un-spiked sample (ug/L)
C2 = concentration of spike (ug/L

9.1.6.4. Limits for post digestion spike recoveries: 85-115%. Results must be within the acceptable limits, if not, repeat analysis and/or re-prepare the sample and reanalyze. If necessary dilute the QC sample (DU, MS, and PDS) to compensate for the matrix effect and reanalyze.

The post digestion spike must not be applied to samples analyzed at a dilution that produces a significant negative response. The analyst must use good judgment when evaluating data where the sample response is negative. Where a significant negative response is present, the digestate should be diluted and reanalyze to determine the extent of the matrix interferences. If necessary, adjust the interference corrections and reanalyze the samples.

9.1.7. Matrix Spike Duplicate (MSD): A matrix spike duplicate is prepared and analyzed when requested by the client. See Sec 9.1.5 for spiking procedures and acceptance limits.

- If matrix spike sample and matrix spike duplicate are both ≥ CRDL, then 20% RPD. If matrix spike sample and matrix spike duplicate are less than the CRDL, the RPD is not calculated; otherwise, ±CRDL.

9.2. Instrument QC

Table 2 below describes the frequency, criteria, and corrective actions for the calibration and Quality control samples.

Table 2: Calibration, Quality control and Corrective Action Summary

QC Item	Frequency	Criteria	Corrective Action
Tune validation-Mass Calibration	Daily	Within 0.1amu from mass unit	Terminate analysis, fix problem and repeat
Tune validation-Resolution Check	Daily	≤0.9amu full width at 10% peak height	Terminate analysis, fix problem and repeat
Instrument Stability Check	Daily	<5% RSD for five replicates	Terminate analysis, fix problem and repeat
Initial Calibration: Multi-point-minimum 3 stds and 1 blank	Daily	Correlation ≥0.995	Recalibrate
Initial Calibration Verification Standard (ICV)	At the beginning of the analysis	+/- 10%	Recalibrate
Continuing Calibration Verification Standard (CCV)	At the beginning and end of the analysis, and every 10 samples	Within +/- 10% of the true value	Fix the problem and reanalyze the previous 10 samples.
Calibration Blank (ICB/CCB)	After ICV and every CCV	Absolute value of the calibration blank must be less than the RL.	Terminate the analysis, correct the problem and reanalyze the previous 10 samples
Interference check standards (ICSA/ICSAB)	At the beginning of an analysis and after every 12 hours of analysis	Pay attention to false positives and false negatives for elements not present in the solutions. Results of the spiked elements should fall within +/-	If an element is consistently out of the +/- 20% range in the ICSAB solution, the problem should be investigated.

QC Item	Frequency	Criteria	Corrective Action
		20%.	
Serial Dilution (1/5 dilution)	One per batch of twenty samples or less	See Section 9.2.6	
Reporting Limit Check Standard (CRI)	At the beginning of analysis run	For *non-drinking water* analysis: Recovery +/- 50% of the true concentration. For *drinking water* analysis: Recovery +/- 15% of the true concentration	Stop the analysis, fix the problem and reanalyze the affected samples
Internal Standards	Analyzed with every standard and sample	200.8- 60-125%	Standard-terminate the analysis and recalibrate Sample- dilute (The analyst should check the IS response for the bracketing CCV/CCBs to see if there is a similar response to the sample. Recalibration may be required.)

9.2.1. Initial Calibration Verification (ICV): Analyze an initial calibration verification solution at the beginning of the run. ICV solution must have the same acid matrix as the calibration standard and it must come from a source other than the calibration standard source. The final concentrations of the various elements are listed in Attachment 1. The results for the target elements in the initial calibration verification (ICV) must be within +/-10% of the true value. If results are outside of the specified limits, terminate the analysis, correct the problem and recalibrate the instrument. See Sec 7.2.4 for the ICV standard preparation instructions.

9.2.2. Continuing Calibration Verification (CCV): The calibration of the ICP-MS must be verified every 10 samples and at the end of the analysis run by analyzing the QC Check Solutions (CCV). The same solution used for the calibration standard 4 (CAL4- drinking water; CAL3 for non-drinking water) is used for the CCV standard. The concentration of the CCV standard must be at or near mid-range levels of the calibration. The final concentrations of the various elements are listed in Attachment 1. The results for the target compounds must be within +/-10% of the true value. If results are outside of the specified limits, terminate the analysis, correct the problem, recalibrate the instrument, and reanalyze the samples following the last good CCV.

9.2.3. Initial and Continuing Calibration Blank (ICB/CCB): ICB and CCB must be analyzed after the calibration curve, every 10 samples and at the end of the analytical run. The absolute value of the calibration verification blank must not exceed the reporting limit. If it does, terminate the analysis,

correct the problem, recalibrate and reanalyze the samples following the last good CCB. Use reagent water for the blank solution.

9.2.4. Reporting Limit Check Standard (CRI): At the beginning of the analysis run verify the accuracy at the RL by analyzing a solution at the RL. RL/PQL Check Solution is analyzed to demonstrate that the ICPMS is capable of detecting the target compounds at the reporting limit (RL). For non-drinking water analysis, the determined concentration must be within +/-50% of the true concentration. For drinking water analysis, the determined concentration must be within +/-15% of the true concentration. See Attachment 1 for list of Rep Lim standard concentration in ug/L. Refer to Sec 7.2.8 for the RL/PQL standard preparation instructions.

9.2.5. Interference Check Sample (ICSA & ICSAB): Verify the inter-element and background corrections by analyzing the interference check solutions (ICSA & ICSAB) at the beginning of the analysis run and after every 12 hours of analysis. The ICS is analyzed in order to demonstrate that proper corrections are being utilized for known interferences. Analyst must evaluate these solutions in order to detect trends that require corrective actions. Pay particular attention to false positives and false negatives for elements not present in the interference check solutions. Results should be within 20% of the true value for each element. See Attachment 2 for list of elements and the corresponding concentrations in ppb.

9.2.6. Serial Dilution QC Check: A 1/5 dilution is prepared and analyzed on one sample per batch (one for every ten samples for Method 200.8, or whichever is greater) to determine if matrix interferences are present.

9.2.6.1. Select a sample digestate that contains one or more target analytes at concentrations greater than 10X the reporting limit.

9.2.6.2. Dilute the digestate by a factor of 5 (DF=5) and analyze the dilution using the same procedures used for the un-diluted aliquot.

9.2.6.3. Compare the results of the diluted and un-diluted aliquots of sample digestate.

9.2.6.4. If the results of the dilution are within ±10% of the results of the undiluted sample, no matrix interference is present. If not, matrix interference should be suspected and the sample digestate should be subjected to a post-digestion spike.

9.2.7. Determination of Linear Range of the ICP-MS

Determine the linear dynamic range of the instrument by analyzing a minimum of three high standards at the upper limit of the instrument. The analytically determined concentration of these standards must be within 10% of the true value. The linear ranges should be redetermined annually or if the instrument is significantly changed.

9.2.7.1. Prepare the standard at concentrations that are expected to define the linear range of the instrument. The calibration standards and the linear range standards should be matrix

matched; that is, they have the same percentage of hydrochloric and nitric acids. See Attachment 1 for Linear Range concentrations in ug/L.

9.2.7.2. Analyze the standard(s) after the initial calibration is validated.

9.2.7.3. Compare the concentration of the linear range standard with its true concentration using the following equation:

$$\text{Percent Difference} = \left|\frac{Ccal - Ctrue}{Ctrue}\right| \otimes 100$$

Where:
Ccal = concentration determined from analysis
Ctrue = true concentration of the standard

Note: If the percent difference is less than or equal to 10%, the linear range is confirmed at that concentration. If the percent difference is greater than 10%, repeat the analysis with a lower concentration. For elements validated in this manner you may report data up to 90% of that linear range before a dilution is required.

10.0. Procedure

10.1. Sample Preparation

10.1.1. For non-potable samples to be analyzed by Method 200.8, follow TestAmerica SOP No. ED-MTP-001.

10.1.2. For Drinking water samples, if turbidity is ≥1, follow the digestion procedure for Total recoverable in TestAmerica SOP No. ED-MTP-001.

10.2. Calibration

10.2.1. Turn the ICP-MS on and initiate the tune screen. Start the tune screen to allow the instrument to become thermally stable before beginning to analyze the calibration standards. While the instrument is warming up, aspirate the interference check solution (or similar solution) to precondition the cones.

10.2.2. Check the tune parameters for the proper sensitivities, precision, oxides, and double charged values following the instrument manufacturer's recommendations. Refer to sections *Preparations Before Analysis*, *Plasma ON/Executing a Startup*, and *Appendix (Recommended Values for Tuning Parameters)* of the MassHunter Workstation User Guide (*Agilent Technologies 7800/7900, p/n G7201-90403).*

10.2.3. Prior to calibration, run an EPA Tune check solution. Refer to Sec. 7.2.7 for the Tune check criteria.

10.2.4. The instrument is required to be calibrated daily or once every 24 hours. For Non-Drinking Water analysis, use instrument method *BM New Method.icpms.template.* For Drinking Water analysis, use instrument method *DW New Method.icpms.template*. These are programs in the Mass Hunter Workstation software containing calibration standards, check tables, etc. Calibrate the instrument using a blank and five standards. Refer to the section *Creating a Method* in the instrument manufacturer's instructions *Agilent ICP-MS (7800/7900) MassHunter Workstation User Guide (p/n G7201-90403)*.

10.2.5. Internal standards are added to the calibration standards, blank and samples in-stream by the use of T-fitting.

10.2.6. Analyze the calibration standards and calibrate the ICP-MS in accordance with the manufacturer's recommendations and the TestAmerica Corporate SOP No. CA-Q-S-005 Calibration Curves (General).

10.2.7. Calibration Curve Read-Back:

10.2.7.1. Low-Level readback – evaluate Cal 1 which is at the LLOQ. The %RE (relative error) must be +/- 50% (see Sec 11.4); if not, stop the analysis and recalibrate.

10.2.7.2. Mid-Level readback – evaluate Cal 4. The %RE (relative error) must be +/- 10% (see Sec 11.4); if not, stop the analysis and recalibrate.

10.3. Sample Analysis

10.3.1. All sample measurements must be made within 90% of the linear range of the instrument. Preparation of all reference materials used for calibration must be documented.

10.3.2. All potable and non-potable samples are typically analyzed undiluted. Dilutions must be prepared in reagent water (Sec 7.1.6 for Potable; Sec 7.1.7 for Non-Potable).

10.3.3. The samples are analyzed only after the ICB/CCB and ICV/CCV criteria are met.

10.3.4. The samples are analyzed in a sequence as follows:

Typical Analytical Sequence
INSTRUMENT WARM-UP (approx. 30 minutes)
TUNE CHECK
STANDARDIZATION/CALIBRATION (Blank, 5 Cal Stds)
ICV
ICB
CRI
ICSA
ICSAB

Typical Analytical Sequence
LRC-A (6020B)
LRC-B (6020B)
Rn Chk
Rn Chk
CCV
CCB
10 Samples
CCV
CCB
10 Samples
CCV
CCB
{continue until done; after 12 hrs run ICSA/ICSAB}

Note: The analytical sequence must end with the analysis of the CCV and CCB. The 10 samples include all QC samples/standards with the exception of CCVs and CCBs.

10.3.5. Determine the concentration of the samples and QC items using the procedures of Section 11.

10.3.5.1. If the sample concentration is greater than 90% of the linear range, the sample digestate must be diluted and reanalyzed.

10.3.5.2. The amount of sample digestate needed to prepare the desired dilution is determined from the following equation:

$$\text{V digest} = \frac{Vf\ vol}{DF}$$

Where:
Vf vol = final volume of diluted sample (mL)
V digest = volume of sample digestate used to make the dilution (mL)
DF = Dilution Factor

10.3.5.3. The dilution factor is calculated as follows:

$$\text{DF} = \frac{Vf\ vol}{V\ digest}$$

Where: Vf vol = final volume of diluted sample extract (mL)
V digest = volume of sample extract used to make the dilution (mL)

Note: The following examples are based on a final volume of 100mL. It may be more convenient to prepare dilutions at smaller final volumes.

EXAMPLE

A sample digestate is analyzed and one of the target analytes exceeds the linear range of the ICP-MS. 1.0mL of the digestate is added to a 100mL volumetric flask and the extract brought up to volume with reagent water. What is the dilution factor?

$$DF = \frac{100ml}{1.0ml} = 100$$

Some samples may require multiple dilutions; that is, a dilution of a dilution will have to be made. In this case, the final dilution factor is the product of the individual dilutions.

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Final results calculation:

$$\text{Concentration = ug/ L} = \frac{C \times V1 \times D}{V2}$$

Where:

C= Element concentration from instrument (ug/L)

V1= Final volume of sample digested (in liters)

D= Dilution performed on sample)

V2= Initial volume of sample digested (in liters).

11.4. Relative Error (%RE):

$$\text{\% Relative Error} = (100)\,(MC-TC)/TC$$

MC = Measured Concentration of the calibration standard
TC = True Concentration for the calibration standard

11.5. Data Reduction

11.5.1. All data is recorded directly into the TALS Analyst Desktop II program.

11.5.2. Standard and standard preparations must be documented in the Analyst Desktop II program located in TestAmerica Laboratory System (TALS). The analyst must enter the following information: Source standard, Initial and final sample volume, spike name and amount used, all reagents and their corresponding lot numbers.

11.5.3. Generate Raw Data PDF file for TALS

11.5.3.1. Using a scanner, scan all raw data, i.e., instrument printouts. This includes, the calibration, calibration curve info, and all samples analyzed (includes instrument and batch QC). Attach the pdf to the Docs section of the analytical batch in TALS (ADII).

11.5.4. Complete the Data Review Checker (DRC) in TALS: Prior to data submission (i.e., prior to 1st leveling data in the Analyst Desktop II module in TALS), the analyst must execute the DRC (Data Review Checker) program in the applicable analytical batch.

11.5.4.1. Open the analytical batch and click on the Edit tab above to enter the Edit Mode.
11.5.4.2. Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on 'Run checklist.'
11.5.4.3. Acknowledge by filling in responses to all unacknowledged findings.
11.5.4.4. Fill in appropriate comments in the response box, then hit 'OK.'
11.5.4.5. Acknowledge all Finding items in the 'Manual Batch Checklist' except for the *"2nd Level review complete?"* this is to be completed by the 2nd level reviewer.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for

analyses performed; these are verified at least annually unless method requirements require a greater frequency.

The IDL for EPA Method 200.8 is defined as the concentration equivalent to the analyte signal which is equal to 3 times the standard deviation of a series of ten replicate measurements of the calibration blank signal at the selected mass(es) and should be determined annually.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. 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 TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out.

- Digested Samples: Corrosive Acid- Materials that are not above regulatory limits will be submitted for elementary neutralization with 50% sodium hydroxide solution (Siedler Chemical SC-1824-03). Major concern is heat generated from the neutralization process. Initial volume of acid waste to be neutralized should be no more than 15 gallons. Finished neutralization with sodium bicarbonate (Siedler Chemical SC-0219-25) to a pH of 6 – 9 in the primary tank. Once pH has been established the primary tank is transferred through filter housing to a secondary tank. The pH is rechecked. If the pH is within specifications, the secondary tank is released to the municipal sewer system.

- Samples above regulatory limits and expired RCRA metals standards (Waste Corrosive Liquid, Acidic, Inorganic, n.o.s.) are collected in satellite accumulation and sent off site through a Waste disposal vendor.

Onyx Profile WIP Number: 590598
Teris Profile Number 50016653

- Expired Standards – The vials are collected in a 1 gallon polyethylene bucket. These vials are then transferred to an open top 55 gallon steel or polyethylene waste drum. These drums are transported to a waste facility for proper disposal.

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

  Teris Profile Number (incineration): 50016710
  Onyx Profile Number: (stabilization) 402535

15.0. References / Cross-References

15.1. Methods for the Determination of Metals in Environmental Samples; US EPA Office of Research and Development. Washington, DC, May 1994. Method 200.8, Revision 5.4.

15.2. TestAmerica Corporate SOP No. CA-Q-S-005, *Calibration Curves (General)*, most current revision.

15.3. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.5. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

15.6. TestAmerica Edison SOP ED-MTP-001, *Digestion of Aqueous Samples for Analysis by ICP and ICPMS USEPA Method No(s). 200.2, 200.7 (Rev 4.4), 200.8 & CLP SOW ISM01.2*, most current revision.

15.7. Agilent ICP-MS (7800/7900) MassHunter Workstation User Guide, p/n G7201-90403, Rev. A, June 2017

16.0. Method Modifications:

None

17.0. Attachments

Attachment 1: Standards and Matrix Spiking concentrations – Drinking water and non Drinking water.

Attachment 2: Interference Check standard elemental concentrations - Drinking water and non Drinking water.

18.0. Revision History

- **Revision 12, dated 18 June 2020**
  - oUpdated SOP header to Eurofins emblem.
  - oThroughout the SOP removed all SW846 Method 6020 references.
  - o For Non-Drinking Water preparations: Throughout the SOP, replaced all references to 2%HNO3/0.5%HCl with 5%HNO3/5%HCl.
  - o Sec 1.1, Table 1: Removed references to typical dilutions and updated the RLs for Non-Drinking Water.
  - o Sec 6.1: Added two ICPMS instruments, Agilent 7800 & 7900; removed reference to resolution and grating; added instrument mass range. Updated the models of the heat exchanger and autosampler.
  - o Sec 7.1.7: Added preparation instructions for 5%HNO3/5%HCl.
  - o Sec 7.2.1: Added CPI stock standard concentrations; CPI International is added as the vendor for the stock cal stds.
  - o Sec 7.2.2: Updated preparation instructions for the non-drinking water working cal standards and added preparation instructions for MS-Cal-Int.
  - o Sec 7.2.3: Updated the Initial calibration verification stock standards.
  - o Sec 7.2.4.2: Updated preparation instructions for the non-potable ICV working standard.
  - o Sec 7.2.4.2.1: Added preparation instructions for MS-ICV-Int.
  - o Sec 7.2.7.1.2: Replaced Inorganic Ventures with CPI International as the vendor source for the Ge stock standard.
  - o Sec 7.2.7.2: Updated preparation instructions for the Internal standard. Changed recommended mass for Ca (non-potable) from 44 to 40. Updated typical referenced internal std for As & Se from Ge to Sc. Updated typical analysis mode for non-potable Ca & Fe. Added Ca44 as the typical mass used for Calcium during potable analysis.
  - o Sec 7.2.9 & 7.2.10: Added ICSA/ICSAB stock standards from CPI International and updated Non-Potable preparation instructions for working ICSA & ICSAB.
  - o Sec 9.1 & 9.11: Updated control limit criteria for the Preparation blank.
  - o Sec 9.1.2 & 9.1.5: Updated preparation instructions for the LCS water samples and aqueous matrix spikes (MS).
  - o Sec 10.2.2: Added reference material, MassHunter Workstation User Guide, for the ICPMS instruments 7800 & 7900. Removed references to ICPMS1&2 (includes Chemstation software and relating Operation Manuals) from this SOP.
  - o Sec 10.2.4: Added instrument method program and reference material, MassHunter Workstation User Guide, for the ICPMS instruments 7800 & 7900.
  - o Sec 10.2.7.1 & 10.2.7.2: Added Low-Level readback and Mid-Level readback evaluation instructions.
  - o Sec 10.3.2: Updated typical dilution instructions.
  - o Sec 10.3.4: Updated Typical Analytical Sequence table to reflect current laboratory practices.
  - o Sec 11.3.2: Added final results calculation instructions.
  - o Sec 11.5: Added %Relative Error calculation equation.
  - o Sec 11.6.5: Added instructions for the Data Review Checker (DRC); removed reference to the Metals data review checklist EDS-WI-146, not applicable.

- Sec 15.3, 15.11, & 15.12: Deleted references for the Agilent 7500 Manuals, not applicable.
- Sec 15.13: Deleted reference to the ICPMS Data Review Checklist, CA-Q-WI-044.
- Sec 15.14: Added reference for the Agilent ICPMS 7800/7900 User Guide.
- Sec 18.0, Attachment 1 (Non-Potable): Updated calibration, ICV, CCV concentrations; removed dilution instructions.
- Sec 18.0, Attachment 2 (Non-Potable & Potable): Updated ICSA & ICSAB analyte list and concentrations.

- **Revision 11, dated 26 April 2018**
  - Sec 7.2.7.4: Added Magnesium as one of the working tune check solution's constituents.
  - Sec 9.1.2: Revised to clarify the acceptance criteria used in the COA when evaluating recoveries for LCS in soil matrix.

- **Revision 10, dated 15 November 2016**
  - Sec 1.1, Table 1: Changed the Drinking water RL from 2.5 ug/L to 2 ug/L. Removed reference to Method 3005.
  - Sec 2.0: Removed reference to method 3005 and replaced SOP ED-MTP-007 with SOP ED-MTP-001.
  - Sec 7.1.6: Reagent recording in ICPMS Reagent Dilution logbook is replaced with TALS Reagent Module.
  - Sec 7.2.1: Replaced stock standard TA-72 with standard TA-72REV2.
  - Sec 7.2.2: Replaced standard TA-72 with TA-72REV2 for CAL1 standard-Drinking Water.
  - Sec 7.2.7.2: Tune check criteria: updated the resolution check criteria to reflect laboratory practices.
  - Sec 7.2.10: Added the preparation instructions for ICSA & ICSAB standards used when analyzing drinking water samples.
  - Sec 9.2, Table 2: Updated Tune validation-Resolution Check criteria to reflect laboraotry practices
  - Sec 10.1: Replaced SOP ED-MTP-007 reference with SOP ED-MTP-001.
  - Sec 11.5.4: Replaced checklist CA-Q-WI-004 with Work Instruction EDS-WI-146.
  - Attachment 1, Drinking water: Replaced the Calibration concentration and RL Std.Conc. of Pb from 2.5 to 2 ug/L.
  - Attachment 2: Added ICSA & ICSAB elemental concentrations for Drinking water analysis.

- **Revision 9, dated 02 April 2015**
  - Throughout the SOP: Removed all reference to SOP# ED-MTP-002.
  - Sec 7.2.1: Added calibration standards TA-88 and TA-72.
  - Sec 7.2.2: Added the new low-level cal std (CAL2) for non-potable water and updated the preparation instructions for the potable cal standards.
  - Sec 7.2.7.1.3: Added Triton X-100 to the list of internal standard stock solutions.
  - Sec 7.2.7.2: Updated the preparation instruction of the working internal standard solution to include Triton X-100; Revised note to state that Selenium is now analyzed under the Hydrogen reaction mode.
  - Sec 7.2.8: CAL1 is now used as the RL check standard for drinking water analysis.

- Sec 9.1.6.4: Added PDS recovery limits (85-115%) for method 200.8. Clarified the procedure when PDS is outside the acceptable limits.
- Sec 9.1 and 9.1.7: Added Matrix Spike Duplicate (MSD) to the list of Sample QC.
- Sec 11.5.4: Updated the metals data review checklist form number.
- Sec 15.13: Added TestAmerica Corp form No. CA-Q-WI-044 to the list of references.
- Attachment 1 (non-Drinking Waters): Updated the calibration concentrations for all metals; updated the matrix spike concentration for Zinc (water & soil).
- Attachment 1 (Drinking Waters): Updated the calibration concentrations for As, B,Sn and Zn.

- **Revision 8, dated 05 December 2012**

  - Sec 1 and 12: Updated LQM section references to reflect the most current LQM revision.
  - Sec 2.0: Deleted the exception that silver analysis does not undergo digestion. Silver may be directly analyzed for drinking waters provided the turibidity <1.
  - Sec 7.1.6: Revised to include the reagent water prep for potable samples not requiring digestion. Revised preparation frequency to 12 months to reflect actual laboratory practices.
  - Sec 7.2.2 & 7.2.3: Sections deleted to remove Hg stock standard info and the preparation instructions of Hg intermediate standard; subsequent sections adjusted accordingly.
  - Sec 7.2.2 (formerly Sec 7.2.4): Removed all references to Hg. Clarified M1CAL3 as the reporting limit for Drinking Water standard. Clarified M1CAL2 is not the reporting limit for Boron.
  - Sec 7.2.3 and 7.2.4 (formerly Sec 7.2.5 & 7.2.6): Hg references have been removed.
  - Sec 7.2.6: Section added; subsequent sections adjusted accordingly.
  - Sec 7.2.7 (formerly Sec 7.2.8): Added 'EPA Tune Check' to the section header. Removed the use of gold standard from this SOP. Hg is not analyzed by this method.
  - Sec 7.2.7.1. Added internal standard stock solutions.
  - Sec 7.2.7.2: Added internal standard preparation instructions. Also clarified that drinking water samples are analyzed without the use of collision cell technology.
  - Sec 7.2.7.3: Added Tune Check stock standard
  - Sec 7.2.7.4: Added preparation instructions of the EPA Tune Check standard.
  - Sec 7.2.11 (formerly Sec 7.2.12): Changed standard vendor for the Matrix spike stock solution to High-Purity Standards.
  - Sec 8: Revised the wait time for sample preparation after in-lab preservation to 24 hours to comply with the updated 40CFR Part 136.
  - Sec 9.1: Preparation Blank's control limits have been updated to reflect method requirements.
  - Sec 9 and 10: Revised section references of standard solutions where applicable.
  - Sec 10.1.2: Revised SOP number reference to reflect actual laboratory practices.
  - Sec 10.2.2: Included Tuning Handbook reference for ICPMS2.
  - Sec 10.2.4: Included Operation Manual for Masshunter software to ICPMS2.
  - Sec 11.5: Added instructions for data recording, raw data PDF creations and Metals Data Review Checklist.
  - Sec 15.4: Deleted SOP No. ED-MTP-007 reference, not applicable; subsequent sections adjusted accordingly.
  - Sec 15.11 through 15.13: Added applicable references.
  - Sec 17.0: Attachments 3 and 4 have been removed.
  - Attachment 1: Deleted 'Linear range conc.' column; new linear range studies are

updated on instruments. Updated MS concentration for Boron.

- **Revision 7, dated 16 July 2010**

  - Sec 1.1 Table 1: Revised Reporting Limits for drinking water and Soil.
  - Sec 3: Revised definitions reference to Appendix 2 of ED-QA-LQM.
  - Sec 7.2.1: Added STLNJ-STD-1 and STLNJ-STD-2 to the list of Calibration Stds
  - Sec 7.2.4: Revised working calibration standard including preparation procedure to reflect actual lab practices.
  - Sec 7.2.9: Revised prep procedure for RL check standard. Also deleted RL/PQL working standard, previously section 7.2.10. Subsequent sections adjusted accordingly.
  - Sec 18.0 Attachment 1: Table updated to reflect correct calibration standard concentrations and drinking water reporting limits.

- **Revision 6, dated 11 September 2009**

  - Sec 9.2 Table 2 & Sec. 9.2.4: Revised Reporting limit criteria for drinking water analysis to ± 15%.

- **Revision 5, dated 08 July 2009**

  - Sec 4.1: Expanded section to include the isobaric polyatomic/molecular ion interferences; clarified using collision/reaction cell for non-potable samples only.
  - Sec 4.2: Added equations used for drinking water analysis when collision/reaction cell can not be used.
  - Sec 4.3: Added Krypton interference which affects arsenic and selenium, and the use of high purity Krypton free argon.
  - Sec 6.1 Instrumentation: Add collision/reaction cell.
  - Sec 7.2.4: Added a low calibration standard (M1CAL1) increasing the number of calibration standards to five; five cal standards will only be analyzed for elements Be, Fe, Pb and Mn.
  - Sec 7.2.8: Added Kr83 in the list of elements in order to monitor Kr levels.
  - Added text in Tune check criteria, Sec 7.2.8: *Resolution is checked by analyzing Magnesium isotopes 24, 25, 26 and Lead isotopes 206, 207, 208.*
  - Sec 8: Clarified holding time and procedure for acidifying unpreserved samples.
  - Sec 9: Added text: *If a batch of samples requires digestion, then the relating QC samples must be carried through the entire digestion process.*
  - Sec 9.1 & 9.1.2: Changed LCS control limit for Method 6020 from ± 25% to ± 20%.
  - Sec 9.2.2: CCV standard- changed solution to M1CAL4.
  - Sec 10.2.4: Calibration- changed the number of standards from three to four standards.
  - Sec 10.3.2: Added in the procedure the requirement for diluting samples 5x (water) and 20x (soil) prior to analysis; subsequent section numbers were adjusted accordingly.
  - Sec 10.3.5.1: revised to include that samples will be diluted if the concentration is >90% of the linear range.
  - Attachment 1: Revised calibration standard concentration specifically Cal std 2 to reflect the correct concentration. Additionally, a fifth cal standard was added to the four elements (Be, Fe, Pb, and Mn) so the RL will be at or above the low level standard; Reporting limits were also revised for these elements.

- Attachment 3: Revised dilution requirement for soil analysis from 10x to 20x.

- **Revision 4, dated 24 March 2009**
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Section 1.1: Relocated Table 1 to this section. Updated 6020 Soil RLs. Revised several RLs for Drinking Water and Digested Water
  - Sec. 2: Added the method and SOP references for digestion of samples to be analyzed for metals via ICPMS.
  - Sec 6.1: Added Heat exchanger and autosampler to the list of instruments.
  - Sec 7.1.6: Added reagent water and its prep instructions to the list of reagents.
  - Sec 7.2 Standards: Included vendor and storage information of standards.
  - Sec 7.2.2: Revised the vendor source and stock concentration of the Hg standard from 1000 ppm to 10 ppm; revised preparation instructions of the Intermediate standard (Hg-Cal-Int).
  - Sec 7.2.4: Revised prep instructions for the working calibration standards (i.e. amount of spiking solution and final volume).
  - Sec 7.2.5: Deleted ICV stock standards STLNJ-Cal 6 & 7 and added ICV stock standards ICVICPMS and ICV2ICPMS.
  - Hg Secondary stock solution in Sec 7.2.5: Revised the vendor source and stock concentration of the Hg secondary standard from 1000 ppm to 10 ppm; revised preparation instructions of the Intermediate standard (Hg-ICV-Int).
  - Sec. 7.2.8: added statement: “Drinking Water Analysis: All tune steps = 3 (thereby eliminating the use of the Collision/Reaction Cell.) Use masses Fe57, Cu65, and Se82 instead of their respective masses listed above.”
  - Sec 7.2.9: Deleted ICPMS RL stock standard and added custom standards (STLNJ-STD-1 & 2) for Reporting Limit Check standard.
  - Sec 7.2.10: Revised prep instructions for the RL/PQL Working standard.
  - Sec 7.2.11: Revised prep instructions for the Working Interference check standards (final volume reduced from 100ml to 50ml; spiking amount adjusted).
  - Sec 8: Sample collection, preservation and storage: Reformat section into Table format and included minimum sample size and reference information.
  - Sec. 9.1: Added prep instruction to the QC samples (MB, LCS, BS and MS).
  - Deleted Single point method of standard addition; previously identified Sec.9.2.8.
  - Sec 10.1: Clarified the digestion procedure utilized for digesting different matrices (wastewater, drinking water and soil).
  - Sec 15: Added applicable references.
  - Sec 17: Added Attachment 2 (Interference Check Standard elemental concentrations).

- **Revision 3, dated March 2007**
  - Section 16. Attachment 1. Modified the ‘Linear Range Concentration’ of 11 elements (Sb, Be, B, Cd, Co, Pb, Se, Ag, Sr, Tl, V) to reflect the most recent LDR study.

- **Revision 2**, **dated February 2007**
  - Revised various sections of the SOP to include the specific sections in the Operator’s manual where applicable information can be found. These sections are Sec. 5.3, 10.1.2., 10.1.3, Appendix A – Analytical Sequence Table.
  - Section 6.3 Calibration Standards. Modified the frequency for the preparation of calibration standards from six months to two weeks as per Method 200.8.

- Section 6.4. Linearity Check Standards. Revised the HNO3 acid concentration used in the preparation of linearity check solutions to '2% by volume' to reflect actual laboratory practices.
- Section 6.5 (Internal Standards). Revised the concentration of the element Au, both spiking and resulting concentrations.

  '*A solution of 1 mg/L (internal standard elements) and 2 mg/L of Au is prepared and added in-stream by the use of a T-fitting. The resulting concentration (in-stream) of the standard is ~100 ug/L (Au) and ~50 ug/L (internal standard elements).'*

- Section 7.4. Section deleted; contents not applicable to this SOP.
- Section 8.1.1 (MDL and IDL determination). Modified the solution used in the analysis of IDL to 'reagent blank solution' to reflect actual laboratory practices.
- Section 8.1.2 (MDL and IDL determination). Added an SOP reference to further describe MDL determination.
- Section 10.3.2. The name of the Reporting limit solution in the analysis sequence was changed from 'Detection Limit Check Solution' to 'Reporting Limit Standard (RL).'
- Section 15.1, deleted; reference not applicable. Subsequent section numbers adjusted accordingly.
- Section 15.1 (previously identified as section 15.2), was revised to include the specific method in the reference information, in this case method 6020.
- Section 15.2 (previously identified as section 15.3), was revised to include the specific method in the reference information, in this case method 200.8.
- Section 15.6. Added Operator's instructions manual in the list of references. The section previously identified as 15.6 is now 15.5.
- Section 16. Attachment 1. Modified the 'Linear Range Conc. (ug/L)' to reflect the most recent LDR study. Updated the 'RL Std. conc.' for Boron, Iron, Tin and Zinc to reflect actual laboratory's reporting limits for these elements.
- Appendix A (Sample Preparation). Specified the name of the SOP used in the digestion of samples.
- Appendix A (Calibration, Quality Control and Corrective action summary). Revised the criteria for the ICSA/ICSAB to clarify that ICSA when used as linear range sample the determined analyte concentrations >90% must be diluted and reanalyzed.
- Appendix B (ICV Solution). Added the frequency for the preparation of the ICV solution.

- **Revision 1, dated January 2007**

  - Specified Operator's manual '*Agilent 7500 ICP-MS Chemstation (G1834B)*' in various sections: Sections 5.3, 10.1.2, 10.1.3, Appendix A – Analytical Sequence section.
  - All references to '6020A' are updated to '6020' to reflect actual method reference (i.e. sections 1.1, 2.2, 8.1.2 etc.).
  - Section 10.1.3. Filename '2008.M' is specified as the program/filename used in the ChemStation software.
  - Section 16 Table 1. Updated Reporting limits of 8 elements (As, Cd, Co, Cu, Ni, Ag, Sr, Sn) to reflect current standard concentrations and laboratory's reporting limits.
  - Section 16 Attachment 1. Revised Matrix spike concentrations for water and soil to include the actual final expected concentrations of the matrix spike.

- Appendix B – Calibration, Quality Control and Corrective action summary. Criteria for internal standards was changed from >30% to 30-120% to reflect actual method criteria limits.
- Appendix B – ICP Interference Check Solutions for SW846 6020. Added the frequency of preparation for the Working Interference Standards ICSA and ICSAB, 'these standards are made fresh weekly.'
- Appendix B. Added data qualifiers for ICP-MS
- Section 15.4. Added USEPA CLP ILM05.3 to the list of references.

Attachment 1

STANDARDS AND MATRIX SPIKING CONCENTRATIONS
For Non-Drinking Waters

Name	Mass	Cal1 CRI	Cal-2	Cal-3	Cal-4/CCV	Cal-5	ICV	*LCS/MS Conc*
		(ug/L)	(ug/L)	(ug/L)	(ug/L)	(ug/L)	(ug/L)	*(ug/L)*
Be	9	0.8	4	50	100	200	80	*25*
B	11	80	400	125	250	500	200	*500*
Na	23	200	1000	12500	25000	50000	20000	*2500*
Mg	24	200	1000	12500	25000	50000	20000	*2500*
Al	27	40	200	12500	25000	50000	20000	*2500*
K	39	200	1000	12500	25000	50000	20000	*2500*
Ca	40	200	1000	12500	25000	50000	20000	*2500*
Ti	47	4	20	50	100	200	80	*50*
V	51	4	20	50	100	200	80	*50*
Cr	52	4	20	50	100	200	80	*50*
Mn	55	8	40	50	100	200	80	*250*
Fe	56	120	600	12500	25000	50000	20000	*2500*
Co	59	4	20	50	100	200	80	*25*
Ni	60	4	20	50	100	200	80	*50*
Cu	63	4	20	50	100	200	80	*50*
Zn	66	16	80	125	250	500	200	*250*
As	75	2	10	50	100	200	80	*50*
Se	78	2.5	12.5	50	100	200	80	*50*
Sr	88	4	20	50	100	200	80	*50*
Mo	95	4	20	50	100	200	80	*50*
Ag	107	2	10	25	50	100	40	*25*
Cd	111	2	10	50	100	200	80	*25*
Sn	118	16	80	50	100	200	80	*50*
Sb	121	2	10	50	100	200	80	*25*
Ba	137	4	20	50	100	200	80	*50*
Tl	205	0.8	4	50	100	200	80	*20*
Pb	208	1.2	6	50	100	200	80	*25*

STANDARDS AND MATRIX SPIKING CONCENTRATIONS
For Drinking Waters

Element	Mass	Calibration Conc. (ug/L) (stds: 4,3,2,1)	ICV/CCV Conc. (ug/L)	RL Std. Conc. (ug/L)	LCS/MS Conc. (ug/L)
Aluminum (Al)	27	1000,500,100,25	400/500	25	500
Antimony (Sb)	121	100,50,10, 2.5	40/50	2.5	5
Arsenic (As)	75	100,50,10, 1	40/50	1	10
Barium (Ba)	137	100,50,10, 2.5	40/50	2.5	10
Beryllium (Be)	9	100,50,10, 2.5	40/50	2.5	5
Boron (B)	11	200,100,20,20	80/100	20	100
Cadmium (Cd)	111	100,50,10, 2.5	40/50	2.5	5
Calcium (Ca)	44	10000,5000,1000, 250	4000/5000	250	500
Chromium (Cr)	52	100,50,10, 2.5	40/50	2.5	10
Cobalt (Co)	59	100,50,10, 2.5	40/50	2.5	5
Copper (Cu)	65	100,50,10, 2.5	40/50	2.5	10
Iron (Fe)	57	10000,5000,1000, 250	4000/5000	250	500
Lead (Pb)	208	100,50,10, 2	40/50	2	5
Magnesium(Mg)	24	10000,5000,1000, 250	4000/5000	250	500
Manganese (Mn)	55	1000,500,100, 25	400/500	25	50
Molybdenum (Mo)	95	100,50,10, 2.5	40/50	2.5	10
Nickel (Ni)	60	100,50,10, 2.5	40/50	2.5	10
Potassium (K)	39	10000,5000,1000, 250	4000/5000	250	500
Selenium (Se)	82	100,50,10, 2.5	40/50	2.5	10
Silver (Ag)	107	100,50,10, 2.5	40/50	2.5	5
Sodium (Na)	23	10000,5000,1000, 250	4000/5000	250	500
Strontium (Sr)	88	100,50,10, 2.5	40/50	2.5	10
Thallium (Tl)	205	20,10,2, 0.5	8/10	0.5	4
Tin (Sn)	118	100,50,10, 4	40/50	4	10
Titanium (Ti)	47	100,50,10, 2.5	40/50	2.5	10
Vanadium (V)	51	100,50,10, 2.5	40/50	2.5	10
Zinc (Zn)	66	100,50,10, 4	40/50	4	50

Attachment 2

Interference Check Standard (ICSA & ICSAB) Elemental Concentrations				
ELEMENT	Non-Potable		Potable	
	ICSA (ug/L)	ICSAB (ug/L)	ICSA (ug/L)	ICSAB (ug/L)
Sodium	100000	100000	62500	62500
Magnesium	100000	100000	25000	25000
Aluminum	100000	100000	25000	25000
Potassium	100000	100000	25000	25000
Calcium	100000	100000	75000	75000
Titanium	2000	2000	500	500
Iron	100000	100000	62500	62500
Molybdenum	2000	2000	500	500
Carbon	200000	200000	50000	50000
Chloride	1000000	1000000	450000	450000
Phosphorus	100000	100000	25000	25000
Sulfur	100000	100000	25000	25000
Silver	n/a	200	n/a	100
Arsenic	n/a	200	n/a	50
Barium	n/a	200	n/a	n/a
Beryllium	n/a	200	n/a	n/a
Cadmium	n/a	200	n/a	50
Cobalt	n/a	200	n/a	100
Chromium	n/a	200	n/a	100
Copper	n/a	200	n/a	100
Lithium	n/a	200	n/a	n/a
Manganese	n/a	200	n/a	100
Nickel	n/a	200	n/a	100
Lead	n/a	200	n/a	n/a
Antimony	n/a	100	n/a	n/a
Selenium	n/a	200	n/a	50
Silicon	n/a	200	n/a	n/a
Tin	n/a	200	n/a	n/a
Strontium	n/a	200	n/a	n/a
Thallium	n/a	100	n/a	n/a
Vanadium	n/a	200	n/a	100
Tungsten	n/a	2000	n/a	n/a
Zinc	n/a	200	n/a	50

Title: Mercury Analysis for Solid and Semisolid Waste Samples using the Leeman Mercury Analyzer (Cold Vapor Technique) by SW846 Method 7471B

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Approvals (Signature/Date):

Laura Demone	08/03/20	Donald Evans	08/03/20
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Carl Armbruster Quality Assurance Manager	Date	Mark Acierno Laboratory Director	Date
Dan Helfrich	08/02/20		
Dan Helfrich Healthy & Safety Manager Coordinator	Date		

1.0 Scope and Application

1.1. Analytes, Matrix(s), and Reporting Limits

SW846 Method 7471B is applicable to the determination of total mercury (organic and inorganic) in soils, sediments, bottom deposits, and sludge-type materials. All samples must be digested prior to analysis. If this digestion procedure is not sufficient to dissolve a specific matrix type or sample, then this method is not applicable for that matrix.

The typical detection limit using a 0.6 gram sample size is 0.017 mg/Kg Hg.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

The sample is digested as described in this SOP and is analyzed using cold vapor atomic absorption. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance at 253.7-nm is measured as a function of mercury concentration.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 Potassium permanganate is added to eliminate possible interferences from sulfide.

4.2 Copper may also be a potential interference although no effect has been observed for samples containing up to 10 mg/Kg Copper.

4.3 Samples high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 254 nm. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine hydrochloride reagent (25 mL).

4.4 Certain volatile organic materials that absorb at 253.7 nm may also cause interference. The analysis of the undigested sample should determine if this type of interference is present.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

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 MSDS 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 MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercury (1,000 PPM in Reagent)	Oxidizer Corrosive Poison	0.1 mg/M^3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	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.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	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.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Potassium Permanganate	Oxidizer	5 Mg/M^3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

6.1.1. Leeman Laboratories Inc. Hydra IIAA Automated Hg Analyzer

6.1.2. Computer and Monitor with Leeman Envoy software.

6.1.3. Top loader balance, 300gm capacity, and minimum sensitivity of ±1.0 mg

6.1.4. Hotblock digestor: Adjustable and capable of maintaining a temperature between 95 ±3°C.

6.2. Supplies

6.2.1 Pipettes and tips in various sizes

6.2.2 100 ml volumetric flasks

6.2.3 15 ml sample cups

6.2.4 Pump tubing:

- Sample, viton, blue tab
- Reductant, red tab

- Drain, blue tab
- Rinse, Black tab

.

6.2.5 Drying Tube – Purchased pre-packed with Magnesium Perchlorate from Leeman Labs. Located prior to the optical cell.

6.2.6 Nitrogen or Argon supply - capable of producing 80 PSI.

7.0 Reagents and Standards

Storage requirements: store at room temperature

Life of Reagent: Concentrated acids: refer to manufacturer's instructions
Laboratory prepared reagents and diluted acids: one year

Record reagent preparations in the TALS Reagent Module.

7.1 Reagents

7.1.1 Nitric acid - Concentrated (Trace Grade or Equivalent); store at room temperature; for stability information, refer to manufacturer's instructions

7.1.2 Hydrochloric acid-Concentrated (Trace Grade or Equivalent); store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.3 Potassium Permanganate (ACS Grade); store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.4 Sodium Chloride (analytical reagent grade); store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.5 Hydroxylamine Hydrochloride (ACS Grade); store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.6 Stannous Chloride (ACS Grade); store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.7 Deionized water - 18 megohm minimum

7.1.8 10% Hydrochloric Acid- Cautiously add 200 ml of concentrated HCl to a container and bring to final volume of 2 liters with deionized water. Store at room temperature; stable for one year.

7.1.9 Stannous chloride solution - Add 50 g of $SnCl_2$ to 500 ml 10% HCl solution.

Store at room temperature; stable for one year.

7.1.10 Sodium chloride/Hydroxylamine Hydrochloride solution - Dissolve 120 g of NaCl and 120 g of hydroxylamine hydrochloride in deionized water and dilute to 1 liter using deionized water. Store at room temperature; stable for one year.

7.1.11 Potassium permanganate ($KMnO_4$) 5% solution w/v - Dissolve 100 g of $KMnO_4$ in deionized water and dilute to 2 liters using deionized water. Store at room temperature; stable for one year.

7.1.12 0.15% Nitric Acid- Add approximately 5 liters of deionized water into an 8 liter carboy container. Cautiously add 12mL of concentrated HNO_3 and bring the final volume up to 8 liters with deionized water.

7.2 Standards

Storage requirements: all standards are stored at room temperature

Shelf-life: Stock standards – refer to manufacturer's instructions
Intermediate standards – made fresh daily
Working standards – made fresh daily
(Note: expiration date must not go beyond the expiration date of the source stock).

Concentration: see Attachment 1 for example certificates of analysis (COA) of Hg standards listed below. The COA lists the manufacturer's Lot number, certified concentration and shelf life.

Document standard preparation in TALS, see Sec 11.5.3.

7.2.1 Stock Mercury Calibration (10 ppm Hg) - Purchase from SCP Science; store at room temperature; for stability information, refer to manufacturer's instructions.

7.2.2 Secondary Stock Mercury Calibration Standard (10 mg/L Hg) - Purchase from Inorganic Ventures; store at room temperature; for stability information, refer to manufacturer's instructions.

7.2.3 Intermediate Calibration Standard (DCAL-Int), 100 ug/L Hg: Dilute 1 ml of Hg calibration stock standard solution (Sec 7.2.1) to 100 ml with 0.15% HNO_3.

7.2.4 Intermediate Initial Calibration Verification Standard (DQCS-Int), 100 ug/L Hg: Dilute 1 ml of Hg stock Calibration Verification standard, 10ppm Hg

(Sec 7.2.2) solution to 100 ml with 0.15% HNO_3.

7.2.5 Calibration Standards: Use six 50 ml hotblock cups to prepare the standards. Spike the calibrations standards cups as follows:

Calibration Standard	DCAL-Int Spike Volume (mL)	0.15% HNO_3 (mL)	Final Conc (ug/L)
Std1 (Cal Blk)	0	5	0
Std 2	0.1	4.9	0.2
Std 3	0.5	4.5	1
Std 4	1	4	2
Std 5	2.5	2.5	5
Std 6	5	0	10

Digest the standards following Sample preparation in Section 10.1.

7.2.6 Initial Verification Standard (ICV) 5.0 ppb: Using a hotblock cup, add 2.5 mL of 0.15 HNO_3 and 2.5 ml of DQCS-INT, (Sec 7.2.4). Digest the standard following Section 10.1. After digestion, the ICV will contain 5.0 ppb of Hg.

7.2.7 Continuing Calibration Verification Standard (CCV) 5.0 ppb: Follow the preparation instructions for Std 5 (see Sec 7.2.5)

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Soils	Plastic Glass	5 grams	Cool 4 ± 2°C	28 Days	SW846 Method 7471B

[1] Inclusive of digestion and analysis.

Samples are to be analyzed without drying. A separate procedure is used to determine the percent solids in the sample.

For sample homogenization procedures refer to TestAmerica Edison SOP ED-GEN-007 (Subsampling).

9.0 Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< RL; 5% of the regulatory limit; 5% of the measured concentration in the sample
Laboratory Control Soil Sample Reference Material (LCSSRM) in soil samples	1 in 20 or fewer samples	Vendor's certified limit
Matrix Duplicate (DUP)[1]	1 in 20 or fewer samples	If original sample and dup are both ≥ 5X RL, then 20% RPD. If original sample and duplicate are less than the RL, the RPD is not calculated; otherwise ±RL.
Matrix Spike (MS)[1]	1 in 20 or fewer samples	80-120%
Matrix Spike Duplicate (MSD)[2]	1 in 20 or fewer samples	80-120%; If original sample and dup are both ≥ 5X RL, then 20% RPD. If original sample and duplicate are less than the RL, the RPD is not calculated; otherwise ±RL.
Serial Dilution (SD)	1 in 20 or fewer samples	±10%

[1] The sample for DUP and MS are randomly selected, unless specifically requested by a client; Use the same environmental sample for the matrix spike and matrix duplicate sample whenever possible. If insufficient sample amount is available, another environmental sample may be used for the duplicate sample.

[2] A MSD is prepared and analyzed when requested by the client.

9.1.1. Method Blank: One laboratory method blank will be analyzed with each batch of samples prepared together (not to exceed 20 samples). The method blank is used to identify possible contamination during acid digestion. Results must be less than the RL, 5% of the regulatory limit for that analyte, or 5% of the measured concentration in the sample. If the analyte concentration in the method blank is above this control limit, the batch must be prepared again and the samples reanalyzed.

9.1.2. Laboratory Control Sample Soil Reference Material (LCSSRM: A laboratory control sample must be analyzed with each group of samples digested. For solid matrices, a vendor supplied solid matrix with certified values is carried through the same preparation procedure as the samples. The results of the solid LCS must fall within the 'QC Performance acceptance limits' of the reference material used for that sample. If not, all samples prepared in association with the LCS must be redigested and reanalyzed.

9.1.3 Matrix Duplicate (DUP): A duplicate is analyzed for each batch of samples digested. If original sample and duplicate are both ≥ RL, then 20% RPD. If original sample and duplicate are less than the RL, the RPD is not calculated; otherwise, ±RL.

9.1.4 Matrix Spike (MS): A matrix spike is prepared and analyzed for each batch of samples. A portion of sample is spiked with 0.5 mL of DCAL-Int (Sec 7.2.3). This is equivalent to 1.0 ppb Hg (on instrument). A recovery of 80-120% is required.

9.1.5 Matrix Spike Duplicate (MSD): A matrix spike duplicate is prepared and analyzed when requested by the client. A portion of sample is spiked with 0.5 mL of DCAL-Int (Sec 7.2.3). This is equivalent to 1.0 ppb Hg (on instrument). A recovery of 80-120% is required. If matrix spike sample and matrix spike duplicate sample are both ≥ RL, then 20% RPD. If original sample and duplicate are less than the RL, the RPD is not calculated; otherwise, ±RL

9.1.6 Serial Dilution (SD): A five fold serial dilution must be performed on one sample per batch. The sample should contain a sufficiently high concentration; minimally a factor of 25 times the estimated detection limit. Dilute the sample by a minimum of five fold (1+4) and reanalyze. The results must agree within 10% of the original determination. If not, a chemical or physical effect should be suspected.

9.2. Instrument QC

9.2.1 **Initial Calibration Verification (ICV):** Initial calibration is verified after calibration using an independent check standard at a concentration near the mid-point of the calibration (5.0ppb); see Sec 7.2.6 for preparation instructions. The results must be within 10% of the true value. If it's outside of the acceptable limits, terminate the analysis, correct the problem and recalibrate the instrument.

9.2.2 **Continuing Calibration Verification (CCV):** Calibration verification is performed after the calibration, after every 10 samples, and at the end of the run. Use a concentration of mercury at the midpoint of the calibration range (5.0 ppb). See Sec. 7.2.7 for preparation instructions. The value obtained must be within 10% of the true value. If not, stop the analysis and recalibrate. Re-analyze the previous ten samples following the last good calibration verification. .

9.2.3 **Initial and Continuing Calibration Blank (ICB/CCB):** ICB/ CCB must be analyzed after the calibration curve, every 10 samples, and at the end of the analytical run. The absolute value of the calibration verification blank must not exceed the reporting limit. If it does, terminate the analysis, correct the problem, recalibrate and reanalyze the samples following the last good CCB. The calibration verification blank is the same blank solution as used for the calibration blank.

10 Procedure

10.1. **Sample Preparation** (includes all samples, standards, and blanks)

10.1.1 Mix the sample well and weigh 0.5 - 0.6 grams of sample (including the LCSSRM) and place in the bottom of an appropriately identified 50 mL hotblock cup. For QA samples, weigh three portions of 0.5 - 0.6 grams of sample and place in the bottoms of three hotblock cups labeled as SAMPLE, DUP, and MS. Before adding any reagents, spike the MS sample with 0.5 mL of DCAL-Int standard.

10.1.2 Except the calibration standards, add 5 mL of deionized water to all hotblock cups (i.e., all field samples, MB, LCSSRM, Dup, and MS).

10.1.3 Add 1.5 ml concentrated HNO_3 and 4.5 ml concentrated HCl. Heat 2 min on the hotblock at 95 $\pm$3°C. Cool.

10.1.4 Add 15 mL deionized water and 15 mL potassium permanganate solution (Sec 7.1.11) to each hotblock cup. Mix well. The same amount of $KMnO_4$ must be added to the standards and samples.

10.1.5 Cap the hotblock cups loosely enough so that pressure does not build up

but also tight enough so that the caps stay on and that volume loss due to heating is minimized. Heat 30 min on the hotblock at 95 ±3°C. Cool.

10.1.6 To each hotblock cup, add 6 ml Sodium chloride - Hydroxylamine hydrochloride solution to reduce excess permanganate.

10.1.7 Using deionized water, bring to a 50 mL final volume. Cap and mix well.

10.2. Calibration

10.2.1 The instrument must be calibrated daily or once every 24 hours and each time the instrument is set up. The instrument is calibrated according to the manufacturer's specifications and must contain at least four standards and a blank. The laboratory currently uses five standards and a blank. The correlation coefficient of the calibration curve must be ≥0.995. If it does not, the problem must be corrected, and the instrument must be recalibrated. Standard preparations must be documented in the in TALS reagent module.

10.2.2 Prepare the calibration standards and Calibration Verification Standards as stated in Sections 7.2.5, 7.2.6., & 7.2.7.

10.2.3. Calibration Curve Read-Back:

10.2.3.1. Low-Level Readback (at the RL) – evaluate the 0.2 ug/L calibration standard. The %RE (relative error) must be +/- 20% (see Sec 11.4). If %RE is outside of the criteria limits, stop the analysis and recalibrate.

10.2.3.2. Mid- Level Readback – evaluate the 5.0 ug/L calibration standard (the mid-level calibration standard). The %RE (relative error) must be +/- 10% (see Sec 11.4). If %RE is outside of the criteria limits, stop the analysis and recalibrate

10.3. Sample Analysis

10.3.1 Following a sample digestion procedure, the samples are ready for instrumental analysis. It is advisable to investigate each matrix for any complexities, which might adversely affect the acquisition of valid data.

10.3.2 The following analytical run sequence is currently used for samples analyzed under Method 7471B:

Instrument Calibration (Blank and five standards)
ICV

ICB
CCV
CCB
10 Samples
CCV
CCB
10 Samples
CCV
CCB
Repeat until run is complete
CCV
CCB

10.3.3 Instrument Operation:

10.3.3.1 Turn on instrument, computer and monitor.

10.3.4 Plumbing the Reagent Lines:

10.3.4.1 One at a time, feed each of the pump tubes into a pump cassette, sliding the tube through the plastic clips at the bottom until the plastic tab is secure. Then, holding the tube taut, slide the loaded cassette onto the pump head and click the clamp, lever up. The tab end of the tube should be located at the front of the pump head.

10.3.4.2 Reductant (Red); Connect tab end of tube to the reductant bottle and the other end to the bottom of the mixing tee.

10.3.4.3 Sample (Blue); Connect tab end of tube to the autosampler probe and the other end to the top of the mixing tee.

10.3.4.4 Drain (Blue) Connect the tab end of tube to the sample discharge tube connected on the Liquid/Gas separator and the other end to the waste line.

10.3.4.5 Rinse (Black): Connect tab end of tube to rinse tubing that is connected to the rinse bottle. Connect the other end to the rinse tubing leading to the rinse cup.

10.3.5 Preparation of Reagents:

10.3.5.1 Pour the $SnCl_2$ solution into the reductant bottle and connect to the red reductant tube connector.

10.3.5.2 Pour the ten percent HCl solution into the Rinse reservoir bottle.

10.3.6 Starting Program:

10.3.6.1 Click the Envoy icon on the computer desktop
10.3.6.2 Click Method, select 7471_7471B, and select OK
10.3.6.3 On the main screen, click the StartUp icon. Wait 15 minutes before analyzing calibrating.
10.3.6.4 Click Sequence Tab on bottom of screen
10.3.6.5 Click Sequence on top of screen
10.3.6.6 Select Open, then select New
10.3.6.7 Using the Prep Batch Sheet and hand scanner, enter the sample barcodes into the Sample ID column. Include the Serial Dilution and all needed sample dilutions
10.3.6.8 Pour out the digested calibration standards and samples into the proper locations on the autosampler.
10.3.6.9 Click the Run Sequence icon to begin calibration and to run samples.

10.3.7 Creating the Raw Data PDF:

10.3.7.1 Click the Analysis Tab, select Detailed

10.3.7.2 Click Load, select Use This To Make PDF

10.3.7.3 Click Report, Click Printer

10.3.7.4 Include the calibration curve graph:

10.3.7.4.1 Go to Methods Tab and click Calibration, click Print

10.3.7.5 Combine both documents using the PDF Creator program. The new document is located in the Documents folder on the C:\ drive. Add this document to the “Doc’s” location in the analytical batch.

10.3.8 Shutting Down the Instrument:

10.3.8.1 Click the Stop icon on the main screen

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Concentration: (mg/Kg) = $\frac{C \times V1 \times D}{W}$

Where: C= Element concentration from instrument (ppb)
V1= Final volume of sample digested (in liters)
D= Dilution performed on sample
W= Initial weight of sample digested (in gram)

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

11.4. Relative Error (%RE):

% Relative Error = (100) (MC-TC)/TC

MC = Measured Concentration of the calibration standard
TC = True Concentration for the calibration standard

11.5. Data Processing:

11.5.1. All data is recorded directly in TALS' Analyst Desktop II program.

11.5.2. Import Data to TALS

11.5.2.1. Click the Analysis tab
11.5.2.2. Select the analytical run that needs to be imported
11.5.2.3. Select Statistics
11.5.2.4. Click Load and select TALS Import, click OK
11.5.2.5. Click Report, click CSV File
11.5.2.6. Name the import file (e.g., batch name, today's date)
11.5.2.7. The newly created import file is in the Import Folder on the desktop. Send to TALS Import Folder

11.5.3. Sample and standard preparations must be documented in the Analyst Desktop II program located in TestAmerica Laboratory System (TALS). The analyst must enter the following information: Source standard, Initial and final sample volume, spike name and amount used, all reagents and their corresponding lot numbers, creation and expiration dates.

11.5.4. All reagents must be recorded in TALS Reagent Module.

11.5.5. Complete the Data Review Checker (DRC) in TALS: Prior to data submission (i.e., prior to 1st leveling data in the Analyst Desktop II module in TALS), the analyst must execute the DRC (Data Review Checker) program in the applicable preparation batch.

11.5.5.1. Open the preparation batch and click on the Edit tab above to enter the Edit Mode.

11.5.5.2. Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on 'Run checklist.'

11.5.5.3. Acknowledge by filling in responses to all unacknowledged findings.

11.5.5.3.1. Highlight the checklist with findings, then highlight the associated list of findings; right click and choose 'Acknowledge Item.'

11.5.5.3.2. Fill in appropriate comments in the response box, then hit 'OK.'

11.5.5.3.3. Acknowledge all Finding items in the 'Manual Batch Checklist' except for the *"2nd Level review complete?"* this is to be completed by the 2nd level reviewer.

11.5.6. Record the following reagents and the volume used for sample preparation in the batch information page under "batch comments" in TALS Analyst Desktop II: concentrated sulfuric acid, concentrated nitric acid, potassium permanganate, potassium persulfate and sodium chloride-hydroxylamine hydrochloride. Record reagents in TALS by opening the prep batch, click on "edit" and then right click to choose "view batch information." Enter the information in the "batch comments" section.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Instrument Detection Limit

The IDL for each analyte must be determined for each wavelength used on each instrument. The IDL must be determined annually or if the instrument is adjusted

in any way that may affect the IDL. The IDL is determined by multiplying the average of the standard deviations obtained from the analysis of seven reagent blanks by 3.14.

12.3 Demonstration of Capabilities

For DOC procedure refer to Section 20 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.4 Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention.

14.0. Waste Management

14.1. 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 TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out:

- Digested Samples: Corrosive Acid- Materials that are not above regulatory limits will be submitted for elementary neutralization with 50% sodium hydroxide solution (Siedler Chemical SC-1824-03). Major concern is heat generated from the neutralization process. Initial volume of acid waste to be neutralized should be no more than 15 gallons. Finished neutralization with sodium bicarbonate (Siedler Chemical SC-0219-25) to a pH of 6 – 9 in the primary tank. Once pH has been established the primary tank is transferred through filter housing to a secondary tank. The pH is rechecked. If the pH is within specifications, the secondary tank is released to the municipal sewer system.

- Samples above regulatory limits and expired RCRA metals standards (Waste Corrosive Liquid, Acidic, Inorganic, n.o.s.) are collected in satellite accumulation and sent off site through a Waste disposal vendor.

Onyx Profile WIP Number: 590598
Teris Profile Number 50016653

15.0. References / Cross-References

15.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd ed.,U.S. Environmental Protection Agency, SW846 Manual, Method 7471B, Revision 2, January 1998.

15.2. Leeman Hydra IIAA Operating Manual.

15.3. TestAmerica Edison Document ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.5. Corporate Environmental Health and Safety Manual CW-E-M-001, most current revision.

15.6. TestAmerica Edison Subsampling SOP, *ED-GEN-007,* most current revision.

15.7. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

16.0. Method Modifications:

Item	Method #	Modification
1 (Sec 10.3.5.1)	7471B	Stannous Chloride is automatically added via the instrument versus the manual addition of Stannous Chloride as stated in the method. This is an instrument manufacturer's improvement that will reduce error due to loss of Mercury.
Sample preparation	7471B	The amount of DI water added to the sample at digestion is decreased to 15 ml from 50 ml (water bath procedure), while keeping the volume of reagents added during sample digestion the same. Sample final volume for hotblock procedure is modified to 50 ml (100 ml – water bath procedure) which allows for lesser sample volume hence less sample waste produced; hotblock digestion cup has maximum volume of 50 ml.

17.0. Attachments

Attachment 1: Certificate of Analysis of stock standards

18.0. Revision History

- Revision 4, dated 03 August 2020
  - Updated SOP header with Eurofin logo.
  - Sec 6.1.1 & 15.2: Replaced Hydra AA with Hydra IIAA.
  - Sec 9.1 Table and 9.1.1: Revised MB control limits to <RL.
  - Sec 9.1, 9.1.4 and 9.1.5: Revised MS and MSD acceptance limits to 80-120%.
  - Sec 9.2.2: Revised CCV acceptance limits to +/-10%.
  - Sec 10.2.3: Added calibration curve read-back criteria.
  - Sec 11.4: Added Relative % error calculation.
  - Sec 11.5: Added Relative % error calculation.
  - Sec 11.5.6.: Added requirements to record reagent volumes in the batch information page of TALS ADII; subsequent sections adjusted accordingly.

- Revision 3, dated 12 October 2018
  - Sec 6.1.2: Removed WinHg instrument software reference and replaced with current instrument software, Envoy.
  - Sec 9.1.2: Revised to clarify the acceptance criteria used in the COA when evaluating recoveries for LCS in soil matrix
  - Sec 10.3.6: Updated the Starting Program instructions to reflect the new instrument software, Envoy.
  - Sec 10.3.7 & 10.3.8: Updated instructions for creating raw data pdf and shutting down instrument.
  - Sec 11.4.2: Updated instructions for importing data to TALS.
  - Sec 11.4.5: Replaced Metals Data Review Checklist with Data Review Checker (DRC) in TALS.
  - Sec 15.8 & 15.9: Removed reference to EDS-WI-007 & EDS-WI-125; WI not applicable.

- Revision 2, dated 29 Nov 2016
  - Sec 6.1.4, Sec 10.1.3, & 10.1.5: updated the hotblock temperature range per method 7471B requirements
  - Sec 6.2.7: added Argon gas
  - Sec 7.2.4: the name of the intermediate ICV standard has been changed from DICV-Int to DQCS-Int
  - Sec 7.2.6: removed the CCV preparation instructions using the second source standard. The name of the intermediate ICV standard has been changed from DICV-Int to DQCS-Int
  - Sec 7.2.7: added preparation instructions for the CCV using the primary source standard
  - Sec 9.1: added the quality control sample MSD to the table
  - Sec 9.1.5: added quality control information for the MSD
  - Sec 11.4.5: Metals Data Review Checklist control # has been updated from ED-WI-007 to CA-Q-WI-042

- Revision 1, dated 12 May 2014
  - Sec 1.1: Revised the typical detection limit from 0.033 mg/kg to 0.017 mg/kg to reflect the 50mL final volume – hotblock procedure.
  - Sec 1 & 12: Updated LQM section references to reflect the most current LQM revision.
  - Sec 6.1.4: Added hotblock digestor to the list of equipment; removed autoclave from the list.
  - Sec 6.2: Added 50 mL hotblock digestion cups to replace the BOD bottles. Removed supplies which are no longer applicable (BOD bottles, graduated cylinders).
  - Sec 7.0 & 11.4.4: Removed Hg Reagent Logbook and replaced with TALS Reagent Module
  - Sec 7.1. Deleted Sulfuric Acid (previously referenced in Sec 7.1.1). Added 0.15% HNO_3 to list of reagents.
  - Sec 7.2.5: Updated Calibration standards' spiking instructions to reflect the 50 ml final volume.
  - Sec 7.2.6: Updated ICV/CCV spiking instructions to reflect the 50 ml final volume.
  - Sec 8.1: Added plastic container to the list of acceptable sample containers.
  - Sec 9.1 & 9.1.3: Clarified the acceptance limits for Matrix duplicate to reflect actual laboratory practices.
  - Sec 9.1.4: Revised the spiking amount added to the MS sample.
  - Replaced LCSS reference with LCSSRM in various sections of the SOP to reflect TALS QC type reference.
  - Sec 10.1: Revised sample preparation procedure to include hotblock digestor procedure; deleted all reference to autoclave digestion procedure.
  - Sec 11.4.5 & 15.9: Added Work instructions EDS-WI-125 (TestAmerica Edison Metals Initial Calibration Data Review for Mercury).
  - Sec 16: Added Sample prep method modification (i.e. final volume).

- Revision 0, dated 04 February 2011
  New

Attachment 1

Certificate of Analysis

Catalogue Number : 141-110-111/141-110-112/141-110-115
Description : *Plasma*CAL - **ICP-MS Verification Standard 1 Solution B**
Lot Number : **SC8298812**
Expiration Date : **January 2010**

Analysis of Solution Standard by Inductively Coupled Plasma Spectroscopy (ICP-AES) traceable to NIST Standard Reference Materials : 3133

Actual Concentrations

Hg : 9.98 µg/ml

Matrix : **10% HNO_3**
Density : **1.040 g/ml @ 22.0°C**

MS 0882
Rec'd
1/14/09

Certified by : _____
Thomas Znoj, Chemistry Manager

Certification Date : **October 28, 2008**

This ICP-AES & ICP-MS Standard is guaranteed to be stable and accurate to within plus or minus 1.0% of the actual concentration up to the expiry date, provided the solution is kept tightly capped and stored under normal laboratory conditions. For these solutions, 18 megohm/cm double deionized water, high-purity acids, Class A glassware and acid-cleaned bottles are used. The Material Safety Data Sheet and this Certificate of Analysis are available on our web site. (Également disponible en Français)

Manufactured according to an ISO 9001:2000 Quality System and ISO 17025 (in-process)
SCP SCIENCE
21800 Clark Graham, Baie D'Urfé, QC, Canada H9X 4B6
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CERTIFICATE OF ANALYSIS

MS0879
Rec'd 1-12-09

tel: 800.669.6799 · 732.901.1900
fax: 732.901.1903
info@inorganicventures.com

1.0 **INORGANIC VENTURES** is an ISO Guide 34 "General Requirements for the Competence of Reference Material Producers" and ISO 9001:2000 registered manufacturer. Our manufacturing laboratory is accredited to ISO/IEC 17025 "General Requirements for the Competence of Testing and Calibration Laboratories."

2.0 **DESCRIPTION OF CRM** **10 µg/mL Mercury in 10% (v/v) HCL**

Catalog Number:	MSHG-10PPM
Lot Number:	**B2-HG02061**
Starting Material:	Hg metal
Starting Material Purity (%):	99.999549
Starting Material Lot No:	05214TX
Matrix:	10% (v/v) HCL

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

Certified Concentration: 10.027 ± 0.020 µg/mL

Certified Density: 1.019 g/mL (measured at 22° C)

The following equations are used in the calculation of the certified value and the uncertainty. Reported uncertainties represent expanded uncertainties expressed at approximately the 95% confidence level using a coverage factor of k = 2.

Certified Value $(\bar{x}) = \frac{\Sigma x_i}{n}$

$(\bar{x})$ = mean
x_i = individual results
n = number of measurements

Uncertainty $(\pm) = \frac{2[(\Sigma s_i)^2]^{1/2}}{(n)^{1/2}}$

Σs_i = The summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume, and the fixed error reported on the NIST SRM certificate of analysis.)

4.0 **TRACEABILITY TO NIST AND VALUES OBTAINED BY INDEPENDENT METHODS**

- "Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties." (ISO VIM, 2nd ed., 1993, definition 6.10)
- This product is Traceable to NIST via an unbroken chain of comparisons to the following NIST SRMs:

Title: Extraction of Semi-Volatile Organic Compounds in Aqueous Samples and Leachates - Separatory Funnel, SW846 Method 3510C

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Sylvanus Klusey	03/26/2018 Date	Dan Helfrich	03/26/2018 Date
Organics Operations Manager		Health & Safety Manager / Coordinator	
Carl Armbruster	03/26/2018 Date	Mark Acierno	03/26/2018 Date
Quality Assurance Manager		Laboratory Director	

1.0 Scope and Application

1.1. **Analytes, Matrix(s), and Reporting Limits** SW846 Method 3510C describes a procedure for isolating semivolatile organic compounds from aqueous samples and leachates, including concentration techniques suitable for preparing the extract for GC/MS analysis. This SOP is applicable to the isolation and concentration of water-soluble and slightly water-soluble semivolatile organics in preparation for analysis by SW846 Methods 8270C or 8270D.

1.2. For a complete discussion of analytes and reporting limits (RLs) please refer to TestAmerica Edison SOP Nos. ED-MSS-002, *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) SW846 Method 8270C*, current revision and ED-MSS-009, *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) SW846 Method 8270D*, current revision

1.3. On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work Request*) and 19 (*Test Methods and Method Validation*) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1. A measured volume of sample (~250 mL) is serially extracted with methylene chloride at a pH less than 2 and again at a pH greater than 11 using separatory funnel extraction. The methylene chloride extract is dried and concentrated to a volume of approximately 2 mL. Nitrogen blowdown is employed as the final concentration step. The extract is subsequently analyzed by SW846 Method 8270C or 8270D (GC/MS) by a large volume injection (LVI) technique. This procedure is referred to throughout as Reduced Volume Extraction (RVE) and Large Volume Injection (LVI).

2.2. An option for preparing aqueous samples using a larger initial volume (~1000 ml) is also described. This procedure is referred to as 'large volume' throughout the document.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Solvents, reagents, glassware, and other sample hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks.

4.2. Phthalate esters contaminate many products commonly found in the laboratory. Plastics, in particular, must be avoided, because phthalates are often used as plasticizers and are easily extracted from plastic material.

Phthalate contamination may result at any time if consistent quality control is not practiced.

4.3. The decomposition of some analytes has been demonstrated under basic extraction conditions. Phthalate esters may exchange and phenols may react to form tannates. These reactions increase with increasing pH, and are decreased by shorter reaction times. Performing the initial extraction at an acid pH will optimize the recovery of phenols

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard.

The following analytes have been tentatively classified as known or suspected, human or mammalian carcinogens: benzo(a)anthracene, benzidine, 3,3'dichlorobenzindine, benzo(a)pyrene, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, dibenz(a,h)anthracene, N-nitrosodimethylamine, 4,4'-DDT, and polychlorinated biphenyl compounds. Primary standards of these toxic compounds should be prepared in hood.

The use of Kevlar gloves is required for the assembly/disassembly of ground glass joints in addition to those tasks that present the potential risk for injury.

The use of separatory funnels to extract aqueous samples with Methylene Chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the sample container has been sealed and inverted, periodic venting may be necessary during the extraction. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, the use of a face shield over safety glasses or goggles is recommended. Keep the sash on the fume hood as low as reasonably possible.

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 MSDS 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 MSDS for each material before using it for the first time or when there are major changes to the MSDS.

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.
Sulfuric Acid	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.
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.
Methanol (MeOH)	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.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

- Separatory funnel rotator, APR Machine or equivalent
- Analytical Evaporator (N-Evap) Organomation
- Centrifuge, Varifuge F; Hereaus Sepatech
- Six Position Steam Bath, Fisher 15-496 or equivalent

6.2. Supplies

- 250 ml Erlenmeyer Flask, AMK Glass ERL-0252 or equivalent
- 2000 ml or 500 ml Separatory Funnel, AMK Glass SFC or equivalent
- 100 mm o. d. glass funnels, Fisher or equivalent

- 10 ml jacketed, graduated Concentrator Tubes, AMK Glass KD-0018 or equivalent
- 19/22 Ground Glass Stoppers
- 3 Ball Snyder Columns, TEC Glass TG6-03 or equivalent
- 1 ml Gastight Syringe, Hamilton 81317 or equivalent
- 150 ml Centrifuge Tube
- 100 ml Graduated Cylinder
- Pasteur 5 ¾" Disposable Pipets, Fisher 13-678-20B or equivalent
- Kuderna Danish Flask (500 ml), TEC Glass TG7-01 or equivalent
- Vials, 2ml amber screw cap with Teflon liner
- Glass Wool
- Dessicator
- Standard Taper Clamps (Size 19, blue)
- Boiling Stones, Troemner P/N 133-B or equivalent, rinsed with Methylene Chloride
- pH Paper
- Watch Glass
- Wax Pencil
- 1 Liter Graduated Cylinder
- Marking Tags

7.0 Reagents and Standards

7.1 Reagents

Note: Each lot of Methylene Chloride, Acetone, Methanol and Sulfuric Acid is screened for contaminants before being used for analysis as detailed in TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7.1.1 Methylene Chloride – JT Baker Ultra-Resi 9254-03 or equivalent

7.1.2 Acetone, J.T. Baker Ultra-Resi 9264-03 or equivalent

7.1.3 Methanol, J.T. Baker, Pesticide Grade, 9077-02 or equivalent

7.1.4 Concentrated Sulfuric Acid - Baxter 2876-9 or equivalent

7.1.5 Sodium Hydroxide Pellets – Baxter 7708-500NY or equivalent

7.1.6 Sodium Sulfate Crystals – Mallinckrodt MA8024-06 or equivalent (Must be baked in the muffle furnace for four hours at 400°C and serially rinsed with Methylene Chloride prior to use.)

7.1.7 Sodium Hydroxide (10 N) - Fill a precleaned 1000 ml volumetric flask with 500 mls deionized water. Weigh out 400 g NaOH pellets and dispense slowly into the flask. Stir slowly until the pellets dissolve, then add more deionized water until the 1000 ml level is

reached. Be careful as this procedure generates heat. Never add water to the reagent that is to be dissolved.

7.2 Standards

7.2.1 Most stock target analyte standard solutions are purchased as prepared solutions; other standards are prepared in the laboratory using neat compounds (see table below). Most stock solutions are diluted (in volumetric glassware) to working concentration using methylene chloride as the diluent as described below. Stock standards of similar quality from other suppliers may be substituted as required.

NOTE: The standards listed here are used as calibration standards and spiking standards. Separate source calibration verification standards are addressed in the analytical SOPs.

Standard Name	Concentration	Vendor	Catalog #
8270 List 1/ Std#1 MegaMix	500/1000/2000pmm	RESTEK	570666
8270 List 1/ Std#10Benzoic Acid	2000ppm	RESTEK	569731
8270 List 1/ Std#9	2000ppm	RESTEK	569730
Custom SVO Mix	2000ppm	SPEX	SVO-TANJ-16-5
Bisphenol A	1000ppm	SPEX	S-509-MC
8270 List 1/ Std#11	2000ppm	RESTEK	569732
8270 Surrogate Standard	5000 ppm	RESTEK	567685
Aromatic Amines Custom Mix	2000 ppm	Supelco	21467482

7.2.1.1. **Spiking Standard:** For use in spiking aqueous samples including TCLP/SPLP leach being prepared for BNA analysis by SW846 Method 8270. Prepare the second source spiking solution for MS/MSD/Blank Spike (LCS) as detailed in the following table using the applicable standards listed in 7.2.1. Use volumetric glassware. Dilute to the volume marker with methanol.

Standard Name	Concentration	Volume of Standard added to final volume of 200ml (solvent)	Final Concentration
8270 List 1/ Std#1 MegaMix	500/1000/2000pmm	20 ml (methanol)	50/100/200 ppm
8270 List 1/ Std#10	2000ppm	10 ml (methanol)	100 ppm
8270 List 1/	2000ppm	10 ml (methanol)	100 ppm

Standard Name	Concentration	Volume of Standard added to final volume of 200ml (solvent)	Final Concentration
Std#9			
Custom SVO Mix-SPEX	2000ppm	10 ml (methanol)	100 ppm
Bisphenol A-SPEX	1000ppm	10ml (methanol)	50ppm
8270 List 1/ Std#11	2000ppm	20ul (methanol) 100ul(methanol)	Used neat for LVI Used neat for non-LVI
Aromatic Amines Custom Mix**	2000ppm	10 ml (methylene chloride)	100 ppm

*** As needed based on clients requirement.*

Note: Neat spiking/surrogate standards are stored per vendor requirements (either room temp or 4 deg C, as appropriate). All prepared standard solutions are refrigerated at 4 deg C.

7.2.2 **8270 Surrogate Standard Spiking Solution**: For use in spiking all blanks, samples and associated QC prior to extraction. Prepare as detailed in the following table using the applicable standards listed in 7.2.1. Use volumetric glassware. Dilute to the volume marker with methanol.

Standard Name	Concentration	Volume of Standard added to final volume of 1000 ml methanol	Final Concentration
8270 Surrogate Standard	5000 ppm	20 ml	100 ppm

7.2.3 Internal standard is prepared added by the analytical department. For details see analytical SOPs No. ED-MSS-002 (SW846 8270C) or ED-MSS-009 (SW846 8270D).

7.2.4 The preparation of all standards must be documented in TestAmerica LIMS (TALS) or a standard preparation logbook. Information such as standard supplier, lot number, original concentration, and a description of how standard was prepared are required along with a laboratory lot number, analyst's initials, date prepared and verification signature. Standards must be made every 6 months or sooner if signs of degradation appear.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

8.1 All samples must be stored at 4ºC (± 2ºC) upon receipt.

8.2 Sample Extract Storage. Samples extracts must be protected from light and refrigerated at 4ºC (± 2ºC) from time of extraction until analysis.

8.3 Sample Extract Holding Time. All sample extracts must be analyzed within 40 days of extraction.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water or Leachate	250ml Amber (RVE)/ Amber glass, 1L	250ml-RVE/ 1000 ml	Cool 4 ± 2ºC	7 days to extraction; Analyze within 40 days of extraction	EPA Method SW846 8270C

9.0 Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of samples. Refer to TestAmerica Edison analytical SOPs No. ED-MSS-002 (SW846 8270C) or ED-MSS-009 (SW846 8270D) for details on analysis and evaluation of these QC elements:

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD, Method Blank)
[4] Statistical control limits are updated annually and are updated into lab reporting software.

9.1.1. **Method blanks** are extracted with every sample batch on each day that samples are extracted.

9.1.2. **Matrix Spike (MS)/Matrix Spike Duplicate (MSD):** A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. (Note: an

LCS/LCSD may be substituted for the MS/MSD if insufficient client environmental sample volume is available).

9.1.3. Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD): A Laboratory Control Sample (LCS) (aka blank spike) must be extracted and analyzed with each batch of 20 environmental samples.

9.1.3.1 A Laboratory Control Sample Duplicate (LCSD) is extracted and analyzed only when insufficient client sample is available for preparation of an MS/MSD pair. The LCS/LSCD is evaluated in the same manner as the MS/MSD (see Section 9.1.2)

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with a six (6) component surrogate standard mix (see Section 7.2.2).

10.0 Procedure

NOTE: The sample preparation procedure for aqueous samples (Section 10.1) contains two options: reduced volume extraction (RVE) (250ml) for large volume injection (LVI) analysis and large volume extraction (1000ml),

10.1. Sample Preparation for Aqueous/ Leachates Samples

10.1.1. Rinse the required number of 500-ml separatory funnels (when RVE is required) or 2000-ml separatory funnels (when large volume extraction is required) and 250-ml Erlenmeyer flasks twice with a 1:1 mixture of Methylene Chloride:Acetone and once with Methylene Chloride.

10.1.2. Place a small amount of glass wool into a 100-mm funnel and fill with pre-baked sodium sulfate crystals. Rinse three (3) times with Methylene Chloride. Also rinse the outside of the funnel stem three (3) times with Methylene Chloride (since the stem is likely to come into contact with the extract). Allow time for all of the rinsate to drain out of the funnel into a waste container.

10.1.3. Record the lab sample numbers on the separatory funnels with red wax pencil.

10.1.4. Make up tags with the following information and place on a 250ml Erlenmeyer flask:

BNAs		**BNs**	**AEs**
Acid Fraction	BN Fraction		
Sample Number	Sample Number	Sample Number	Sample Number
Fraction-Matrix	Fraction-Matrix	Fraction-Matrix	Fraction-Matrix
Date of Extraction	Date of Extraction	Date of Extraction	Date of Extraction

10.1.5. Place the 100ml funnel containing rinsed sodium sulfate crystals onto the flask.

10.1.6. Mark the fluid level on the sample bottles with a black Sharpie. Pour each sample into its corresponding separatory funnel. Fill each sample bottle to the black line with tap water. Pour this into the graduated cylinder used for measuring sample volumes. Note the volume for each sample on the Organic Extraction Data Sheet.

10.1.7. Rinse out a graduated cylinder with lab reagent water two to three times. Using the graduated cylinder obtain 250 ml when RVE is used (or 1000-ml when large volume is required) of lab reagent water from the Millipore filtering apparatus located in the Wet Chemistry laboratory for each of the method blank and the laboratory control sample (LCS) (aka blank spike).

10.1.8. Pour each the reagent water for the method blank and LCS into the corresponding separatory funnels.

10.1.9. Rinse syringes eight (8) to ten (10) times with Methylene Chloride.

10.1.10. If you are performing BNAs add 0.2 ml when RVE is used (or 1.0 ml when large volume is required) of Surrogate Standard Spiking Solution (see Section 7.2.2) to each sample/QC sample and proceed to Section 10.1.11. If you are performing □□□□□□ extraction, add 0.2 ml when RVE is used (or 1.0 ml when large volume is required) of Surrogate Standard Spiking Solution (see Section 7.2.2) to each sample/QC sample and proceed to Section 10.1.20. If you are performing □□□□□□ extraction add 0.2 ml when RVE is used (or 1.0 ml when large volume is required) of Surrogate Standard Spiking Solution (see Section 7.2.2) to each sample/QC sample and proceed to Section 10.1.11.

10.1.10.1. If extracting QC samples (MS, MSD, LCS or LCSD), add 0.2 ml when RVE is used (or 1.0 ml when large volume is required of the Spiking (see Section 7.2.1.1) to the appropriate separatory funnel. Note: When spiking the samples, make sure to get all bubbles out of the syringe. In addition, hold the syringe just above the level of the liquid when adding the spike. Do not touch the tip of the syringe to the liquid or the side of the separatory funnel.

10.1.11. Add concentrated sulfuric acid to each sample to adjust the pH to <2. (Usually you only need to add 1ml using small disposable Pasteur pipette). Note: pH adjustments must be documented in the extraction log.

10.1.12. Shake each sample for a short time and check pH using pH paper. The pH must be 2 or less. If the pH has not been lowered sufficiently, add more acid.

10.1.13. Add 15 ml of Methylene Chloride when RVE is used (or 60 ml when large volume is required) to each sample bottle.

10.1.14. Swirl the bottle and add 15 ml of Methylene Chloride when RVE is used (or 60 ml when large volume is required) to its corresponding separatory funnel.

10.1.15. After making sure the funnels are properly secured, start the rotators. Stop the rotator and vent the funnels after about 10 seconds. Resume rotating for 2 minutes.

10.1.16. Stop the rotation and allow the sample to settle.

10.1.17. Drain the bottom (organic) layer from the separatory funnel into the funnel/Erlenmeyer apparatus.

10.1.18. Repeat steps 10.1.13 through 10.1.17 twice, adding the Methylene Chloride directly to separatory, rather than rinsing the sample container as in 10.1.15.

10.1.18.1. If an emulsion forms during extraction, rinse a centrifuge tube well with methylene chloride and then drain the lower layer from the separatory funnel into the tube. Centrifuge for 3 to 5 minutes at 2000 rpm. Make sure the levels (and weights) of the samples directly opposite one another in the centrifuge are approximately equal. After the centrifuging process is finished, there will be two layers: water on top and the methylene chloride on the bottom. A 1 ml disposable pipette should be used to transfer the methylene chloride (bottom) layer from the centrifuge tube to the appropriate Erlenmeyer flask. With this method, care must be taken not to transfer the water (top) layer. The top layer that remains is poured back into the separatory funnel with the rest of the original sample.

10.1.19. If you are preparing the sample for acid extractables analysis only, you are finished and you can now discard the remaining liquid in each separatory funnel and proceed to Section 10.1.27. If you are required to extract base/neutrals, proceed with Section 10.1.20.

10.1.20. Adjust the pH of the sample to >11 by adding 10N sodium hydroxide (NaOH) to the sample in each separatory funnel. NOTE: pH adjustment must be documented in the extraction logbook.

10.1.21. Shake each separatory funnel for a short time and check pH. It should be basic, >pH 11. If the pH is not as high as it should be, add more 10N sodium hydroxide (NaOH).

10.1.22. Add 15 ml of Methylene Chloride when RVE is used (or 60 ml when large volume is required)

10.1.23. After making sure the funnels are properly secured, start the rotators. Stop the rotator and vent the funnels after about 10 seconds. Resume rotating for 2 minutes.

10.1.24. Stop the rotation and allow the sample to settle.

10.1.25. Drain the bottom (organic) layer from the separatory funnel into the funnel/Erlenmeyer apparatus.

10.1.26. Repeat steps 10.1.22 through 10.1.25 twice.

10.1.26.1. If an emulsion forms during extraction, rinse a centrifuge tube well with methylene chloride and then drain the lower layer from the separatory funnel into the tube. Centrifuge for 3 to 5 minutes at 2000 rpm. Make sure the levels (and weights) of the samples directly opposite one another in the centrifuge are approximately equal. After the centrifuging process is finished, there will be two layers: water on top and the methylene chloride on the bottom. A 1 ml disposable pipette should be used to transfer the methylene chloride (bottom) layer from the centrifuge tube to the appropriate Erlenmeyer flask. With this method, care must be taken not to transfer the water (top) layer. The top layer that remains is poured back into the separatory funnel with the rest of the original sample.

10.1.27. Pour the entire methylene chloride extract from the Erlenmeyer flask into a KD concentration tube apparatus (pre-rinsed three times with acetone). Rinse the Erlenmeyer flask from which the sample came twice with methylene chloride and pour both rinsates into the KD apparatus.

10.1.28. Attach Snyder column (pre-rinsed three times with acetone) to the top of the KD apparatus.

10.1.29. For RVE extractions blow the extract directly down to a final volume of 2-ml on the N-Evap. Remove the Snyder column from the top of the KD flask. Remove the blue taper clamp from the ground glass joint and dry the exterior with a Kimwipe. Transfer the concentrator tube with the 5ml extract to the N-Evap and “blow down” the extract until the volume is 2.0 ml.

10.1.30. For large volume (1000 ml) extractions concentrate the extract to approximately 5-ml in Steam Bath Remove the Snyder column from the top of the KD flask. Remove the blue taper clamp from the ground glass joint and dry the exterior with a Kimwipe. Transfer the concentrator tube with the 5ml extract to the N-Evap and “blow down” the extract until the volume is 1.0 ml. Bring the volume of each extract up to 2ml with methylene chloride

10.1.31. Split each extract into two (2) -1ml aliquots and transfer each aliquot to a separate 2ml amber screw cap vial with Teflon liner. Label one vial as 'SIM" and the second vial as 'Total'. Transfer custody of the vials to the Semivolatile GC/MS laboratory for analysis (see TestAmerica Edison analytical SOPs No. ED-MSS-002 (SW846 8270C*)* or ED-MSS-009 (SW846 8270D).

10.2. Required Documentation:

The organic prep technician is responsible for completing the following items.

10.2.1. The Standards Prep Logbook or TALS Reagent Database must be completed in full with the required information whenever standards are logged and/or prepared.

10.2.2. Each time an extraction is performed, the applicable TALS data record must be completed and reviewed the Organic Prep Supervisor or designee. Lot numbers of all reagents and solvents used or added to samples during preparation must be documented in the database.

10.2.3. Each sample extracted must be included in a batch and be recorded in the TALS database.

10.2.4. Following the extraction procedure, the technician must complete all TALS data fields pertaining to the samples extracted.

11.0. Calculations/Data Reduction

n/a

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For demonstration of capability procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, [illegible], for the laboratory's training program.

13.0. Pollution Control

13.1 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."

14.0. Waste Management

14.1 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 TestAmerica Edison SOPs Nos. ED-SPM-007 ([illegible]) and ED-SPM-008 ([illegible]) The following waste streams are produced when this method is carried out:

- Extractions Waste water. This material is created when 50% Acetone and 50% Methylene Chloride are added to 1 liter of sample water. The water is shaken with the solvent. The solvent is collected with the compounds of interest and the water is discarded into the Extractions Waste Water drum. This drum is removed to the walk-in hood in the waste room. A ½ inch PVC pipe is inserted into the bung hole of the drum and air is passed through the solution over night. The solution is then transferred into the first drum of the neutralization system and neutralized to a pH of 6 – 9. This solution is discharged into the municipal sewer system.

- Mixed Solvent Waste. This material is collected from rinsing and other processes into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.
  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

- Waste sodium sulfate. This material is collected from various methods which require the removal of water from solvent which carries the analyte (s) of interest. The solvent is passed through the sodium sulfate and the sodium sulfate plus the water is disposed of. The sodium sulfate is collected in buckets inside the hoods. The material is air dried and disposed of in the municipal waste dumpster.

- Waste sulfuric acid. This material is generated from clean up of PCB extracts for sulfur compounds. The acid is collected in satellite accumulation in the hood. The container is removed to the waste room for neutralization with 50 % sodium hydroxide (Siedler Chemical SC-1824-03), water and sodium bicarbonate (Siedler Chemical SC-0219-25). Ice is used to control temperature in the plastic drums of the neutralization system. When neutralization is complete (pH 6 -9) the material is transferred to the municipal sewer system.

15.0. References / Cross-References

15.1 United States Environmental Protection Agency, “Method SW3510C, Separatory Funnel Extraction”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.2 TestAmerica Edison SOP No. ED-MSS-002, [illegible], current revision.

15.3 TestAmerica Edison SOP No. ED-MSS-009, [illegible], current revision

15.4 TestAmerica Edison Document No. ED-QA-LQM, [illegible], current revision.

15.5 TestAmerica Environmental Health and Safety Manual, CW-E-M-001.

15.6 TestAmerica Corporate Quality SOP No. CA-Q-S-001 ([illegible]), current revision.

15.7 TestAmerica Edison SOP No. ED-GEN-023 ([illegible]), current revision.

15.8 TestAmerica Edison SOP No. ED-GEN-022, [illegible] current revision

15.9 TestAmerica Edison SOPs Nos. ED-SPM-007 ([illegible])

15.10 TestAmerica Edison SOP No. ED-SPM-008 ([illegible])

16.0. Method Modifications:

N/A

17.0. Attachments

N/A

18.0. Revision History

Revision 12, effective 03/26/2018

- Updated throughout to clarify that the lab's standard procdure is to use Reduced Volume Extraction (~250 ml initial volume) with an option to use large volume extraction (~1000 ml initial volume).

Revision 10, effective 11/29/2016:

- Sections 7.2.1 and 7.2.1.1 : updated current sources of standards.
- Section 7.2.1.1: added standards storage information as note at end of section.
- Section 8.0: added option for 250 ml amber sample containers (LVI option)

Revision 9, effective 11/21/2014:

- Section 7.2: updated current sources of all standards.
- Throughout document as required: added option for preparation of leachates by LVI.
- Section 10.1.29 through 10.1.31: clarified the concentration techniques for both full volume and LVI extracts.

Revision 8, effective 11/28/2012

- Throughout document: updated references to Lab Quality Manual section numbers.
- Added references as necessary throughout to TestAmerica Edison SOP No. ED-MSS-009 ([illegible] D, current revision.
- Section 2.2 added describing option for analysis of lower initial volume for subsequent analysis using large volume injection (LVI) technique.
- Section 6.2: added 500 ml separatory funnel.
- Sections 7.2.1 and 7.2.1.1: added '5 Compound BNA Custom Mix' and 'Aromatic Amine Custom Mix' to list of standards and prep instructions table.
- Section 10.1 (Sample Prep for Aqueous Samples): revised throughout to include option for extraction of reduced aqueous volume (250ml) for subsequent analysis by LVI technique. Added note as preface to Section 10.1 alerting analyst to the two available extraction volume options (1000ml and 250ml).

Revision 7, effective 12/6/10

- Section 3: revised to reference new location for definitions

- Sections 7.2.1 and 7.2.1.1: Added 4 compounds and additional details to the description of standard preparation of the Spiking Standard.
- Section 7.2.4: added option to document standards preparation within TALS rather than a laboratory notebook.
- Section 10.3: revised to include TALS as the main repository for raw data associated with sample prep.

Revision 6, effective November, 2008

- Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
- Revised title to include 'Leachates'.
- Section 1.3: Added reference to Quality Assurance Manual for method modifications.
- Section 3: revised to reference new location for definitions.
- Section 5: Revised to include most up to date corporate health and safety references and information.
- Section 7: added details of the solvent testing and approval program.
- Section 7.2.1: Added additional details to the description of standards and the preparation of the Spiking Standards and Surrogate Standards. Removed references to the Internal Standard which is now added by the analytical group and is discussed in TestAmerica Edison SOP No. ED-MSS-002, [illegible], current revision.
- Section 9: Quality Control: added additional details to the discussion of the various QC sample types
- Section 10: Revised and clarified to reflect current procedures. Removed reference to internal standard addition (now completed by analytical group).
- Section 11: Removed reference to Organic Calculation SOP.
- Section 12: updated and revised the MDL requirements to reflect text in the current revision of the TestAmerica Edison Laboratory Quality Manual (LQM).
- Section 15: References: Expanded to include more specific SOP references
- Section 16: Added Section 16 (Method Modifications).
- Section 18: Added this Revision History section

Title: Extraction of Pesticides and PCBs in Water by Separatory Funnel using SW846 Method 3510C

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Approvals (Signature/Date):

06/27/19		06/27/19	
Mamadou Leye Organic Prep Department Manager	Date	Dan Helfrich Health & Safety Manager	Date
06/27/19		06/27/19	
Carl Armbruster Quality Assurance Manager	Date	Mark Acierno Laboratory Director	Date

06/27/19
Sylvanus Klusey — Date
Operations Manager - Organics

Facility Distribution No. ELECTRONIC **Distributed To: FACILITY INTRANET**

1.0 Scope and Application

1.1. **Analytes, Matrix(s), and Reporting Limits:** SW846 Method 3510C describes a procedure for isolating organic compounds from aqueous samples, including concentration techniques suitable for preparing the extract for GC and/or GC/MS analysis. This SOP is specifically applicable to the isolation and concentration of water-soluble and slightly water-soluble Pesticides and PCBs using Method 3510C in preparation for analysis GC methods SW846 8081B, 8082A and GC/MS method EPA 680.

1.2. For a complete discussion of analytes and reporting limits (RLs) refer to the SOP for the applicable analytical method:.

- TestAmerica Edison SOP No. ED-GCS-016, *Analysis of Organochlorine Pesticides by SW846 Method 8081B,* current revision.
- TestAmerica Edison SOP No. ED-GCS-017, *Analysis of Polychlorinated Biphenyls by SW846 Method 8082A,* current revision.
- TestAmerica Edison SOP No. ED-MSS-010, *Determination of PCBs in Water and Soil/Sediment by GC/MS, EPA Method 680*, current revision.

1.3. On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work Request*) and 19 (*Test Methods and Method Validation*) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1 A measured volume of aqueous sample is spiked with surrogates and serially extracted with methylene chloride using a separatory funnel. The extract is dried by passing it through activated sodium sulfate, concentrated using a nitrogen blowdown technique and exchanged into hexane prior to analysis by Methods 8081B, 8082A or 680. Cleanup of extracts may be required prior to analysis.

2.2 The preferred option for preparing aqueous samples for analysis by SW8081B/8082A includes use of a reduced initial volume (~250ml) for analysis by a large volume injection (LVI). A technique for extraction of a larger volume (1000 ml) is also described.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

Phthalate esters contaminate many products commonly found in the laboratory. Plastics, in particular, must be avoided, because phthalates are often used as

plasticizers and are easily extracted from plastic material. Phthalate contamination may result at any time if consistent quality control is not practiced.

Method interference may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts or elevated baselines in gas chromatograms. All these materials must be routinely demonstrated to be free from interference under the conditions of the analysis, by analyzing method and reagent blanks.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should not be used.

The use of separatory funnels to extract aqueous samples with methylene chloride creates excessive pressure very rapidly. 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 goggle when it is performed.

The use of Kevlar gloves is required for the assembly/disassembly of ground glass joints in addition to those tasks that present the potential risk for injury.

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 MSDS 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 MSDS for each material before using it for the first time or when there are major changes to the MSDS.

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.
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.
1 – Always add acid to water to prevent violent reactions (if applicable).			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Equipment

- Steam Bath - Fisher Scientific 66738 or equivalent.
- Muffle Furnace - Thermolyne Type 6000 or equivalent.
- N-Evap - Meyer Analytical Evaporator Model No. 112 or equivalent
- Separatory Funnel Rotator, APR Machine or equivalent
- Centrifuge, Varifuge F; Hereaus Sepatech

6.2. Supplies

- 400 ml Clear Glass Jar
- 500 ml Separatory Funnel, AMK Glass SFC-0095 or equivalent
- 100 mm o. d. glass funnels, Fisher or equivalent
- 10 ml jacketed, graduated Concentrator Tubes, AMK Glass KD-0018 or equivalent
- 19/22 Ground Glass Stoppers

- 50 ul Gastight Syringe, Hamilton 80900 or equivalent
- 100 ul Gastight Syringe, Hamilton 81000 or equivalent
- 150 ml Centrifuge Tube
- 100 ml Graduated Cylinder
- Pasteur 5¾" Disposable Pipettes, Fisher 13-678-20B or equivalent
- Glass Wool
- Desiccators
- Standard Taper Clamps (Size 19, blue)
- Wax Pencil
- 1 Liter Graduated Cylinder
- Marking Tags
- pH paper

7.0 Reagents and Standards

7.1 Reagents

Note: Each lot of Methylene Chloride and Acetone, is screened for contaminants before being used for analysis as detailed in TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent Acid ot Testin and Approval*) and TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testin and Approval*).

- Methylene Chloride - JT Baker Ultra-Resi 9254-03 or equivalent
- Sodium Sulfate Crystals – Mallinckrodt MA8024-06 or equivalent
- Acetone, J. T. Baker Ultra-Resi 9264-03 or equivalent
- Hexane, Pesticide Grade, Baxter 217-4or equivalent
- Organic free reagent water - Reagent water is defined as a water in which an interference is not observed at the MDL of each parameter of interest (ASTM Specification D1193, Type ii). At TestAmerica Edison this water is generated by the Barnstead/Thermolyne Water System (Model # D11991 Serial # 1191020210415).

7.1.1 Reagent preparation

7.1.1.1 Anhydrous sodium sulfate crystals (Mallinckrodt MA8024-06 or equivalent) must be baked in the muffle furnace for four hours at 400^{o}C and serially rinsed with methylene chloride prior to use.

7.2 Standards

7.2.1 Standards are purchased as concentrated solutions (see Section 7.2.2). Most stock solutions are diluted (in volumetric glassware) to working concentration using acetone as the diluent as described in Section 7.3.

7.2.2 **Standard mixes and sources:** Table 1 lists the standard concentrate mix sources. Table 2 details the individual components of these mixes.

Table 1a:
Pesticide/PCB Standard Mixes and Sources (8081/8082)*

Standard Name ("Lab Name")	Concentration in ug/ml (each component)	Source - Catalog #
Pesticide Surrogate Spike Mix	200	RESTEK-32000
Aroclor Spike Mix (1660 Aroclor)	1000	RESTEK-32039
Organochlorine Pesticides Mix ("Pest Spike")	2000	RESTEK-32415
Chlordane (technical) ("Technical Chlordane Spike")	5000	RESTEK-32072
Toxaphene	5000	RESTEK-32071

*May be substituted with equivalent standards from alternate sources.

Table 1b:
PCB Homologue Standard Mixes and Sources (EPA 680)*

Standard Name ("Lab Name")	Concentration in ug/ml (each component)	Source - Catalog #
Retention Time Calibration Standard Mixture	Varies	Ultra-CB682-MN
Concentration Calibration Standard Mix	Varies	Ultra-CB681-MN
$^{13}C_{12}$-Decachlorobiphenyl Surrogate	40	Cambridge-EC1410-3
Lindane13C6	100	Cambridge-CL1282-S

Table 2a:
Components of Pesticide/PCB Standard Mixes (8081/8082)

Parameter	Catalog Nos.	Lab Standard Name	Conc (ug/ml)
Decachlorobiphenyl (DCB)	Pesticide Surrogate Spike Mix	RESTEK-32000	200
2,4,5,6-Tetrachloro-m-xylene (TCMX)	Pesticide Surrogate Spike Mix	RESTEK-32000	200
Aroclor 1016	Aroclor Spike Mix	RESTEK-32039	1000
Aroclor 1260	Aroclor Spike Mix	RESTEK-32039	1000
Aldrin	Organochlorine Pesticides Mix	RESTEK-32415	2000
alpha-BHC	Organochlorine Pesticides Mix	RESTEK-32415	2000
beta-BHC	Organochlorine Pesticides Mix	RESTEK-32415	2000
Lindane	Organochlorine Pesticides Mix	RESTEK-32415	2000
g-BHC	Organochlorine Pesticides Mix	RESTEK-32415	2000
DDD	Organochlorine Pesticides Mix	RESTEK-32415	2000
4,4'-DDE	Organochlorine Pesticides Mix	RESTEK-32415	2000

Table 2a: Components of Pesticide/PCB Standard Mixes (8081/8082)			
Parameter	**Catalog Nos.**	**Lab Standard Name**	**Conc (ug/ml)**
4,4'-DDT	Organochlorine Pesticides Mix	RESTEK-32415	2000
Dieldrin	Organochlorine Pesticides Mix	RESTEK-32415	2000
alpha-Endosulfan	Organochlorine Pesticides Mix	RESTEK-32415	2000
beta-Endosulfan	Organochlorine Pesticides Mix	RESTEK-32415	2000
Endosulfan Sulfate	Organochlorine Pesticides Mix	RESTEK-32415	2000
Endrin	Organochlorine Pesticides Mix	RESTEK-32415	2000
Endrin aldehylde	Organochlorine Pesticides Mix	RESTEK-32415	2000
Endrin ketone	Organochlorine Pesticides Mix	RESTEK-32415	2000
Heptachlor	Organochlorine Pesticides Mix	RESTEK-32415	2000
Hetptachlor epoxide	Organochlorine Pesticides Mix	RESTEK-32415	2000
Methoxychlor	Organochlorine Pesticides Mix	RESTEK-32415	2000
Chlordane (technical)	Technical Chlordane Spike	RESTEK-32072	5000
Toxaphene	Toxaphene Spike	RESTEK-32071	5000

Table 2b: Components of PCB Homologue Standard Mixes (680)			
Parameter	**Catalog Nos.**	**Lab Standard Name**	**Conc (ug/ml)**
PCB-104	Ultra-CB682-MN	Retention Time Calibration Standard Mix	100
PCB-208	Ultra-CB682-MN	Retention Time Calibration Standard Mix	200
PCB-77	Ultra-CB682-MN	Retention Time Calibration Standard Mix	100
Total Nonachlorobiphenyls	Ultra-CB682-MN	Retention Time Calibration Standard Mix	200
DCB Decachlorobiphenyl	Ultra-CB681-MN	Concentration Calibration Standard Mix	250
Total Dichlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	50
Total Heptachlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	150
Total Hexachlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	100
Total Monochlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	50
Total Octachlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	150
Total Pentachlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	100
Total Tetrachlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	100
Total Trichlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	50
Decachlorobiphenyl-13C12	Cambridge-EC1410-3	$^{13}C_{12}$-Decachlorobiphenyl Surrogate	40
Lindane13C6	Cambridge-CL1282-S	Lindane13C6	100

7. 3. Standards Preparation

7.3.1. All standard stock solutions are diluted to the working concentrations with hexane or acetone (as indicated) using Class A volumetric glassware. **Note:** septa on all surrogate and spike vials are to be replaced immediately after use. Additionally, all surrogate and spike vials are to be returned to the standards refrigerator immediately after use.

7.3.2. **Pesticide Spiking Standard (LCS/MS/MSD): For the reduced volume extraction option:** prepare a 4 ug/ml spiking solution by diluting 10 ml of the 20ug/ml standard prepared above to a final volume of 50ml in acetone. For spiking instructions refer to Section 10.

7.3.3. **8082 PCB Spiking Standard (LCS/MS/MSD):**. **For the reduced volume extraction option:** prepare a 20 ug/ml spiking solution by diluting 10 ml of the 100 ug/ml stock standard prepared above to a final volume of 50ml in acetone. For spiking instructions refer to Section 10.

7.3.4. **680 PCB Homologue Spiking Standard (LCS/MS/MSD):** The EPA Method 680 PCB homologue spiking standard is prepared by diluting 1.0 ml of the Retention Time Calibration Standard Mix (Ultra-CB682-MN) and 1.0 ml of the Concentration Calibration Standard Mix (Ultra-CB681-MN) to 100mL of hexane using volumetric glassware. For spiking instructions refer to Section 10.

7.3.5. **Technical Chlordane Spiking Standard (LCS/MS/MSD):** The technical Chlordane spiking solution is prepared at a final concentration of 100 ug/ml by diluting 0.2 ml of the 5000 ug/ml Technical Chlordane Spike solution (RESTEK-32072, see Tables 1 and 2) to a 10 ml final volume using acetone. For spiking instructions refer to Section 10.

7.3.6. **Toxaphene Spiking Standard (LCS/MS/MSD):** A Toxaphene spiking solution is prepared at a final concentration of 100 ug/ml by diluting 0.2 ml of the 5000 ug/ml Toxaphene Spiking Standard (RESTEK-32071, see Tables 1 and 2) to a 10 ml final volume with acetone. For spiking instructions refer to Section 10.

7.3.7. **Pesticide Surrogate Spiking Standard: For the reduced volume extraction option:** prepare a 2 ug/ml spiking solution by diluting 10 ml of the 10 ug/ml stock standard described above to a final volume of 50ml in acetone. For spiking instructions refer to Section 10.

7.3.8. **680 PCB Homologue Surrogate Spiking Standard:** The EPA Method 680 surrogate spiking standard is prepared by diluting 3.125 ml of Decachlorobiphenyl-13C12 Surrogate (Cambridge-EC1410-3) and 1.0 ml of Lindane13C6 (Cambridge-CL1282-S) to 50.0 ml hexane using volumetric glassware. For spiking instructions refer to Section 10.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water	Amber Glass	250 ml (8 oz)	Cool 4 ± 2°C	7 days to extraction; 40 days to analysis	USEPA SW846
Water	Amber glass	1000 ml	Cool 4 ± 2°C	7 days to extraction; 40 days to analysis	USEPA Method 680

8.1. Extracts must be stored under refrigeration (Cool 4 ± 2°C) in the dark and analyzed within 40 days of extraction.

9.0 Quality Control

9.1 Sample QC - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample used for MS/MSD is randomly selected by the organic prep lab, unless specifically requested by a client.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into TALS (LIMS).

9.1.1. Method Blanks are extracted with each sample batch on each day that samples are extracted. The analytical results for the method blank must fall below the reporting limit for each compound of interest. If a target compound is detected in the blank at a concentration higher than the reporting limit, first the extract is reanalyzed for confirmation. If results are still outside of limits the entire batch of samples extracted with the affected blank must be re-extracted and reanalyzed.

9.1.2. Laboratory Control Sample (LCS): A Laboratory Control Sample (LCS) or blank spike must be extracted and analyzed for with each batch of 20 environmental samples. The LCS data is used to ensure that the analytical system is in control. It is also used to assess performance if the MS/MSD recoveries fall outside of established limits. The recoveries of the LCS must fall within lab generated acceptance criteria. If the spiked sample recovery results fall outside the laboratory generated limits (refer to the current active TALS

method limit group database), the LCS recovery is evaluated. If LCS recovery is within limits the poor sample recovery results are attributed to matrix interference. If the LCS recovery results are outside QC limits, first the extract is reanalyzed and if it is still outside the limits the entire QC batch must be re-extracted and reanalyzed.

9.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD): A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. MS/MSD recoveries are evaluated against lab generated limits (refer to refer to the current active TALS method limit group database). If the MS/MSD recovery limits fall outside of lab limits the LCS recovery is evaluated and corrective action is taken as described in the applicable analytical SOP (see Section 15.0, References).

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with surrogate standard solution containing DCB and TCMS (see Section 7.3.6). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (refer to refer to the current active TALS method limit group database). If the surrogate recovery limits are outside of acceptance limits the sample extract is reanalyzed to confirm. If the recoveries are still outside of limits the sample must be re-extracted and reanalyzed or the data flagged as “estimated concentration”.

10.0. Procedure

10.1. Rinse a 500 ml separatory funnels twice each with a 1:1 methylene chloride/acetone mix and once with methylene chloride. Drain the solvents before adding sample to the funnels.

10.2. Place a small amount of glass wool into a 100-mm funnel and fill with pre-baked sodium sulfate crystals. Rinse three times with methylene chloride. Also rinse the outside of the funnel stem three times with methylene chloride, as it is likely to come into contact with the extract. Allow time for all of the rinsate to drain out of the funnel into a waste container.

10.3. Write the lab sample id number for each sample on the separatory funnels using a red wax pencil.

10.4. Make up hangtags for each sample as follows and hang on the corresponding

PCBs	PST
Sample Number: xxx	Sample Number: xxx
Fraction-Matrix:xxx	Fraction-Matrix:xxx
Date of Extraction:xx/xx/xx	Date of Extraction:xx/xx/xx

10.5. Place a 100 ml funnel containing rinsed sodium sulfate crystals onto each flasks

10.6. Mark the fluid level on each sample bottle with a black magic marker.

10.7. Rinse out a 1000ml graduated cylinder two to three times with organic free reagent water (for TCLP/SPLP leachates or reduced volume extraction use a 250 ml graduated cylinder). Obtain 1000ml of organic free reagent water (100 ml for TCLP/SPLP leachates or 250ml for reduced volume extraction) from the Millipore filtering apparatus for each of the method blank and the LCS.

10.8. Measure the initial pH of each sample with wide range pH paper and record the observed value in TALS prep batch as 'Received/initial pH'. If the initial pH of the sample is not between 5-9 adjust the pH to within 5-9 using 1:1(v/v) sulfuric acid or 10N Sodium hydroxide, as appropriate. Record the final adjusted pH value in the TALS batch comment section. Lesser strengths of acid or base solution may be employed, provided that they do not results in a significant change (<1%) in the volume of sample.

10.9. Pour each sample (including the method blank and LCS water) into its corresponding separatory funnel.

10.10. Rinse the spiking syringes 8 to 10 times with Acetone.

10.11. **For methods 8081/8081**: Add 50 ul of Pesticide Surrogate Spike Mix (see Section 7.2) to each sample and QC sample. When spiking the samples, make sure to get all bubbles out of the syringe. In addition, hold the syringe just above the level of the liquid when adding the spike. Don't touch the tip of the syringe to the liquid or the side of the separatory funnel

10.12. **For method 680**: add 1.0 ml of the PCB Homologue Surrogate Spiking Standard (see Section 7.3.8) to each sample and QC sample. When spiking the samples, make sure to get all bubbles out of the syringe. In addition, hold the syringe just above the level of the liquid when adding the spike. Don't touch the tip of the syringe to the liquid or the side of the separatory funnel

10.13. Depending upon the fraction and requested target analytes (Refer to Section 7.3 for information of spiking solutions).

- **PCBs by 8082**: add 50 ul of the 20ug/ml PCB Spike solution (see Section 7.3.3) to each LCS and designated MS/MSD
- **Pesticides (total) and TCLP/SPLP Pesticides by 8081**: 50 ul of the 4 ug/ml Pest Spike solution (see Section 7.3.2) to each LCS and designated MS/MSD
- **Technical Chlordane by 8081:** 20 ul of the 100 ug/ml Technical Chlordane Spike mix (see Section 7.3.5) to each LCS and designated MS/MSD.
- **Toxaphene by 8081:** 20 ul of the 100 ug/ml Toxaphene Spike mix (see Section 7.3.6) to each LCS and designated MS/MSD.

- **PCB Homologues by 680:** 1ml of the 680 PCB Homologue Spiking Standard (see Section 7.3.4) to each LSC and designated MS/MSD).

When spiking the samples, make sure to get all bubbles out of the syringe. In addition, hold the syringe just above the level of the liquid when adding the spike. Don't touch the tip of the syringe to the liquid or the side of the separatory funnel. Add 15 ml of methylene chloride for the reduced volume extraction option.

10.14. Add 15 ml of methylene chloride to each sample bottle. Swirl the solvent in the bottle and add the 15 ml for of methylene chloride to its corresponding separatory funnel.

10.15. Secure the separatory funnels to the rotators. Start the rotators.

10.16. Stop the rotator and vent the funnels after about 10 seconds. Resume rotating for 2 minutes.

10.17. After the rotator stops let the sample settle.

10.18. Drain bottom layer (organic layer) of the sample into the funnel/Concentrator apparatus.

10.19. Repeat steps 10.12 through 10.16 twice, now adding the methylene chloride directly to separatory funnel, rather than rinsing the sample container as in 10.12.

10.20. If an emulsion forms during the extraction, rinse a centrifuge tube well with methylene chloride and then drain the lower layer from the large separatory funnel into the tube. Centrifuge for 3 to 5 minutes at 2000 rpm. Make sure the levels (and weights) of the samples directly opposite one another in the centrifuge are approximately equal. After the centrifuging process is finished, there will be two layers: water on top and the methylene chloride with the desired sample extract on the bottom. A 1-ml disposable pipette should be used to transfer the bottom layer from the centrifuge tube to the appropriate concentration tube. With this method, care must be taken not to transfer any of the top layer. The top layer that remains is poured back into the separatory funnel with the rest of the original sample.

10.21. Find the empty sample bottles previously marked with a black marker with tap water up to the black line. Pour this into the graduated cylinder used for measuring sample volumes. Record each sample volume into the TALs extraction batch database.

10.22. Rinse funnel/sodium sulfate attached to concentration tube, twice with about 20ml of methylene chloride.

10.23. Sample can be concentrated between 30°C-40°C in the N-Vap to 2 ml final volume in the methylene chloride

10.24. Add about 10ml of Hexane to extract to exchange

10.25. Concentrate the hexane extract to 1 ml on the steam bath (temperature set between 30°C-40°C) using the N-EVAP apparatus.

10.26. The extract is now ready for the appropriate clean up.

10.27. If an emulsion forms during extraction, rinse a centrifuge tube well with Methylene Chloride and then drain the lower layer from the large separatory funnel into the tube. Centrifuge for 3 to 5 minutes at 2000 rpm. Make sure the levels (and weights) of the samples directly opposite one another in the centrifuge are approximately equal. After the centrifuging process is finished, there will be two layers: water on top and the Methylene Chloride with the desired sample extract on the bottom. A 1-ml disposable pipette should be used to transfer the bottom layer from the centrifuge tube to the appropriate concentration tube. With this method, care must be taken not to transfer any of the top layer. The top layer that remains is poured back into the 2000-ml separatory funnel with the rest of the original sample.

10.28. Required Documentation:

The organic prep technician is responsible for completing the following items.

10.28.1. The TALS reagent database must be completed in full with all required information whenever standards are logged and/or prepared.

10.28.2. All required data types (sample volumes, reagent and standard volumes, reagent and standard lots, spike witness, etc…) must be entered into the TALS batch database each time an extraction is performed. The department manager is responsible for ensuring that the data is reviewed for completeness and accuracy.

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11.0. Calculations / Data Reduction

None

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 20 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a

calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to the current revision of TestAmerica Edison SOP No. ED-GEN-022, □□□□□□□□, for the laboratory's training program.

13.0. Pollution Control

13.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

13.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage

14.0. Waste Management

14.1. The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. 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. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.2. The following waste streams are generated as a result of this analysis:

- Extractions Waste water. This material is created when 50% Acetone and 50% Methylene Chloride are added to 1 liter of sample water. The water is shaken with the solvent. The solvent is collected with the compounds of interest and the water is discarded into the Extractions Waste Water drum. This drum is removed to the walk-in hood in the waste room. A ½

inch PVC pipe is inserted into the bung hole of the drum and air is passed through the solution over night. The solution is then transferred into the first drum of the neutralization system and neutralized to a pH of 6 – 9. This solution is discharged into the municipal sewer system.

- Mixed Solvent Waste. This material is collected from rinsing and other processes into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.
  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

- Waste sodium sulfate. This material is collected from various methods which require the removal of water from solvent which carries the analyte (s) of interest. The solvent is passed through the sodium sulfate and the sodium sulfate plus the water is disposed of. The sodium sulfate is collected in buckets inside the hoods. The material is air dried and disposed of in the municipal waste dumpster.

- Waste sulfuric acid. This material is generated from clean up of PCB extracts for sulfur compounds. The acid is collected in satellite accumulation in the hood. The container is removed to the waste room for neutralization with 50 % sodium hydroxide (Siedler Chemical SC-1824-03), water and sodium bicarbonate (Siedler Chemical SC-0219-25). Ice is used to control temperature in the plastic drums of the neutralization system. When neutralization is complete (pH 6 -9) the material is transferred to the municipal sewer system.

15.0. References / Cross-References

15.1. United States Environmental Protection Agency, “Method 3510C: Separatory Funnel Liquid-Liquid Extraction”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.2. [illegible] Physical and Chemical Methods Branch, Environmental Monitoring and Support Laboratory, Office of Research and Development, USEPA, Cincinnati, OH, November 1985.

15.3. TestAmerica Edison SOP No. ED-GCS-016, [illegible] current revision.

15.4. TestAmerica Edison SOP No. ED-GCS-017, [illegible] current revision.

15.5. TestAmerica Edison SOP No. ED-MSS-010, [illegible], current revision.

15.6. TestAmerica Edison Document No. ED-QA-LQM, [illegible], current revision.

15.7. TestAmerica Corporate Document No. CW-E-M-001, [illegible] current revision.

15.8. TestAmerica Corporate Quality SOP No. CA-Q-S-001, [illegible], current revision.

15.9. TestAmerica Edison SOP No. ED-GEN-023, [illegible], current revision.

15.10. TestAmerica Edison SOP No. ED-GEN-013, [illegible], most current revision

15.11. TestAmerica Edison SOP No. ED-GEN-022, [illegible] current revision.

16.0. Method Modifications:

NONE

17.0. Attachments

NONE

18.0. Revision History

- Revison 13, dated Jun 27, 2019:
    - Sections 1 and 15 (and throughout document as applicable): Deleted references to obsolete methods SW8082 and SW8081A as well as references to associated SOPs.
    - Section 2.2: revised wording to clarify that the RVE/LVI technique is the standard procedure.
    - Throughout document: removed references to equipment no longer used in the procedure: Synder columns, Kuderna-Danish concentrators, Erlenmyer flasks.

- Revision 12, dated May 16, 2017:
    - Updated throughout to include aqueous preps for EPA method 680.
- Revision 11, dated Dec 13, 2016:
    - Section 6.2: added pH paper to list of supplies.
    - Section 10.8: added instruction for measuring, adjusting and documenting sample pH values.
- Revison 10, dated Jun 15, 2015:
    - Section 2.1 and throughout document: Revised standard initial volume for Reduced Volume Extraction (RVE) to 250 ml.
    - Section 7.2: Tables 1 and 2 updated with new Restek standards.
    - Sections 7.3: updated spiking standards preparation instructions as necessitated by switch to Restek standards.
    - Section 8.0: updated minimum volume to 250ml.
- Revison 9, dated May 3, 2013:
    - Section 7.2.2, Table 1: corrected concentration of the Chlordane (technical) standard solution to 1000 ug/ml. Deleted 'TCLP Pest Mix 1' and 'TCLP Pest Mix 2'. Added Toxaphene (Supleco 48103-U).
    - Section 7.2.2, Table 2: corrected concentration of the Chlordane (technical) standard solution to 1000 ug/ml. Deleted all TCLP Pest Spike standards. Added Toxaphene (Supleco 48103-U).
    - Deleted Section 7.3.4, TCLP Pesticide Spiking Standard (LSC/MS/MSD). Remaining sections renumbered accordingly.
    - Section 10.11. Deleted last bullet (TCLP Pesticides). Added '…and TCLP/SPLP Pesticides' to the second bullet (Pesticides). Added '(or 20 ul of the 100ug/ml mix for reduced volume extraction)' to the Toxaphene bullet.
- Revision 8, dated February 26, 2013:
    - Throughout document: updated Lab Quality Manual section number references as required.
    - Section 1.2: added references to all applicable analytical SOPs.
    - Section 2.0: added Section 2.1 which describes option for Reduced Volume Extraction.
    - Section 6.2: added a 500 ml separatory funnel.
    - Section 7.0: updated throughout to include additional standards preps for reduced volume extraction.
    - Section 8.0: updated to include smaller sample container for the reduced volume extraction option.
    - Section 10.0: updated to include spiking and concentration instructions for the reduced volume extraction option.

- Revision 7, dated October 31, 2011

    - Section 7.0: added 'Reagent Water' and details concerning it's preparation to the list of reagents.

- Revision 6, dated October 30, 2009

    - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
    - Section 1.1 Added reference to Quality Assurance Manual for method modifications.
    - Section 1.1: Expanded to include references to applicable analytical SOPs.
    - Section 3: revised to reference new location for definitions.
    - Section 5: Revised to include most up to date corporate health and safety references and information.
    - Section 7.2.2: Added tables detailing components found in the various spiking standards mixes.
    - Section 7.3: Updated the instructions for preparation of spiking standards.
    - Section 7.3: Added tables with spiking standards prep details.
    - Section 8: Updated with additional details including a table outlining containers, preservation and holding times.
    - Section 9.1: Expanded QC sample preparation details.
    - References: Expanded to include more specific SOP references
    - Section 18: Added this Revision History section
    - Throughout document: added references to TestAmerica LIMS (TALS).
    - Throughout document: deleted references to organophosphorus pesticides as the Edison lab no longer analyzes for these.

Title: Procedure for the Microwave Extraction of Solids, SW846 3546

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Sylvanus Klusey Organic Operations Manager	3/12/18 Date	Dan Helfrich Health & Safety Manager	3/12/18 Date
Carl Armbruster Quality Assurance Manager	3/12/18 Date	Mark Acierno Laboratory Director	3/12/18 Date

1.0 Scope and Application

The procedure uses microwave energy to produce elevated temperature and pressure conditions (i.e., 100° -115°C and 50 - 175 psi) in a closed vessel containing the sample (soil, sediment and sludge) and organic solvent(s) to achieve analyte recoveries equivalent to those from Automated Soxhlet extraction (Method 3541), using less solvent and taking significantly less time than the Automated Soxhlet procedure.

This method is applicable to the extraction of semi-volatile petroleum products in soil, sediment and sludge which may then be analyzed by a variety of chromatographic procedures. The method may also be applicable for the extraction of additional target analytes, provided that the analyst demonstrates adequate performance for the intended application as further described in TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

This method only is applicable to solid samples with small particle sizes. If practical the sample may be air dried and ground to a fine powder prior to extraction.

1.1 **Analytes, Matrix(s), and Reporting Limits:** This standard operating procedure details the procedures used by TestAmerica Edison for the microwave extraction of soils, sediments, sludges, and waste solids for semivolatile organic compounds by SW846 Method 3546 for subsequent analysis of:

- Diesel Range Organics (C10-C28 & C8-C44) by SW846 Methods 8015B, 8015C and 8015D (see TestAmerica Edison SOP Nos. ED-GCS-009 and ED-GCS-020);
- TPH by NJDEP-OQA-QAM-025 (see TestAmerica Edison SOP No. ED-GCS-011);
- Organochlorine Pesticides and PCBs by SW846 8081A/B and 8082/A (see TestAmerica Edison SOPs No.ED-GCS-003, ED-GCS-004, ED-GCS-016 and ED-GCS-017)
- Semivolatile Organic Compounds by Gas Chromatography/ Mass Spectrometry (GC/MS) by SW846 8270 and 8270D (TestAmerica Edison SOPs ED-MSS-002 and ED-MSS-009)
- PCBs by GC/MS using EPA Method 680 (TestAmerica Edison SOP ED-MSS-010)

1.2 The microwave prep for NJDEP Extractable Petroleum Hydrocarbons (EPH) (10/08), current revision, is addressed separately in TestAmerica Edison SOP ED-GCS-012 (current revision).

1.3 For a complete discussion of analytes and reporting limits (RLs) please refer to the applicable TestAmerica Edison analytical SOPs referenced in Section 1.1 and in References (Section 14.0).

1.4 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (□□□□□ □□□□ □□□□ □□□□□) and 19 (□□□□□ □□□□□□□ □□□□□ □□□□□□□ □□□□□□□) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

A representative soil, clay, sediment, sludge, solid or waste sample is weighed and placed into a 75mL Teflon vessel and extracted using an appropriate solvent in a microwave extractor. The extraction vessel containing the sample and solvent system is heated to the extraction temperature and extracted for 10 minutes. The mixture is allowed to cool. The vessel is opened and the contents are filtered. The solid material is rinsed and the various solvent fractions are combined. The extract is then dried, concentrated using a nitrogen blow down device, and as necessary, solvent exchanged for use in clean up, fractionation, or determinative methods.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 Solvents, reagents, glassware, and other sample processing hardware may contribute to interferences with sample analysis. All materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing a method blanks.

4.2 Phthalate esters are commonly extracted from laboratory items. Plastics in particular, must be avoided because phthalates are easily extracted from these materials. The presence of these contaminants in extracts is problematic because it will give false positive results for the methods that list phthalates as target compounds or it will interfere with the target compounds of other methods that are co-extracted with them.

4.3 All glassware used must be scrupulously cleaned before use in trace analysis. Refer to TestAmerica Edison SOP No. ED-GEN-013 (□ □□□□ □□□□ □□□□□), current revision.

4.4 Use caution if sodium sulfate is used for this method. Salts are known to super heat when used in microwave. Typically samples are analyzed with the addition of 5.0 grams sodium sulfate.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should not be used. During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard.

The use of the microwave to extract samples creates excessive pressure and temperature within the digestion vessels very rapidly. Cooling the sample after digestion is extremely important.

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 MSDS 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 MSDS for each material before using it for the first time or when there are major changes to the MSDS.

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.
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.
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.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Zymark Turbovap II concentrator
- Electronic Balance, capable of weighing to 0.01g.
- N-Evap Analytical Evaporator
- Fisher 8 Position Steam Bath
- MARS 5 microwave oven with temperature sensor (CEM Corp) capable of sensing

temperature within 2.5°C and adjusting microwave field output within 2 sec of sensing.

- MARS 40 position carousel (CEM Corp)
- Visiprep DL column holder

6.2 Supplies

- 10, 50 and 200 mL Concentrator tubes.
- Vials, - 1ml, 4ml, and 10ml with PTFE lined snapcaps.
- Glass syringes, 25ul, 50ul, 100ul, 500ul, 1000ul
- Pastuer pipets
- Glass Buchner funnels (stainless steel funnels may be substituted) – Fisher Scientific.
- 500 ml Kuderna-Danish (KD) flask
- Stainless steal spatulas
- Glass wool- Contaminant-free (silane treated or oven baked at 400^{O}C)
- 75mL Teflon Express vessels with stopper and cap (CEM Corp.)
- Glass fiber filter paper - #1 185mm (Whatman)
- Weigh boats
- Tongue depressors, wood

7.0 Reagents and Standards

Note: Each solvent lot is screened for contaminants before being used for analysis as detailed in TestAmerica Corporate Quality SOP No. CA-Q-S-001 ([illegible]) and TestAmerica Edison SOP No. ED-GEN-023 ([illegible]).

7.1. Reagents

7.1.1 Organic free reagent water - Reagent water is defined as a water in which an interference is not observed at the MDL of each parameter of interest (ASTM Specification D1193, Type ii). At TestAmerica Edison this water is generated by the Barnstead/Thermolyne Water System (Model # D11991 Serial # 1191020210415).

7.1.2 Methylene Chloride, JT Baker Resi-analyzed, P/N 9254-03 (or equivalent)

7.1.3 Hexane – JT Baker Ultra-Resi 73513-42-5 or equivalent

7.1.4 Acetone, J.T. Baker Ultra- ResiAnalyzed, Catalog No. 9264-03 (or equivalent)

7.1.5 Nitrogen, high-purity

7.1.6 Construction Sand (Home Depot or Lowes), free from organic compounds. (Baked in the muffle furnace for four hours at 400^{O}C and stored in a dessicator prior to use.)

7.1.7 Sodium Sulfate Anhydrous Powder, Mallinckrodt MA8020-06 or equivalent (Must be baked in the muffle furnace for four hours at 400^{O}C and stored in a dessicator prior to use.)

7.2. Standards

7.2.1 Stock target analyte standard spiking solutions are purchased as prepared solutions (see table below). Stock solutions are diluted (in volumetric glassware) to a working concentration using methylene chloride ($MeCl_2$) hexane or Acetone as the diluent as indicated below below. Stock standards of similar quality from other suppliers may be substituted as required.

NOTE: Second sources (from separate lots) are used for quanitation standards and spiking.

Stock Standards (Methods 8015B,8015C and 8015D DRO & NJDEP OQA-QAM-025)				
Standard Mix	**Concentration**	**Source**	**Catalog #**	**Used in method(s):**
TPH Mix 3	1000 ppm	Sigma Aldrich	861394U	*NJDEP OQA-QAM-025
o-Terphenyl	10000 ppm	Sigma Aldrich	47580U	*SW846 8015B, 8015C &8015D DRO *NJDEP OQA-QAM-025
Chlorobenzene	10000 ppm	Sigma Aldrich	4 6860U	*NJ DEP OQA-QAM-025
Diesel Fuel #2	50000 ppm	Restek	31259	*SW846 8015B, 8015C &8015D DRO *NJDEP OQA-QAM-025

Stock Standards - Pesticides/PCBs* (Methods SW846 8081A/B and 8082/A)				
Standard Mix	**Concentration**	**Source**	**Catalog #**	**Used in method(s):**
Pesticide Surrogate Spike Mix ("Pest/PCB Surrogate")	10 ug/ml	Supelco	861275	SW846 8081/8082
Aroclor Spike Mix ("PCB Spike")	100 ug/ml	Supelco	861274	SW846 8082
SS CLP Organochlorine Pesticides Mix ("Pest Spike")	2000 ug/ml	Supelco	4S7426-U	SW846 8081
Chlordane (technical) ("Technical Chlordane Spike")	1000 ug/ml	Supelco	48065-U	SW846 8081
Toxaphene	1000 ug/ml	Supelco	48065-U	SW846 8081

*May be substituted with equivalent standards from alternate sources.

Components of Pesticide/PCB Standard Mixes (Methods SW846 8081A/B and 8082/A)			
Parameter	**Catalog Nos.**	**Lab Standard Name**	**Concentration (ug/ml)**
Decachlorobiphenyl (DCB)	Supelco – 4S8913	Pest/PCB Surrogate	10.0
2,4,5,6-Tetrachloro-m-xylene (TCMX)	Supelco – 4S8913	Pest/PCB Surrogate	10.0
Aroclor 1016	Supelco - 861274	PCB Spike	100

Components of Pesticide/PCB Standard Mixes (Methods SW846 8081A/B and 8082/A)			
Parameter	**Catalog Nos.**	**Lab Standard Name**	**Concentration (ug/ml)**
Aroclor 1260	Supelco - 861274	PCB Spike	100
Aldrin	Supelco - 4S7426-U	Pest Spike	2000
Alpha-BHC	Supelco - 4S7426-U	Pest Spike	2000
Alpha-Chlordane	Supelco - 4S7426-U	Pest Spike	2000
Beta-BHC	Supelco - 4S7426-U	Pest Spike	2000
Delta-BHC	Supelco - 4S7426-U	Pest Spike	2000
Dieldrin	Supelco - 4S7426-U	Pest Spike	2000
Endosulfan I (Alpha)	Supelco - 4S7426-U	Pest Spike	2000
Endosulfan II (Beta)	Supelco - 4S7426-U	Pest Spike	2000
Endosulfan Sulfate	Supelco - 4S7426-U	Pest Spike	2000
Endrin Aldehyde	Supelco - 4S7426-U	Pest Spike	2000
Endrin Ketone	Supelco - 4S7426-U	Pest Spike	2000
Endrin	Supelco - 4S7426-U	Pest Spike	2000
Gamma-BHC (Lindane)	Supelco - 4S7426-U	Pest Spike	2000
Gamma-Chlordane	Supelco - 4S7426-U	Pest Spike	2000
Heptachlor	Supelco - 4S7426-U	Pest Spike	2000
Heptachlor Epoxide (Isomer B)	Supelco - 4S7426-U	Pest Spike	2000
Methoxychlor	Supelco - 4S7426-U	Pest Spike	2000
4,4'-DDD	Supelco - 4S7426-U	Pest Spike	2000
4,4'DDE	Supelco - 4S7426-U	Pest Spike	2000
4,4'-DDT	Supelco - 4S7426-U	Pest Spike	2000
Chlordane (technical)	Supleco – 48065-U	Technical Chlordane Spike	1000
Toxaphene	Supelco 48103	Toxaphene Spike	1000

Stock Standards – BNAs (Method 8270x)			
Standard Mix	**Concentration (ug/ml)**	**Lab Vendor**	**Catalog #**
8270 List 1/ Std#1 MegaMix	500/1000/2000	RESTEK	567672
8270 List 1/ Std#5 N-Nitrosodiphenylamine	2000	RESTEK	567676
8270 List 1/ Std#2 Amines	2000	RESTEK	567673
8270 List 1/ Std#3 Benzoic Acid	2000	RESTEK	567674
Custom SVO Mix	2000	SPEX	SVO-TANJ-16-5
Bisphenol A	1000	SPEX	S-509-MC
8270 List 1/ Std#8	2000	RESTEK	568724
8270 Surrogate Standard	5000	RESTEK	567685
Aromatic Amines Custom Mix	2000	Supelco	21467482

Stock Standards – PCB Homologues (Method 680)			
Standard Mix	**Concentration (ug/ml)**	**Lab Vendor**	**Catalog #**
Retention Time Calibration Standard Mixture	Varies by component (100 to 200)	Ultra	CB-682MN
Concentration Calibration Standard Mix	Varies by component (50 to 250)	Ultra	CB-681MN
13C12-Decachlorobiphenyl Surrogate	40	Cambridge	EC-1410-3
Lindane13C6	100	Cambridge	CLM-1282-S

Components of PCB Homologue Mixes (Methods 680)			
Parameter	**Catalog Nos.**	**Lab Standard Name**	**Concentration (ug/ml)**
PCB-104	Ultra CB-682MN	Retention Time Calibration Standard Mixture	100
PCB-208	Ultra CB-682MN	Retention Time Calibration Standard Mixture	200
PCB-77	Ultra CB-682MN	Retention Time Calibration Standard Mixture	100
Total Nonachlorobiphenyls	Ultra CB-682MN	Retention Time Calibration Standard Mixture	200
DCB Decachlorobiphenyl	Ultra CB-681MN	Concentration Calibration Standard Mix	250
Total Dichlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	50
Total Heptachlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	150
Total Hexachlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	100
Total Monochlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	50
Total Octachlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	150
Total Pentachlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	100
Total Tetrachlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	100
Total Trichlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	50

7.2.2 Spiking Standards Prep for GC FID (DRO/QAM): The table below provides instruction in preparation of the various working spiking standards for use in this preparation method using the applicable standards listed in 7.2.1. Use volumetric glassware. Dilute to the volume marker with the indicated solvent.

Working Spiking Standards Preparation					
Standard Name	**Vendor/ Cat #**	**Initial Conc. (ug/ml)**	**Vol. of Standard (ml)**	**Vol. of solvent mls (solvent)**	**Final Conc. (ug/ml)**
DRO Spike/ QAM-025 LCS Spike Diesel Fuel #2	Restek/31259	50000	4	100 ($MeCl_2$)	2000
DRO Surrogate -o-terphenyl	Supelco/47580U	10000	1.0	500 (Acetone)	20
QAM-025 MS/MSD Spike TPH Mix 3	Sigma/861394-U	1000	1.47	10 ($MeCl_2$)	147
QAM-025 Surrogate -Chlorobenzene -o-terphenyl	Supelco/46860-U Supelco/47580-U	10000 10000	1.0 1.0	500 500	20 20

7.2.3 Spiking Standards Prep for GC ECD(8081/8082):

7.2.3.1 All standard stock solutions are diluted to the working concentrations with hexane or acetone (as indicated) using Class A volumetric glassware. **Note:** septa on all surrogate and spike vials are to be replaced immediately after use. Additionally, all surrogate and spike vials are to be returned to the standards refrigerator immediately after use.

7.2.3.2 Pesticide Spiking Standard (LCS/MS/MSD): The Pesticide Mix BS/MS/MSD solution with 20 single component pesticides is prepared at a final concentration of 20 ug/ml by diluting 500 ul (0.50 ml) of the 2000 ppm Pest Spike stock solution (Supelco 4S7426-U, see tables in Section 7.2.1) to a 50 ml final volume with acetone. For spiking instructions refer to Section 10.

7.2.3.3 PCB Spiking Standard (LCS/MS/MSD): The PCB spiking solution (Supelco – 861274, see Tables 1 and 2) is a custom mix received from the vendor at its working concentration of 100 ug/ml in Acetone. For spiking instructions refer to Section 10.

7.2.3.4 Technical Chlordane Spiking Standard (LCS/MS/MSD): A technical Chlordane spiking solution is prepared at a final concentration of 100 ug/ml by diluting 1 ml of the 1000 ug/ml Technical Chlordane Spike solution (Supelco 48065-U, see tables in Section 7.2.1) to a 10 ml final volume using acetone. For spiking instructions refer to Section 10.

7.2.3.5 Toxaphene Spiking Standard (LCS/MS/MSD): A Toxaphene spiking solution is prepared at a final concentration of 100 ug/ml by diluting 1 ml of the 1000 ug/ml Toxaphene Spike solution (Supelco 48103, see tables in

Section 7.2.1) to a final volume 10 mL.using acetone. For spiking instructions refer to Section 10.

7.2.3.6 Pesticide/PCB Surrogate Spiking Standard: The Pesticide/PCB surrogate spiking solution is a concentrated 10 ml solution of DCB/TCMX at a concentration of 10 ug/ml. (Supelco Catalog # 861275, see tables in Section 7.2.1). The concentrate is used without further dilution prior to the spiking of samples.

7.2.4 Spiking Standard for BNA: For use in spiking Soils samples prepared for BNA analysis by SW846 Method 8270. Prepare the spiking solution for MS/MSD/Blank Spike (LCS) as detailed in the following table using the applicable standards listed in 7.2.1. Use volumetric glassware. Dilute to the volume marker with methanol

Standard Name	Concentration	Volume of Standard added to final volume of 200ml (solvent)	Final Concentration
8270 List 1/ Std#1 MegaMix	500/1000/2000pmm	20 ml (methanol)	50/100/200 ppm
8270 List 1/ Std#5 N-Nitrosodiphenylamine	2000ppm	10 ml (methanol)	100 ppm
8270 List 1/ Std#2 Amines	2000ppm	10 ml (methanol)	100 ppm
8270 List 1/ Std#3 Benzoic Acid	2000ppm	10 ml (methanol)	100 ppm
Custom SVO Mix	2000ppm	10 ml (methanol)	100 ppm
Bisphenol A	1000ppm	10ml (methanol)	50ppm
8270 List 1/ Std#8	2000ppm	20ul (methanol) 100ul(methanol)	Used neat for LVI Used neat for non-LVI
Aromatic Amines Custom Mix**	2000ppm	10 ml (methylene chloride)	100 ppm

*** As needed based on clients requirement.*

7.2.4.1 BNA Surrogate Standard Spiking Solution: For use in spiking all blanks, samples and associated QC prior to extraction. Prepare as detailed in the following table using the applicable standards listed in 7.2.1. Use volumetric glassware. Dilute to the volume marker with methanol.

Standard Name	Concentration	Volume of Standard added to final volume of 1000 ml methanol	Final Concentration
8270 Surrogate Standard	5000 ppm	20 ml	100 m

7.2.5 Spiking Standard for PCB Homologues (EPA 680): For use in spiking LCS and MS/MSD prior to extraction. Prepare by adding 1.0ml

of the Retention Time Calibration Standard and 1.0ml of the Concentration Calibration Standard Mix to hexane in volumetric glassware and dilute to final volume of 100ml. 1.0mL of this solution is spiked into LCS/MS/MSD prior to extraction.

7.2.5.1 PCB Homologue Surrogate Standard Spiking Solution (EPA 680): (2.0ug/mL and 2.5ug/mL) – Add 3.125mL of $^{13}C_{12}$-Decachlorobiphenyl and 1.0mL of Lindane13C6 and dilute to a final volume of 50mL in hexane in volumetric glassware. 1.0mL of this solution is spiked into all blanks, samples and QC items prior to extraction.

7.2.6 The preparation of all standards must be documented in a standard preparation logbook or in the TALS reagent module. Information such as standard supplier, lot number, original concentration, and a description of how standard was prepared are required along with a laboratory lot number, analyst's initials, date prepared and verification signature. Standards must be made every 6 months or sooner if signs of degradation appear.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Soils	Glass	4 oz	Cool 4 ± 2°C	14 Days	a) NJDEP-OQA-QAM-025; b)SW846 Method 8000

Samples are collected in amber jars with Teflon lined caps.
[1]All sample extracts must be analyzed within forty (40) days of extraction.

9.0 Quality Control

9.1. Sample QC – The following quality control samples are prepared with each batch of samples.

QC Analysis	Frequency	Acceptance Criteria	Corrective Action
Laboratory Control Sample (LCS/LCSD)[1]	1 in 20 or fewer samples	See applicable analytical SOPs	See applicable analytical SOPs for control criteria
Method Blank (MB)	1 in 20 or fewer samples	See applicable analytical SOPs	See applicable analytical SOPs for control criteria

QC Analysis	Frequency	Acceptance Criteria	Corrective Action
MS\MSD[2] (Client specified)	1 in 20 or fewer samples	See applicable analytical SOPs	See applicable analytical SOPs for control criteria
Surrogates[3]	All Samples including QC	See applicable analytical SOPs	See applicable analytical SOPs for control criteria

[1] LCS Duplicate (LCSD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract except for NJDEP EPH 10/09 where LCS/LCSD is required.
[2] The sample selections for MS/MSD are randomly selected, unless specifically requested by a client. For NJDEP EPH 10/09, only MS is required (MSD for NJDEP EPH 10/09 is by client request only).
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into LIMS.

9.2. Instrument QC

N/A

10.0 Procedure

10.1. Sample Preparation

10.1.1 Decant and discard any water layer on a sediment sample. Discard any foreign objects such as rocks, leaves and sticks. Mix sample thoroughly with wooden tongue depressor prior to subsampling

10.1.2 The initial amount and treatment of soil samples is method dependent:

- For QAM-025, DRO (8015): 15 grams of soil is weighed into a 75mL Teflon microwave vessel (the weight is recorded to two significant figures). Where practical samples should be air dried and ground prior to extraction. Drying should be performed in a hood to avoid contamination.
- For 8270/ 8081/8082: 15 grams of soil is weighed into an aluminum weighing dish (the weight is recorded to two significant figures). 15 mls of acetone is added to the dish and the soil/acetone mixture is stirred well with a wooden tongue depressor to create a slurry. The soil/acetone slurry is transferred to a 75 ml Teflon microwave vessel. The dish is then rinsed with 15 ml of methylene chloride which is also added to the microwave vessel.
- For EPA 680: 30 grams of soil is weighed into a 75mL Teflon microwave vessel (the weight is recorded to two significant figures).

10.1.3 For laboratory blanks and control spikes, an equal portion of clean sand is prepped along with samples.

10.1.4 All samples, blanks, and spikes are fortified with an appropriate amount of the applicable method surrogates. All matrix spikes and lab

control spikes are fortified with an appropriate amount of the applicable method spiking solution. See Attachment 1 for general spiking instructions. (Refer to Section 7.2 for information on preparation of spiking solutions):

- PCBs (8082): add 50 ul of the PCB Spike solution to each LCS and designated MS/MSD;
- Pesticides (total, 8081): 100 ul of the Pest Spike solution to each LCS and designated MS/MSD;
- Technical Chlordane: 100 ul of the Technical Chlordane Spike mix to each LCS and designated MS/MDS;
- Toxaphene: 100 ul of the Toxaphene Spike mix to each LCS and designated MS/MSD;
- BNA (8270): 500ul of spike/Surrogate solution to each LCS and designated MS/MSD);
- PCB Homolgues (EPA 680): 1.0 ml each of Spiking solution (see Sec.7.2.5) and Surrogate solution (see Sec. 7.2.5.1) to each LCS and designated MS/MSD.

NOTE: When spiking the samples, it is critical to remove all bubbles from the syringe.

10.1.5 Add the following solvent to each sample vessel depending upon the fraction (**note**: for Pesticides and PCBs by methods 8081 and 8082 the solvent addition is addressed in Section 10.1.2 above)

- 30mL of methylene chloride for DRO (8015)/QAM-025;
- 30 mL of Methylene Chloride/Acetone blend (50:50) for BNA (8270)

30 mL of Hexane for PCB homologues by EPA 680

10.1.6 A stopper is placed over the opening along with the vessel cap. The vessel is then tightened using the MARS capping wrench. This capping wrench tightens the cap to the appropriate torque.

10.1.7 The sample vessels are placed into the 40-position carousel. Each position has an insulator sleeve that the vessel slides into – slide the vessel into the appropriate sleeve – and press firmly down to ensure a complete connection.

10.1.8 Place the carousel (with samples and vessels) into the microwave unit.

Start Run: (load method – using key pad on face of unit)
Press: Home

Press Select: load method
Press Select: from user directory
Press Select: 3546 Microwave – Xpress method
Press Start.

Microwave Operating Parameters				
Power Max	**Power %**	**Oven Ramp**	**Degrees Celsius**	**Hold Time**
1200W	100	10.0 minutes	110	10.0 minutes
1600W[1]	100	20.0 minutes	115	10.0 minutes

[1] For pesticide/PCB and BNA samples only

10.1.9 A 5-minute cool down will close out the run.

10.1.10 Remove vessels from carousel and allow cooling (either at room temperature or in a cold water bath).

10.1.11 When cool down is complete, remove carousel from microwave.

10.1.12 Pre-wash a funnel filled with sodium sulfate with three successive washings of 30 ml acetone. Pour the extract carefully through the sodium sulfate funnel (to remove any moisture) into the 200mL concentrator tube. Rinse the 75mL vessel with 10 mL methylene chloride three times to ensure quantitative transfer.

10.1.13 Set the water temperature in the concentrator to 35°C and adjust the pressure to around 5psi of nitrogen.

10.1.14 Place the tube into the apparatus and slowly raise the pressure to 20psi. If splashing occurs, start with a lower pressure and raise it when the solvent gets lower.

10.1.15 Concentrate to just under the 1mL mark on the nipple of the tube. Remove from the bath and transfer to an auto sampler vial with a glass Pasteur pipette..

10.1.16 Add a few drops of clean methylene chloride to the tube, rinsing the narrow part and add to the vial to adjust to 1mL or other method specific final volume. If solvent exchange to hexane is required, add approximately 30mL (mix well) and return to concentrator set at 35°C. See Attachment 2.

10.1.17 Total concentration time should be approximately 20 minutes for methylene chloride. If a sample takes a longer than usual amount of time to concentrate or if the extract becomes viscous the final volume should be adjusted to a larger volume and documented.

10.1.18 The extract is now ready for the appropriate clean up and or analysis.

10.2. Calibration

10.2.1 The balance calibration is checked daily prior to use. Refer to TestAmerica Edison SOPs ED-GEN-010, *alibration o Anal tical alances*, current revision and ED-GEN-006, *tandard eratin rocedure or re enti e aintenance and alibration rocedures or All Anal tical nstruments and Ancillar qui ment*, current revision.

11.0 Calculations / Data Reduction

N/A

12.0 Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For demonstration of capability procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, *rainin*, for the laboratory's training program.

13.0 Pollution Control

13.1. 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."

14.0 Waste Management

14.1 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 TestAmerica Edison SOPs No. ED-SPM-008 (*□aborator□ □ aste □is□osal □rocedures□current re□ision□*. The following waste streams are produced when this method is carried out:

- *□ i□ed □ol□ent □ aste*: Mixed solvent waste is collected in a small beaker inside the bench top hood. This waste is then transferred into the satellite accumulation container in the Organic Prep. Lab. on a daily basis. This material is transferred into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.

  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

- *Auto sam□ler □ials and e□□ired standards*: These vials are collected in satellite accumulation within the instrument laboratory. The vials are then placed into a 55 steel open top drum in the waste room. When the drums are full, the drum will be collected by the waste vendor for disposal. This waste is treated for incineration.

  Teris Profile Number: 50016652
  Onyx Profile WIP Number: 282493

15.0 References / Cross-References

15.1 United States Environmental Protection Agency, “Method SW3546, Microwave Extraction”, Test Methods for Evaluating Solid Wastes, SW846 Laboratory Manual, Physical/Chemical Methods, February 2007.

15.2 United States Environmental Protection Agency, “Method SW8015B, Non-Halogenated Organics, Test Methods for Evaluating Solid Wastes, SW846 Third Edition,Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.3 United States Environmental Protection Agency, Method SW8015C, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, February 2007.

15.4 United States Environmental Protection Agency, Method SW8015D, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 4, June 2003.

15.5 NJDEP Method No. OQA-QAM-025, “Quantitation of Semivolatile Petroleum Products in Water, Soil, Sediment and Sludge” (current revision)

15.6 United States Environmental Protection Agency, “Method 8081A, Organochlorine Pesticide by Gas Chromatography”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.7 United States Environmental Protection Agency, “Method 8082, Polychlorinated Biphenyls PCBs) by Gas Chromatography”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.8 United States Environmental Protection Agency, “Method 8081B, Organochlorine Pesticide by Gas Chromatography”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 2, February 2007.

15.9 United States Environmental Protection Agency, “Method 8082A, Organochlorine Pesticide by Gas Chromatography”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 1, February 2007.

15.10 United States Environmental Protection Agency, “Method SW8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.11 United States Environmental Protection Agency, “Method SW8270D, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 4, February 2007.

15.12 TestAmerica Edison SOP No. ED-GCS-008, *□re□aration and Anal□sis o□□iesel □an□e □r□anics □□ □□ □□□□□□□in □oil and □ ater □am□les b□ □□ □□□ □et□ods □□□□□ □□e□arator□ □unnel□□□□□□ □Automated □o□□let □□traction□and* □□□□□□□□ □□□□□ □□current revision.

15.13 TestAmerica Edison SOP No. ED-GCS-009, *□re□aration and Anal□sis o□□iesel □an□e □r□anics □□ □□ □□□□□□□in □oil and □ ater □am□les b□ □□ □□□ □et□ods □□□□□ □□e□ □unnel□□□□□□ □Automated □o□□let□and □□□□□ □□ □□□□□□* current revision.

15.14 TestAmerica Edison SOP No. ED-GCS-011,□□□□ □□A□A□ □□□□ □*uantitation o□□emi□olatile □etroleum □roducts in □ ater □oil □ediment and □lud□e*□current revision.

15.15 TestAmerica Edison SOP No. ED-GCS-003, *Anal□sis o□□ r□anoc□lorine □esticides b□ □as □□romato□ra□□□ □□ □□□ □et□od □□□□A*, current revision.

15.16 TestAmerica Edison SOP No. ED-GCS-004, *Anal□sis o□□ol□c□lorinated □i□□en□ls b□ □as □□romato□ra□□□ □□ □□□ □et□od □□□□*, current revision

15.17 TestAmerica Edison SOP No. ED-GCS-016, *Anal□sis o□□ r□anoc□lorine □esticides b□ □as □□romato□ra□□□ □□ □□□ □et□od □□□□□*, current revision.

15.18 TestAmerica Edison SOP No. ED-GCS-017, *Anal□sis o□□ol□c□lorinated □i□□en□ls b□ □as □□romato□ra□□□ □□ □□□ □et□od □□□□A*, current revision

□□.□□ TestAmerica Edison SOP No. ED-MSS-010, □*etermination o□□□□s in □ ater and □oil□□ediment b□ □ □ □□ □□ □□A □ et□od □□□□*current revision.

15.20 TestAmerica Edison Document No. ED-QA-LQM, □*aborator□ □ualit□ □anual*, current revision.

15.21 TestAmerica Environmental Health and Safety Manual, CW-E-M-001.

15.22 TestAmerica Corporate Quality SOP No. CA-Q-S-001, □*ol□ent □ Acid □ot □estin□□ A□□ro□al*, current revision.

15.23 TestAmerica Edison SOP No. ED-GEN-023, □*ul□ □ol□ent □estin□ and A□□ro□al*, current revision.

15.24 TestAmerica Edison SOP No. ED-GEN-013, □*lass□are □leanin□□*current revision

15.25 TestAmerica Edison SOP No. ED-GEN-022, □*rainin□□*current revision

15.26 TestAmerica Edison SOP No. ED-SPM-008, □*aborator□ □ aste □is□osal □rocedures*□current revision.

15.27 TestAmerica Edison SOP No. ED-GEN-006, □*tandard □ □eratin□ □rocedure □or □re□enti□e □aintenance and □alibration □rocedures □or All Anal□tical □nstruments and Ancillar□ □qui□ment*□current revision.

15.28 TestAmerica Edison SOP No. ED-GEN-010, □*alibration o□Anal□tical □alances*□ current revision.

15.29 TestAmerica Edison SOP No. ED-GEN-007, □*ubsam□lin□*, current revision.

15.30 Operation Manuals for the Zymark Turbo Vap II Concentrator.

15.31 Operating manual for the MARS 5 Microwave unit.

16.0 Method Modifications:

N/A

17.0 Attachments

Attachment 1: Spiking information
Attachment 2: Specific Extraction Conditions

18.0 Revision History

Rev 11, March 12, 2018

- Updated Section 10.1 to revise procedures for the microwave extraction of solids for subsequent analysis by EPA methods 8081, 8082 and 8270.
- Updated to remove all references to addition of sodium sulfate to samples prior to microwave extraction.
- Fixed section numbering and formatting errors.

Rev 10, May 16, 2017

- Updated throughout to add procedures for microwave extractions of solids for subsequent analysis by EPA Method 680 (PCBs by GC/MS).

Rev 9, Dec 15, 2016

- Updated throughout to remove procedures for microwave extraction of solids for NJ EPH method. Those procedures are fully covered in TestAmerica Edison SOP ED-GCS-012, Preparation and Analysis of Extractable Petroleum Hydrocarbons (EPH) in Solid and Water Samples using NJDEP EPH Method 10/08, August 2010 (Rev. 3): Analysis of Extractable Petroleum Hydrocarbon Compounds (EPH) in Aqueous and Soil/Sediment/Sludge Matrices (current revision)
- Added following text to Section 1.2: The microwave prep for NJDEP Extractable Petroleum Hydrocarbons (EPH) (10/08), current revision, is addressed separately in TestAmerica Edison SOP ED-GCS-012 (current revision).
- All references to NJDEP EPH methodology removed.

Rev 8, Jan 22, 2015

- Updated to include procedures for microwave extraction of solids for analysis my SW846 Methods 8270C and 8270D (Semivolatile Organic Compounds by Gas Chromatography/ Mass Spectrometry (GC/MS). Specifically
    - Added 8270 method references and SOP numbers to Sections 1.1 and 15.0
    - Added a table to Section 7.2.1 detailing the BNA standard mixes.
    - Added new Section 7.2.4 detailing the preparation of BNA spiking standards

- Added Section 7.2.4.1 detailing the preparation of BNA surrogate spiking standards.
- Added details of 8270 spiking and extraction to Section 10.0 as appropriate.
- Added BNA Spiking details to Attachments 1 and 2. COAs for BNA spiking not included in SOP but are on-file in the department.

Rev 7, June 14, 2013

- Section 1.1: updated the DRO method references to include SW8015C and SW8015D. Also update the SOP references for the DRO analytical methods.
- Section 7.2.1: updated stock standards table to include SW8015C and 8015D. Revised source for o-Terphenyl stock. Removed TPH Mix 1 from list. Removed 8015x DRO from the list of methods for which TPH Mix 3 is used. Added 8015x DRO to the list of methods for which Diesel Fuel #2 is used.
- Section 7.2.2.1: Revised DRO surrogate spike source and prep instructions. Revised DRO and QAM-025 spiking standard source and prep instructions.
- Section 15.0: References: added references for methods SW8015C and 8015D.
- Attachment 1: updated spiking information for DRO.

Rev 6, October 10, 2012

- Throughout document:: Revised LQM reference sections to reflect the most current revision
- Throughout document: updated as detailed below to include prep instructions for solid samples undergoing analysis by SW846 8081A/B and 8082/A.
- Section 1.1: added references to the 8081/8082 SOPs: TestAmerica Edison SOPs No.ED-GCS-003, ED-GCS-004, ED-GCS-016 and ED-GCS-017.
- Section 6.2: added weigh boats and wooden tongue depressors.
- Section 7.2.1: added acetone as a diluent and added tables detailing the Pestcide and PCB standards.
- Section 7.2.2: added “GCFID (DRO/EPH/QAM)” to section header.
- Section 7.2.3: added instructions for prep of Pest/PCB spiking standards.
- Section10.1.4: Added Pest/PCB spiking instructions.
- Section 10.1.5: added instructions for addition of extraction solvent (Methylene Chloride:Acetone for Pest/PCB).
- Section 10.1.8: updated table to include Microwave Parameters for Pest/PCB prep.
- Section 10.1.12: added a methylene chloride rinse.

- Section 15: added method and SOP references for SW846 8081A/8081B and SW846 8082/8082A.
- Attachments: updated to include Pest/PCB information.

Rev 5, August 26, 2011

- Section .6.2: added vendor and part number details for silica gel columns (Phenomenex Part No:8L-S012-JCH)
- Section 10.1.2: Added details for the pre-washing of sodium sulfate funnels with three successive 30 ml volumes of acetone. Added details for the rinsing of the 75 ml vessel with three successive volumes of methylene chloride.

Rev 4, April 21, 2010

- Section 7.2.1: Corrected the footnotes in the tables 'Stock Standards – Aliphatics (Method NJDEP EPH 10/08)' and 'Stock Standards-Aromatics (Method NDJEP EPH 10.08)'.
- Attachment 3: Added this attachment which consists of select certificates of analysis for Method NJDEP EPH 10/08.
- Section 7.2.2.1: Table 'Working Spiking Standards Preparation'" replaced asterisks (*) with dashes (-) in order to clarify that these are simply bullets rather than referencing footnotes.

Rev 3, April 19, 2010

- Section 7.2.2.1: added procedure for preparation of a 500 ppm Aliphatic Neat Spike Mix.
- Section 7.2.2: Spiking Standards Prep: revised the procedure for prep of the EPH Aliphatic Spike in the 'Working Spiking Standards Preparation' table as follows:
    - Replaced 'MA EPH-n-Hydrocarbons (Absolute/91488) with the 'Florida TRPH Standard (Restek/31266)'
    - Included used of the intermediate 500 Aliphatic Neat Spike Mix.

Rev 2, February 9, 2009

- Section 3.0, Definitions – updated to reflect current location of definitions (Appendix 2 of Lab Quality Manual
- Section 5.2, Primary Materials Used – added 'hexane' to table (used in the NJDEP EPH method).
- Section 7.2.1: added stock standards associated with NJDEP EPH 10/08 method.
- Section 7.2.2: Spiking Standards Prep: updated 'Working Spike Standards' table to include revised NJDEP EPH 10/09 spiking standards; revised other as required.
- Section 9.1, Sample QC: updated with revise NJDEP EPH 10/09 QC requirements.
- Section 10.1, Sample Preparation: revised initial sample weight to 15 grams.
- Section 10.1.8: added Microwave Operating Parameters for the NJDEP EPH 10/09 method.
- Section 10.2.1: changed the specifications of the silica column.

- Section 10.2.5: changed the volume of the hexane rinsate.
- Section 10.2.7: changed the volume of the methylene chloride rinsate.
- Section 10.2.11: Added text describing procedures for NJDEP EPH 10/09 Demonstration of Fractionation Capability.
- Section 14.1 and Section 15.0 : Removed reference to TestAmerica Edison SOP No. ED-SPM-007 (*□is□osal o□□am□les and Associated □aborator□ □ aste□current re□ision□* (SOP is now retired).
- Section 15.0: updated references to NJDEP EPH method as well as TestAmerica Edison SOP for NJDEP EPH analysis.
- Section 17.0, Attachments: corrected descriptions of attachments.

Rev 1, June 29, 2009

- Section 1.0, Scope and Application – corrected typo in the temperature range 100 – 115^{o}C
- Section 5.2, Primary Materials Used – removed 'sodium hydroxide' and 'sulfuric acid' from table as they are not used in this method.
- Section 6.1, Instrumentation – added Visiprep DL column holder to the instrumentation list
- Section 6.2, Supplies – added 5 g silica gel column (20 ml capacity)
- Sections 7.2.1, 7.2.2, Attachment 1 and Attachment 2: corrected a typo in the abbreviation for Methylene Chloride ($MeCl_2$).
- Section 10.1.1.2 - sentence correction to show actual process and typo error correction. Phrase 'may also' changed to 'shall'.
- Section 10.1.12 – replaced the word 'salt' with 'Sodium Sulfate' to show actual reagent being used.
- Section 10.1.17 – typo error correction to '20 minutes'
- Section 10.2.5 – Added the following text to the end of the section: "The amount on hexane will need to be adjusted with every lot of silica columns. The Hexane amount should never exceed 20 mls."
- Corrected carbon ranges for the reference DRO SOPs throughout the document.

Rev 0, March 27, 2009: New

Attachment 1

Spiking Information – Microwave Extraction

Method	Surrogate Amount (all samples and QC)	Spike Amount (LCS/MS/ MSD)	Final volume	Initial Solvent	Final Solvent
DRO (8015)	1 mL	1 mL	1 mL	$MeCl_2$	$MeCl_2$
QAM-025	1 mL	1 mL	1 mL	$MeCl_2$	$MeCl_2$
Pest/PCB (8081/8082)	0.05 mL	0.1/0.05 mL	10 mL	MeCl2/Acetone (50:50)	Hexane Exchange from MeCl2/Acetone
BNA (8270)	500uL	500uL	1 mL	MeCl2/Acetone (50:50)	$MeCl_2$
PCB Homologues (EPA 680)	1.0	1.0	1 mL	Hexane	Hexane

Attachment 2

Specific Extraction Conditions

Method	Initial extraction pH	Secondary extraction pH	Final Solvent	Volume of extract required for cleanup(mL)	Final extract volume for analysis (mL)
DRO (8015)	as received	NA	$MeCL_2$	NA	1.0
QAM-025	as received	NA	$MeCL_2$	NA	1.0
Pest/PCB (8081/8082)	as received	NA	Hexane	3	10.0
BNA (8270)	as received	NA	$MeCL_2$	NA	1.0
PCBs (EPA 680)	as received	NA	Hexane	NA	1.0

Title: SW846 Method 3550C, Ultrasonic Extraction of Pesticides & PCBs in Soils

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

	08/25/20		08/25/20
Mamadou Leye Organic Prep Department Manager	Date	Dan Helfrich Health & Safety Manager	Date
	08/25/20		08/25/20
Carl Armbruster Quality Assurance Manager	Date	Mark Acierno Laboratory Director	Date

Sylvanus Klusey — 08/25/20 — Date
Operations Manager - Organics

Facility Distribution No. Electronic **Distributed To:** Facility Intranet

1.0 Scope and Application

1.1 **Analytes, Matrix(s), and Reporting Limits:** This standard operating procedure details the procedures used by Eurofins TestAmerica Edison for the extraction of soils, sediments, sludges, and waste solids for semivolatile organochlorine pesticide and PCB compounds by SW846 Method 3550C (Ultrasonic Extraction) for subsequent analysis by SW846 Methods 8081B and 8082A.

1.2 For a complete discussion of analytes and reporting limits (RLs) please refer to the current revisions of Eurofins TestAmerica Edison SOPs for the applicable determinative method: ED-GCS-016 (SW846 Method 8081B) and ED-GCS-017 (SW846 Method 8082A). Alternatively, refer to the current Eurofins TestAmerica LIMS (TALS) Method Limit Group (MLG) database for this information.

1.3 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 18 (*Review of Work Request*) and 20 (*Test Methods and Method Validation*) of Eurofins TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1. Low concentration method - A 15-g sample is mixed with anhydrous sodium sulfate to form a free-flowing powder. This is solvent extracted three times using ultrasonic extraction. The extract is separated from the sample by vacuum filtration or centrifugation. The extract is ready for cleanup and/or analysis following concentration.

2.2. Medium/high concentration method - A 2-g sample is mixed with anhydrous sodium sulfate to form a free-flowing powder. This is solvent extracted once using ultrasonic extraction. A portion of the extract is removed for cleanup and/or analysis.

2.3. Blanks and replicate analysis samples must be subjected to the same cleanup as the samples associated with them.

3.0 Definitions

For a complete list of definitions refer to Appendix 5 in the most current revision of Eurofins TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Solvents, reagents, glassware, and other sample hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks.

4.2. Phthalate esters contaminate many products commonly found in the laboratory. Plastics, in particular, must be avoided, because phthalates are often used as plasticizers and are easily extracted from plastic material. Phthalate contamination may result at any time if consistent quality control is not practiced.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP and should not be used.

Ultrasonic disrupters can produce high intensity noise and must be used in an area with adequate noise protection.

During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard.

The KD apparatus has ground glass joints which can become stuck. Technicians must use Kevlar or other cut/puncture resistant gloves when separating stuck joints.

The use of a vacuum system for extract drying is applicable to this method. 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. Ensure that the vacuum exhaust hose used is securely anchored inside of a fume hood so that solvent-vapors are not pumped into the working environment.

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 MSDS 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 MSDS for each material before using it for the first time or when there are major changes to the MSDS.

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.
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.			

6.0 Equipment and Supplies

6.1. Equipment & Instrumentation

- Sonic Disrupter-300W min., with pulsing capability
- 1/8” Microtip Horn
- Sound Enclosure, Tekmar 10-0351-0000 or equivalent
- Sound Enclosure-2 holes, Tekmar 10-0429-0000 or equivalent
- Analytical Balance
- Muffle Furnace, Thermolyne 6000 or equivalent
- Vacuum pump
- N-Evap, Organomation
- Dessicator, VWR D1420-02 or equivalent

6.2. Supplies

- 50 ul Gastight Syringe, Hamilton 80900 or equivalent
- 100 ul Gastight Syringe, Hamilton 81000 or equivalent
- 250 ul Gastight Syringe, Hamilton 81100 or equivalent
- 1ml Gastight Syringe, Hamilton 81317 or equivalent
- 10 ml Graduated Pipette, Fisher 11-393-63D or equivalent
- Marking Tags
- Glass wool
- 20 ml Scintillation Vials, Fisher or equivalent
- Disposable Pasteur Pipettes, 5 ¾ “, Fisher 13-678-20B or equivalent
- Tongue Depressors
- 400ml Griffin beaker, Fisher 02-555-29C or equivalent
- 250ml Erlenmeyer flask, AMS Glass ERL-0252 or equivalent
- 100ml graduated cylinder

- 1000 ml filtering flask, Kontes 953827-000 or equivalent
- 1000 ml filtering funnel, Kontes 953781-0050 or equivalent
- 90mm funnel support, Kontes 953826-0090 or equivalent
- 10ml jacketed concentrator tubes, AMK Glass KD-0018 or equivalent
- Lab jack, VWR S9298-1 or equivalent
- Boiling stones, Troemner 133-B or equivalent
- Glass Fiber Filter, Fisher 09-804-90C (9.0 cm) or equivalent

7.0 Reagents and Standards

7.1 Reagents

Note: Each lot of solvent is screened for contaminants before being used for analysis as detailed in Eurofins TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*).

7.1.1 Methylene Chloride - JT Baker Ultra-Resi 9254-03 or equivalent

7.1.2 Acetone, J. T. Baker, Ultra-Resi 9264-03 or equivalent

7.1.3 Hexane (High Purity Grade) - Baxter 217-4 (or equivalent)

7.1.4 Hexanes/Acetone Blend (50/50 v/v Blend of Ultra-ResiAnalyzed Hexanes and Acetone) – Cycletainer, Baker 9525-V8

7.1.5 Sodium Sulfate Anhydrous Powder, Mallinckrodt 8020-06 or equivalent (Must be baked in the muffle furnace for four hours at 400^{O}C and stored in a dessicator prior to use.)

7.1.6 Organic-free, deionized reagent water

7.2 Standards

7.2.1 Stock target analyte standard spiking solutions are purchased as prepared solutions (see table below). Stock solutions are diluted (in volumetric glassware) to a working concentration using methanol (MeOH) as the diluent as described below. Stock standards of similar quality from other suppliers may be substituted as required.

Table 1: Pesticide/PCB Standard Mixes and Sources*		
Standard Name ("Lab Name")	**Concentration in ug/ml (each component)**	**Source - Catalog #**
Pesticide Surrogate Spike Mix ("Pest/PCB Surrogate")	200	RESTEK-32000
Aroclor Spike Mix ("PCB Spike")	1000	RESTEK-32039
Organochlorine Pesticides Mix ("Pest Spike")	2000	RESTEK-32415
Chlordane (technical) ("Technical Chlordane Spike")	5000	RESTEK-32072
Toxaphene	5000	RESTEK-32071

*May be substituted with equivalent standards from alternate sources.

Table 2: Components of Pesticide/PCB Standard Mixes			
Parameter	**Catalog Nos.**	**Lab Standard Name**	**Concentration (ug/ml)**
Decachlorobiphenyl (DCB)	RESTEK-32000	Pest/PCB Surrogate	200
2,4,5,6-Tetrachloro-m-xylene (TCMX)	RESTEK-32000	Pest/PCB Surrogate	200
Aroclor 1016	RESTEK-32039	PCB Spike	1000
Aroclor 1260	RESTEK-32039	PCB Spike	1000
Aldrin	RESTEK-32415	Pest Spike	2000
Alpha-BHC	RESTEK-32415	Pest Spike	2000
Alpha-Chlordane	RESTEK-32415	Pest Spike	2000
Beta-BHC	RESTEK-32415	Pest Spike	2000
Delta-BHC	RESTEK-32415	Pest Spike	2000
Dieldrin	RESTEK-32415	Pest Spike	2000
Endosulfan I (Alpha)	RESTEK-32415	Pest Spike	2000
Endosulfan II (Beta)	RESTEK-32415	Pest Spike	2000
Endosulfan Sulfate	RESTEK-32415	Pest Spike	2000
Endrin Aldehyde	RESTEK-32415	Pest Spike	2000
Endrin Ketone	RESTEK-32415	Pest Spike	2000
Endrin	RESTEK-32415	Pest Spike	2000
Gamma-BHC (Lindane)	RESTEK-32415	Pest Spike	2000
Gamma-Chlordane	RESTEK-32415	Pest Spike	2000
Heptachlor	RESTEK-32415	Pest Spike	2000
Heptachlor Epoxide (Isomer B)	RESTEK-32415	Pest Spike	2000
Methoxychlor	RESTEK-32415	Pest Spike	2000
4,4'-DDD	RESTEK-32415	Pest Spike	2000
4,4'DDE	RESTEK-32415	Pest Spike	2000
4,4'-DDT	RESTEK-32415	Pest Spike	2000
Chlordane (technical)	RESTEK-32072	Technical Chlordane Spike	5000
Toxaphene	RESTEK-32071	Toxaphene Spike	5000

7.2.2 Standards Preparation

All standard stock solutions are diluted to the working concentrations with hexane or acetone (as indicated) using Class A volumetric glassware. **Note:** septa on all surrogate and spike vials are to be replaced immediately after use. Additionally, all surrogate and spike vials are to be returned to the standards refrigerator immediately after use.

7.2.2.1 **Pesticide Spiking Standard (LCS/MS/MSD):** The Pesticide Mix BS/MS/MSD solution with 20 single component pesticides is prepared at a final concentration of 20 ug/ml by diluting 500 ul (0.50 ml) of the 2000 ppm Pest Spike stock solution (RESTEK-32415, see Tables 1 and 2) to a 50 ml final volume with acetone. For spiking instructions refer to Section 10.

7.2.2.2 **PCB Spiking Standard (LCS/MS/MSD):** The PCB spiking solution 100ug/ml is prepared by diluting 10ml of (RESTEK-32039, see Tables 1 and 2) in to 100 ml of Acetone . For spiking instructions refer to Section 10.

7.2.2.3 **Technical Chlordane Spiking Standard (LCS/MS/MSD):** A technical Chlordane spiking solution is prepared at a final concentration of 100 ug/ml by diluting 0.2 ml of the 5000 ug/ml Technical Chlordane Spike solution (RESTEK-32072, see Tables 1 and 2) to a 10 ml final volume using acetone. For spiking instructions refer to Section 10.

7.2.2.4 **Toxaphene Spiking Standard (LCS/MS/MSD):** A Toxaphene spiking solution is prepared at a final concentration of 100 ug/ml by diluting 0.2 ml of the 5000 ug/ml (RESTEK-32071, see Tables 1 and 2) to a 10 ml final volume with acetone. For spiking instructions refer to Section 10.

7.2.2.5 **Pesticide/PCB Surrogate Spiking Standard:** The Pesticide/PCB 10ug/ml surrogate spiking solution is prepared by diluting 10 ml of 200 ug/ml (RESTEK-32000, see Tables 1 and 2) in to 200 ml of Acetone. For spiking instructions refer to Section 10.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

8.1 Sample Extract Storage. Samples extracts must be protected from light and refrigerated at 4ºC (± 2ºC) from time of extraction until analysis.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Solid	Clear Glass	8 oz.	Cool 4 ± 2°C	14 Days until extraction; analyze within 40 days of extraction	SW846

9.0 Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of samples. (For additional details on analysis and evaluation of these QC samples refer to the current revisions of Eurofins TestAmerica Edison SOPs for the applicable determinative methods: ED-GCS-016 (SW846 Method 8081B) and ED-GCS-017 (SW846 Method 8082A).

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample for MS/MSD is randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD, Method Blank)
[4] Statistical control limits are updated annually and are updated into lab reporting software.

9.1.1. **Method blanks** are extracted with every sample batch on each day that samples are extracted.

9.1.2. **Matrix Spike (MS)/Matrix Spike Duplicate (MSD):** A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. (Note: an LCS/LCSD may be substituted for the MS/MSD if insufficient client environmental sample volume is available).

9.1.3. **Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD):** A Laboratory Control Sample (LCS) (aka blank spike) must be extracted and analyzed with each batch of 20 environmental samples.

9.1.3.1 A Laboratory Control Sample Duplicate (LCSD) is extracted and analyzed only when insufficient client sample is available for preparation of an MS/MSD pair. The LCS/LSCD is evaluated in the same manner as the MS/MSD (see Section 10 for spiking instructions).

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with a two (2) component surrogate standard mix (see Section 10 for spiking instructions)

10.0. Procedure

10.1. Low Level Extraction Procedure

10.1.1. Organize one (1) 400 ml beaker and one (1) 250 ml flask for each sample/QC sample to be extracted. Rinse each piece of glassware three (3) times with acetone. Afterwards, cover each piece with foil.

10.1.2. Label both beakers and flasks with the lab sample ID number. Beakers are labeled on the side as well as on the foil top. Make up hang tags as follows and place on flasks.

Fraction: PST or PCB
Sample Number:
Matrix:
Date of Extraction:

10.1.3. Decant and discard any water layer from sediment sample. Mix each sample thoroughly. Discard any foreign objects such as sticks, leaves, and rocks. Weigh approximately 15 grams (±0.05 grams) of soil into a weigh boat and record the actual sample weight in the extraction log. (**NOTE:** Add the surrogates and the matrix spiking compounds to the sample aliquot prior to mixing the sample with Acetone). Mix the sample with approximately 10 ml of Acetone until a slurry mixture is obtained. Rinse the dish with about 5ml of acetone.

10.1.4. Weigh out 30 g of sodium sulfate into two clean beakers without any soil. These will serve as the Method Blank and the Laboratory Control Sample (LCS) respectively.

10.1.5. Rinse the syringes to be used for adding spiking solution and surrogates 8 to 10 times with acetone. Spike the sample with the appropriate surrogate/spike solutions. Add the surrogates and the matrix spiking compounds to the sample aliquot prior to mixing the sample with sodium sulfate. **NOTE:** All spiking procedures must be witnessed!

10.1.6. Mix the sample and the sodium sulfate together well. Stir and crush clumps.

10.1.6.1 **Surrogates**: 50ul of the 10 ug/ml Pesticide Surrogate Spike Mix (Section 7.2.1) is added to each sample being analyzed for pesticides and/or PCBs (see Tables 1 and 2).

10.1.6.2 **Pesticide Matrix Spikes and LCS**: 100ul of the 20ug/ml Pest Spiking Standard solution (See Section 7.2.1) is added into a total of 30 g sodium sulfate anhydrous for Laboratory Control Sample (LCS) (aka BS). Similarly, 100ul of the Pest Spiking Standard solution is added into 15 g of the sample being extracted as the Matrix Spike (MS) and into 15 g of the sample being extracted for Matrix Spike Duplicate (MSD). These QC sample types undergo the same prep steps as regular samples (Sections 10.1.1 thru 10.1.6) including addition of surrogates.

10.1.6.3 **PCB Matrix Spikes and LCS:** 50ul of the 100ug/ml PCB Spiking Standard solution (See Section 7.2.1) is added into a total of 30 g sodium sulfate anhydrous for Laboratory Control Sample (LCS). Similarly, 100ul of the PCB Spiking Standard solution is added into 15 g of the sample being extracted as the Matrix Spike (MS) and into 15 g of the sample being extracted for Matrix Spike Duplicate (MSD). These QC sample types undergo the same prep steps as regular samples (Sections 10.1.1 thru 10.1.6) including addition of surrogates.

10.1.6.4 **Chlordane Matrix Spikes and LCS:** 100ul of the 100ug/ml Technical Chlordane Spiking Standard solution (See Section 7.2.1) is added into a total of 30 g sodium sulfate anhydrous for Laboratory Control Sample (LCS). Similarly, 100ul of the Technical Chlordane Spiking Standard solution is added into 15 g of the sample being extracted as the Matrix Spike (MS) and into 15 g of the sample being extracted for Matrix Spike Duplicate (MSD). These QC sample types undergo the same prep steps as regular samples (Sections 10.1.1 thru 10.1.6) including addition of surrogates.

10.1.6.5 **Toxaphene Matrix Spikes and LCS:** 100ul of the 100ug/ml Toxaphene Spiking Standard solution (See Section 7.2.1) is added into a total of 30 g sodium sulfate anhydrous for Laboratory Control Sample (LCS). Similarly, 100ul of the Toxaphene Spiking Standard solution is added into 15 g of the sample being extracted as the Matrix Spike (MS) and into 15 g of the sample being extracted for Matrix Spike Duplicate (MSD). These QC sample types undergo the same prep steps as regular samples (Sections 10.1.1 thru 10.1.6) including addition of surrogates.

10.1.7. Prepare a 50:50 blend of hexane and acetone (alternatively use prepared 50:50 hexanes/acetone blend delivered via the Baker cycletainer delivery system. See Section 7.1).

10.1.8. Immediately after spiking and mixing with sodium sulfate add 100 ml of the hexane/acetone mixture to each sample. (Addition of solvent immediately after spiking will limit surrogate and spike evaporation).

10.1.9. Situate the beaker so that the probe of the sonicator horn is submerged approximately halfway into the solvent layer. The horn must be positioned above the soil layer. If the probe is positioned properly, you should see extreme motion within the soil layer, a type of turning over action. Sometimes the soil is very thick or hard. When this occurs, you must turn the beaker to mix its contents. For low-level sonication, a ¾" horn should be used.

10.1.10. Sonicate for a minimum of 3 minutes, with output control set at 10, mode switch on pulse, duty cycle set at 50%.

10.1.11. When the sonicator stops, decant off the solvent layer from the beaker into a rinsed 250-ml Erlenmeyer flask.

10.1.12. Again add 100 ml of the hexane/acetone mixture to each beaker containing sample.

10.1.13. Again sonicate for approximately 3 minutes, and decant the solvent into the 250-ml Erlenmeyer flask when the sonication process is finished.

10.1.14. Again add 100 ml of the hexane/acetone mixture to each beaker containing sample.

10.1.15. Sonicate for approximately 3 minutes.

10.1.16. When the sonicator stops, lower the beaker on the lab jack and rinse the soil particles from the sonicator horn into the beaker using the hexane/acetone mixture.

10.1.17. Cover the beaker with foil. Set it next to its corresponding labeled Erlenmeyer flask.

10.1.18. Wipe the sonicator horns thoroughly with a Kimwipe and rinse each with hexane/acetone mixture.

10.1.19. For each sample attach a 90 mm funnel support and to a 1000 ml filtering flask. The 90 mm funnel support has a connector to hook-up into a vacuum pump. Place a 1000 ml filtering funnel on top of the funnel support. Secure a 90 mm pinch clamp to the filtering funnel and the funnel support. Use ¼ - 3/8 " ID, 1/8" wall Tygon tubing to connect the filtering apparatus to the vacuum pump**.**

10.1.20. Rinse top portion of each filter apparatus with hexane/acetone mixture by first pouring a small amount of the solvent in. Next, thoroughly rinse the inside of the filter funnel with a rinse bottle containing hexane/acetone mixture. (During all rinsing steps the vacuum pump is on.)

10.1.21. Rinse the top portion of the filter apparatus twice with hexane/acetone mixture.

10.1.22. Turn off the vacuum pump and place a 9.0 cm Fisher brand (09-804-90 C) circular glass fiber filter on top of the funnel support with the rough side up. Replace the filtering funnel and secure with the pinch clamp. Connect the filtering apparatus to the vacuum pump and rinse with hexane/acetone mixture.

10.1.23. Detach the top portion of the filtering apparatus (filter funnel/funnel support/pinch clamp) from the filtering flask and discard the waste solvent from the flask.

10.1.24. Rinse the filter flask twice with hexane/acetone mixture.

10.1.25. Re-attach the top portion of the filtering apparatus to the filter flask and turn vacuum pump on.

10.1.26. Pour each sample extract from its Erlenmeyer flask into the top of its filtering apparatus (pre-labeled with the sample ID).

10.1.27. Pour a small amount of hexane/acetone into each sample beaker, swirl, and pour liquid/soil mixture into filtering apparatus.

10.1.28. Rinse the Erlenmeyer twice with hexane/acetone mixture, pouring the rinse material into the filtering apparatus. Rinse the filtering apparatus as in Step 10.1.20.

10.1.29. After filtration is complete, turn vacuum pump off.

10.1.30. Transfer the concentrator tube with the 5ml extract to the N-Evap (30^{o}C) and evaporate (“blow down”) the solvent volume to 1.0 mL using a gentle stream of clean dry Nitrogen.

10.1.31. If analysis is not immediately required, insert a size 19/22 stopper into the concentrator tube and put in refrigerator. Be sure to dry the joint with a Kimwipe before removing Snyder column to prevent water from dripping into the sample.

10.1.32. Proceed with the appropriate clean ups prior to analysis.

10.2. Medium/High Level Extraction Procedure

10.2.1. Weigh 2 g of sample into clean 20 ml scintillation vial

10.2.2. Label vials the vials with the lab sample number using a wax pencil or Sharpie. Make up tags as follows and place on vials.

Fraction: PST or PCB
Sample Number:
Matrix:
Date of Extraction:

NOTE: Add the surrogates and matrix spiking compounds to the sample aliquot prior to mixing the sample with acetone drying agent.

10.2.3. Rinse the syringes to be used for adding spiking solution and surrogates 8 to 10 times with acetone. Spike the sample with the appropriate surrogate/spike solutions prior to mixing with sodium sulfate. Add 2 g of sodium sulfate to each sample. **NOTE:** All spiking procedures must be witnessed!

10.2.4. Also weigh 2 g of sodium sulfate in to two clean 20-ml vials. These will serve as the method blank and Laboratory Control Sample (LCS).

10.2.5. Mix the spiked sample with surrogate and the sodium sulfate together well. Stir and crush any clumps.

10.2.5.1 **Surrogates**: 50ul of the 10 ug/ml Pesticide Surrogate Spike Mix (See Section 7.2.1) is added to each sample being analyzed for pesticides and/or PCBs (see Tables 1 and 2).

10.2.5.2 **Pesticide Matrix Spikes and LCS**: 100ul of the 20ug/ml Pest Spiking Standard solution (See Section 7.2.1) is added into a Laboratory Control Sample (LCS) (aka BS). Similarly, 100ul of the Pest Spiking Standard solution is added into 2 g of the sample being extracted as the Matrix Spike (MS) and into 2 g of the sample being extracted for Matrix Spike Duplicate (MSD). These QC sample types undergo the same prep steps as regular samples (Sections 10.2.1 thru 10.2.4) including addition of surrogates.

10.2.5.3 **PCB Matrix Spikes and LCS:** 50ul of the 100ug/ml PCB Spiking Standard solution (See Section 7.2.1) is added into a Laboratory Control Sample (LCS) (aka BS). Similarly, 100ul of the PCB Spiking Standard solution is added into 2 g of the sample being extracted as the Matrix Spike (MS) and into 2 g of the sample being extracted for Matrix Spike Duplicate (MSD). These QC sample types undergo the same prep steps as

regular samples (Sections 10.2.1 thru 10.2.4) including addition of surrogates.

10.2.5.4 **Chlordane Matrix Spikes and LCS:** 100ul of the 100ug/ml Technical Chlordane Spiking Standard solution (See Section 7.2.1) is added into a Laboratory Control Sample (LCS). Similarly, 100ul of the Technical Chlordane Spiking Standard solution is added into 2 g of the sample being extracted as the Matrix Spike (MS) and into 2 g of the sample being extracted for Matrix Spike Duplicate (MSD). These QC sample types undergo the same prep steps as regular samples (Sections 10.2.1 thru 10.2.4) including addition of surrogates.

10.2.5.5 **Toxaphene Matrix Spikes and LCS:** 100ul of the 100ug/ml Toxaphene Spiking Standard solution (See Section 7.2.1) is added into a Laboratory Control Sample (LCS). Similarly, 100ul of the Toxaphene Spiking Standard solution is added into 2 g of the sample being extracted as the Matrix Spike (MS) and into 2 g of the sample being extracted for Matrix Spike Duplicate (MSD). These QC sample types undergo the same prep steps as regular samples (Sections 10.2.1 thru 10.2.4) including addition of surrogates.

10.2.6. Add Hexane to a final volume of 10 ml.

10.2.7. Sonicate each sample for a minimum of 2 minutes at an output setting of 5, mode switch on pulse, and duty cycle at 50%.

10.2.8. Transfer an aliquot of the extract into a vial labeled with the lab sample ID. Transfer to the analytical lab or perform cleanups as appropriate.

11.0. Calculations/Data Reduction

n/a

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 20 (Test Methods and Method Validation) of Eurofins TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for

analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For demonstration of capability procedure refer to Section 20 in the most current revision of Eurofins TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to Eurofins TestAmerica SOP No. ED-GEN-022, *Training*, for the laboratory's training program.

13.0. Pollution Control

13.1 It is Eurofins 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."

14.0. Waste Management

14.1 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 Eurofins TestAmerica Edison SOPs Nos. ED-SPM-007 (*Disposal of Samples and Associated Laboratory Waste, current revision)* and ED-SPM-008 (*Laboratory Waste Disposal Procedures, current revision).* The following waste streams are produced when this method is carried out:

- Mixed Solvent Waste. This material is collected from rinsing and other processes into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.
  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

- Waste sodium sulfate. This material is collected from various methods which require the removal of water from solvent which carries the analyte (s) of interest. The solvent is passed through the sodium sulfate and the sodium sulfate plus the water is disposed of. The sodium sulfate is collected in buckets inside the hoods. The material is air dried and disposed of in the municipal waste dumpster.

15.0. References / Cross-References

15.1 United States Environmental Protection Agency, "Method SW3550C, "Ultrasonic Extraction", Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, February 2007.

15.2 Eurofins TestAmerica Edison SOP No. ED-GCS-016, *Analysis of Organochlorine Pesticides by Gas Chromatography, SW846 Method SW8081B*, current revision.

15.3 Eurofins TestAmerica Edison SOP No. ED-GCS-017, *Analysis of Polychlorinated Biphenyls by Gas Chromatography, SW846 Method SW8082A*, current revision.

15.4 Eurofins TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, current revision.

15.5 Eurofins TestAmerica Environmental Health and Safety Manual, CW-E-M-001.

15.6 Eurofins TestAmerica Corporate Quality SOP No. CA-Q-S-001, *Solvent & Acid Lot Testing & Approval*, current revision.

15.7 Eurofins TestAmerica Edison SOP No. ED-GEN-023, *Bulk Solvent Testing and Approval*, current revision.

15.8 Eurofins TestAmerica Edison SOP No. ED-GEN-022, *Training,* current revision

15.9 Eurofins TestAmerica Edison SOPs Nos. ED-SPM-007, *Disposal of Samples and Associated Laboratory Waste,* current revision.

15.10 Eurofins TestAmerica Edison SOP No. ED-SPM-008 *Laboratory Waste Disposal Procedures,* current revision

16.0. Method Modifications:

Item	Method	Modification
2.1 & 10.1.3	*SW846 3550C*	15 g is utilized for the low level extraction method (Method 3550C suggests 30g).

17.0. Attachments

N/A

18.0. Revision History

Revision, 2, 08/25/2020

- Updated throughout with Eurofins branding.
- Section 1: Updated location of 'Management Review of Work Request'' to Section 18 of Lab Quality Manual.
- Section 4: Updated location of 'Definitions' to Appendix 5 of Lab Quality Manual.

- Updated controlled document titles and control numbers throughout and in Section 7 References as needed.
- Revised Section 10 to reflect acetone slurry procedure and remove K-D procedure..

Revision, 1, 04/07/2014

- Revised throughout to reflect new source of standards (Restek).

Revision 0, 02/22/2011 NEW

Title: SW846 Method SW8081B, Analysis of Organochlorine Pesticides by Gas Chromatography

Approvals (Signature/Date):

Catalina Dalangin SVOA GC Manager	9/13/16 Date	Dan Helfrich Health & Safety Manager	9/13/16 Date
Carl Armbruster Quality Assurance Manager	9/13/16 Date	Mark Acierno Laboratory Director	9/13/16 Date

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

Method 8081B is used to determine the concentrations of various organochlorine pesticides in extracts from solid and liquid matrices, using dual fused-silica, open-tubular, capillary columns with electron capture detectors (ECD). The list of analytes and their corresponding reporting limits are as follows:

Parameter		Soil	Water	Leachate
	CAS Registry No.	Reporting Limits (ug/Kg)	Reporting Limits (ug/L)	Reporting Limits (mg/L)
Aldrin	309-00-2	6.7	0.02	---
Alpha-BHC	319-84-6	2.0	0.02	---
Beta-BHC	319-85-7	2.0	0.02	---
Delta-BHC	319-86-8	2.0	0.02	---
Gamma-BHC (Lindane)	58-89-9	6.7	0.02	0.00050
Chlordane	57-74-9	67	0.50	0.0050
4,4' –DDD	72-54-8	6.7	0.02	---
4,4' –DDE	72-55-9	6.7	0.02	---
4.4' –DDT	50-29-3	6.7	0.02	---
Dieldrin	60-57-1	2.0	0.02	---
Endosulfan I	959-98-8	6.7	0.02	---
Endosulfan II	33213-65-9	6.7	0.02	---
Endosulfan sulfate	1031-07-8	6.7	0.02	---
Endrin	72-20-8	6.7	0.02	0.00050
Endrin aldehyde	7421-93-4	6.7	0.02	---
Endrin ketone	53494-70-5	6.7	0.02	---
Heptachlor	76-44-8	6.7	0.02	0.00050
Heptachlor epoxide	1024-57-3	6.7	0.02	0.00050
Methoxychlor	72-42-5	6.7	0.02	0.00050
Toxaphene	8001-35-2	67	.50	0.0050
Gamma- Chlordane	5103-74-2	6.7	0.02	---
Alpha-Chlordane	5103-71-9	6.7	0.02	---
Mirex	2385-85-5	6.7	0.010	---

The most current MDLs and RLs for this method can be found in the active TestAmerica LIMS (TALS) SW846 8081B Method Limit Group (MLG) database.

1.2 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work Request*) and Section 19 (*Test Methods and Method Validation)* of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1. Samples undergo a preparation step prior to analysis by SW846 Method 8081B. A measured volume or weight of sample (15g for soil, 1 g for waste, 250 ml for water, and 250 ml for TCLP) is extracted using the appropriate matrix-specific sample extraction technique. (Reference the applicable Organic Sample Prep SOPs listed below). The effective final volume is usually between 5 and 20 ml in hexane.

2.1.1. Aqueous samples are extracted using SW846 Method 3510C (SOP No. ED-ORP-014: *□□t□a□tion o□□esti□ides and □□□s in □ ate□□□ □e□a□ato□□ □□nnel*).

2.1.2. Solid samples are extracted using SW846 Method 3550B: Sonication (*□□□ □o□□□□□□ □□□□□□□□□□t□a□tion o□□esti□ides□□□□s in □oil □sin□ □o□□ □e□el □□t□a□tion*) or SW846 Method 3546: Microwave (SOP No. ED-ORP-044: *□□o□ed□e □o the Mi□□o□a□e □□t□a□tion o□□olids□□ □□□ □□□□)*□

2.1.3. Organic liquids are prepared using SW846 Method 3580A (*□□□ □o□□□□ □□□□□□□□□□ aste □il□tion □o□□esti□ides and □□□s*).

2.1.4. Extract cleanup steps are employed depending on the nature of the matrix interferences. Suggested cleanups include SW846 Method 3620B (*□□□ □o□ □□□□□□□□□□□□ □lo□isil □lean□□ □o□ □esti□ide□□□□ □a□□le □□t□a□ts*) and SW846 Method 3660B (*□□□ □o□ □□□□□□□□□□□□ The □e□o□al o□ □le□ental □□l□□□□o□ □esti□ide□□□□ □a□□le □□t□a□ts*).

2.2. After cleanup, a small aliquot of the extract is injected into a gas chromatograph (GC) equipped with a capillary column. The GC is temperature programmed to separate the compounds which were recovered during the extraction step by boiling point. The effluent of the gas chromatograph is interfaced to an Electron Capture Detector (ECD) which is used to detect the compounds eluting from the GC.. Quantitation is accomplished by comparing the area response of each target analyte relative to an internal standard established through a five-point calibration (six points for second order regression). Specific calibration and quality control steps are included in the method that must be performed and must meet the specifications of SW846 Method 8081B.

2.3. For pesticide analysis a system performance check (DDT/Endrin breakdown) and a calibration verification standard must be run prior to analysis. Failure of either will generally indicate the need for injection port/column maintenance and/or recalibration.

2.4. Samples are analyzed after all the necessary checks have been performed. Samples analyzed for pesticides require an additional post analysis Quant Report to be printed and attached to the chromatographic report.

2.5. All samples are then manually reviewed. Secondary column confirmation of target compounds and quantitation are conducted by the analyst as required.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Interferences by phthalate esters introduced during sample preparation can pose a major problem in pesticide determinations.

4.1.1. Interferences from phthalate esters can be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

4.2. The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting organochlorine pesticides. Sulfur contamination should be expected with sediment samples. If sulfur is encountered, employ the sulfur removal procedures detailed in SW846 Method 3660B (□□□ □o□□□□ □□□□□*The* □e□o□al o□□le□ental □□l□□□□o□ □esti□ide□□□ □a□□le □□t□a□ts). Note that the recover of Endrin Aldehyde is adversely affected by the TBA cleanup procedure detailed in this method. Accordingly, this compound must be determined prior to sulfur cleanup.

4.3. Co-eluting chlorophenols are eliminated by using SW846 Method 3620B (□□□ □o□ □□□□ □□□□□□□□□*lo□isil □lean□□ □o□□esti□ide□□□□ □a□□le □□t□a□ts*).

4.3.1. Check Florisil prior to use to assure quantitative recovery of targeted analytes. Duplicate checks are required for each new lot or every three hundred samples whichever is more frequent.

4.3.2. Check Florisil by spiking 1ml of the Pest Std Mix A midpoint (Supelco Catalog No. 47977) and 0.5 ml of trichlorophenol (Absolute Standardsl Catalog No. 20024) onto the cartridge and concentrating to final volume of 1 ml. Inject 1 ul onto a capillary column, conducting the elution and analyzing the extract. Recovery is acceptable if all pesticides are recovered at 80 - 110% and the recovery of trichlorophenol is <5% and co-eluting interfering peaks are absent from the extract.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

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 MSDS 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 MSDS for each material before using it for the first time or when there are major changes to the MSDS.

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.
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.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation:

6.1.1. Gas Chromatograph: The system used is an HP and an Agilent Technologies (Avondale, PA) model 5890/6890 Gas Chromatograph (GC). Each GC is equipped for simultaneous quantitation and confirmation columns using two separate detector channels on dual megabore capillary columns that are suitable for the analysis of organochlorine pesticides. All operations are as automated as possible with the equipment utilized.

6.1.2. Injection system: Sample injection is accomplished by a single auto injector. The auto injector is serviced by a robot arm that shuttles samples between the sample tray and the injector turret.

6.1.2.1. The samples are injected into a split/splitless injection port equipped with electronic pressure control (EPC). The injection port is normally operated in splitless mode during injection. The EPC is operated in the ramp pressure mode.

6.1.2.2. Liners: The injection port is each fitted with replaceable, heavy-walled siltek-coated glass double gooseneck liner. The liner contains a plug of silanized glass wool approximately 1 cm in length. The glass wool is positioned in the liner between the double gooseneck. The liner is replaced on a regular maintenance schedule.

6.1.2.3. Oven and Columns: Temperature programmable gas chromatograph ovens are required, capable of integrated temperature control between 35^{O}C and 350^{O}C.

6.1.2.3.1. Two dissimilar columns are used for analysis. A Restek RtxCLPesticides, 30m x 0.53mm ID x 0.5um film thickness column is used for sample quantitation. The secondary confirmation column is a Restek RtxCLPesticides II, 30m x 0.53mm ID x 0.42um film thickness column.

6.1.2.4. Detectors: Sample detection is by electron capture. The GC is equipped with dual Electron Capture Detectors (ECD), one for each column.

6.1.2.4.1. Each detector is supplemented with make-up gas to provide sufficient detector flow for maintaining the electron plasma. This is in addition to the gas exiting the column. The make-up gas (P-5 & Nitrogen) is fed from a supply other than the injection port.

7. Reagents and Standards

7.1. Reagents

7.1.1. Gases: Hydrogen is used as the carrier and injection port purge gas. It is introduced to the GC at the injection port. P-5 and Nitrogen) is used as make-up gas. It is introduced to the GC via the make-up gas adapter at the end of the capillary column. Hydrogen is supplied via a Parker Balston H2 Generator. Nitrogen & P-5 is supplied by Air Gas

7.1.1.1. The gas streams are polished using three traps or filters before introduction to the GC The traps are as follows:

7.1.1.1.1. Hydrocarbon trap
7.1.1.1.2. H_2O trap
7.1.1.1.3. O_2 scrubber

7.1.1.2. Both the moisture trap and the Oxygen scrubber are of the indicating type. They require either replacement or reconditioning upon color change of the active agents. Refer to the instructions for the individual traps to determine if it is still active. The hydrocarbon trap is a simple activated carbon trap. With high quality gas, it should last for an extended period of time (1-yr. minimum).

7.1.2. Solvents used in the extraction and cleanup procedures include n-hexane, methylene chloride, and acetone that are exchanged to n-hexane prior to analysis.

7.1.3. Hexane is required in this procedure. All solvents must be pesticide quality or equivalent. Each lot of solvent is screened for contaminants before being used for analysis as detailed in TestAmerica Corporate Quality SOP No. CA-Q-S-001 (□ol□ent □ □□id □ot Testin□ □ □□□□o□al) and TestAmerica Edison SOP No. ED-GEN-023 (□□/□ □ol□ent Testin□ and □□□□o□al).

7.2. Standards

7.2.1. Standards are purchased as concentrated solutions (see Section 7.2.2).

NOTE: Independent sources are used for quantitation standards and spiking standards

7.2.1.1. Most stock solutions are diluted (in volumetric glassware) to working concentration using hexane as the diluent as described in Section 7.2.2.1.

7.2.2. Standard mixes and sources *.

Standard Name	Source
Organochlorine Pesticide Mix AB #3	Restek Catalog No. 32415
Organochlorine Pesticide Mix AB #3.sec	Restek Catalog No. 32415.sec (second source)
Pesticide Surrogate Mix	Restek Catalog No. 32000
Endrin/DDT	Supelco Catalog No 48282
Chlordane	Restek Catalog No. 32021
Toxaphene	Restek Catalog No. 32005
Toxaphene (different pattern)	Restek Catalog No. 32071
Mirex	Accustandard Cat. No. P-066S-10X
1-Bromo-2-nitrobenzene (internal standard)	Restek Cat. No. 32279

*Suppliers with equivalent standards may be substituted..

The components of each standard mix are as follows (note: 'sec' indicates second source):

Parameter	Supplier	Catalog No.	Concentration of Standard (ug/ml)
Aldrin	Restek	32415 & 32415.sec	2000
Alpha-BHC	Restek	32415 & 32415.sec	2000
Beta-BHC	Restek	32415 & 32415.sec	2000
Delta-BHC	Restek	32415 & 32415.sec	2000
Gamma-BHC (Lindane)	Restek	32415 & 32415.sec	2000
Alpha -Chlordane	Restek	32415 & 32415.sec	2000
Gamma -Chlordane	Restek	32415 & 32415.sec	2000
Technical Chlordane	Restek	32021	1000
4,4' –DDD	Restek	32415 & 32415.sec	2000
4,4' –DDE	Restek	32415 & 32415.sec	2000
4.4' –DDT	Restek	32415 & 32415.sec	2000
Dieldrin	Restek	32415 & 32415.sec	2000
Endosulfan I	Restek	32415 & 32415.sec	2000
Endosulfan II	Restek	32415 & 32415.sec	2000
Endosulfan sulfate	Restek	32415 & 32415.sec	2000
Endrin	Restek	32415 & 32415.sec	2000
Endrin aldehyde	Restek	32415 & 32415.sec	2000
Endrin ketone	Restek	32415 & 32415.sec	2000
Heptachlor	Restek	32415 & 32415.sec	2000
Heptachlor epoxide	Restek	32415 & 32415.sec	2000
Methoxychlor	Restek	32415 & 32415.sec	2000
Toxaphene	Restek	32005	1000
Toxaphene	Restek	32071	5000
Mirex	Accustandard	861428-U	1000
4,4-DDT	Supelco	18282	500
Endrin	Supelco	48282	500
Decachlorobiphenyl (DCB)	Restek	32000	200
Tetrachloro-m-xylene (TCmX)	Restek	32000	200
1-Bromo-2-nitrobenzene (internal standard)	Restek	32279	1000

7.2.2.1. Standards Preparation

7.2.2.1.1. Calibration Mix (Organochlorine Pesticide Mix)

The 5 point calibration standards are prepared as detailed in the following table using volumetric glassware and hexane as the diluent:

Initial Calibration Standards Prep (Organochlorine Pesticide Mix)					
Stock Std	**2.5 ppb**	**50 ppb**	**100 ppb**	**250 ppb**	**500 ppb**
Organochlorine Mix AB#3 ***(2000 ug/ml)*** *□este□ □□at □o□□□□□□)in □e□ane□Tol□ene*	Volume brought up to 200 ml hexane: □□□□ □/	Volume brought up to 200 ml hexane: □□□ □/	Volume brought up to 500 ml hexane: □□ □/	Volume brought up to 200 ml hexane: □□ □/	Volume brought up to 200 ml hexane: □□ □/
Initial Calibration Standards Prep (TCMX/DCB Surrogate Mix)					
Stock Std	**25 ppb***	**50 ppb**	**100 ppb**	**150 ppb**	**200 ppb**
Tetrachloro-m-xylene/ Decachlorobiphenyl Surrogates Mix (200 ug/ml) *□este□ □□at□□o□ □□□□□) in □□etone*	Volume brought up to 200 ml hexane: □□□□ □/	Volume brought up to 200 ml hexane: □□ □/	Volume brought up to 500 ml hexane: □□□ □/	Volume brought up to 200 ml hexane: □□□ □/	Volume brought up to 200 ml hexane: □□□ □/
Initial Calibration Standards Prep (Mirex)					
Stock Std	**2.5 ppb**	**50 ppb**	**100 ppb**	**250 ppb**	**500 ppb**
Mirex ***(1000 ug/ml)*** *□□□□standa□d □□at□□o□ □□□□□□□□□□) in Methanol*	20x dilution of 50 ppb	10x dilution of 500 ppb	5x dilution of 500 ppb	2x dilution of 500 ppb	Volume brought up to 100 ml hexane: □□ □/

Note: 20 ul of 5 ug/ml Internal Standard solution is added to all calibration standards prior to analysis.

7.2.2.2. **Pesticide Surrogate Spike Mix (10 ug/ml**) : The Pesticide 10ug/ml surrogate spiking solution is prepared by diluting 10 ml of 200 ug/ml of the Pesticide Surrogate Mix (Restek-32000) in to 200 ml of Acetone. For spiking instructions refer to the applicable prep SOP.

7.2.2.3. **Pesticide Surrogate Spike Mix (2 ug/ml) for the reduced volume extraction option:** prepare a 2 ug/ml spiking solution by diluting 10 ml of the 10 ug/ml stock standard described in 7.2.2.2 to a final volume of 50ml in acetone. For spiking instructions refer to the applicable prep SOP.

7.2.2.4. **Pesticide Internal Standard Spike Mix (5 ug/ml):** The Pesticide 5 ug/ml internal standard spike mix is prepared by

dilution 1 ml of 1000 ug/ml of the 1-Bromo-2-Nitrobenzene standard (Restek 32279) in to 200 ml of Hexane. 20 ul of this solution is added to all standards, QC samples and field sample extracts prior to analysis.

7.2.2.5. **Pesticide Spiking Standard (20 ug/ml):** The Pesticide Spiking Mix containing the single component pesticides is prepared at a final concentration of 20 ug/ml by diluting 500 ul of the Organochlorine Pesticide Mix AB#3 (Restek 32415 to a 50 ml final volume with acetone. For spiking instructions refer to the applicable prep SOP.

7.2.2.6. **Pesticide Spiking Standard (4 ug/ml) for the reduced volume extraction option**: prepare a 4 ug/ml spiking solution by diluting 10 ml of the 20ug/ml standard prepared in Section 7.2.2.4 above to a final volume of 50ml in acetone. For spiking instructions refer to the applicable prep SOP.

7.2.2.7. **System Performance Solution (Breakdown Check)** 3,3-DDT and Endrin at 0.25 ug/ml): The breakdown check is prepared by taking 250 ul of 500 ug/ml DDT/Endrin Mix (Supelco Catalog No 48282) and bringing it up to a volume of 500 ml with hexane

7.2.2.8. **Technical Chlordane Calibration Solution** (1.0 ug/ml solution w/ surrogates TCMX & DCB at 0.10 ug/ml): 100 ul of 1000 ug/ml Technical Chlordane Standard (Restek Catalog No 32021) and 50 ul of 200 ug/ml Surrogate Mix (Restek Catalog No. 32000). Dilute to 100 ml in Hexane. □□***T***□□ ICAL consists of single midpoint calibration standard for multiple responders Technical Chlordane and Toxaphene.

7.2.2.9. **Toxaphene Calibration Solution** (1.0 ug/ml solution w/surrogates TCMX & DCB at 0.1 ug/ml) : 20 ul of 5000 ug/ml Toxaphene stock (Restek Catalog No 32071) and 50 ul of 200 ug/ml Surrogate Mix (Restek Catalog No. 32000). Dilute to 100 ml in Hexane. □□***T***□□ ICAL consists of single midpoint calibration standard for multiple responders Technical Chlordane and Toxaphene.

7.2.2.10. **Initial Calibration Verification (ICV)** Preparation: follow the instructions above for midpoint standards substituting the second source standard (catalog no. suffix = sec) for the primary standards.

8. Sample Collection, Preservation, Shipment and Storage

8.1. Extracts must be stored under refrigeration in the dark and analyzed within 40 days of extraction.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Amber glass, 250 ml	250 ml	Cool 4 ± 2°C	7 days to extraction; Analyze within 40 days of extraction	SW846
Soils	Glass, 2 or 4 oz	100 g	Cool 4 ± 2°C	14 days to extraction; Analyze within 40 days of extraction	SW846

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

9. Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]
Internal Standard[1]	Every sample[3]	Response within -50% to +100% of most recent cal standard

LCS Duplicate (LCSD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into LIMS.

9.1.1. **Method Blanks** are extracted with each sample batch on each day that samples are extracted. The analytical results for the method blank must fall below the reporting limit for each compound of interest. If a target compound is detected in the blank at a concentration higher than the reporting limit, first the extract is reanalyzed for confirmation. If results are still outside of limits the entire batch of samples extracted with the affected blank must be re-extracted and reanalyzed.

9.1.2. Laboratory Control Sample (LCS): A Laboratory Control Sample (LCS) or blank spike must be extracted and analyzed for with each batch of 20 environmental samples. The recoveries of the LCS must fall within lab generated acceptance criteria (refer to the current TALS Method Limit Group database). If the LCS recovery results are outside of these limits, the extract is reanalyzed. If LCS recoveries are still outside of QC limits after extract reanalysis but recoveries for the Matrix Spike/ Matrix spike Duplicate (MS/MSD) are within QC limits, the data is reported and a Non Conformance Memo (NCM) is written.

9.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD): A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. MS/MSD recoveries are evaluated against lab generated limits (refer to the current TALS Method Limit Group database). If the MS/MSD recovery limits fall outside of lab limits the LCS recovery is evaluated. If the LCS recoveries meet criteria the data is reported and a Non-Conformance Memo (NCM) is written.

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with a 2 component surrogate standard mix containing TCMX & DCB (see Section 7.2). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (refer to the current TALS Method Limit Group database). Minimum requirements for surrogate evaluation:

- Both surrogates must have reportable results that meet the acceptance criteria;
- Reported surrogates must be from a column with a passing CCV;
- At least one surrogate must pass on any column from which target analytes are identified and reported.

If both TCMX and DCB recovery are outside of acceptance limits the sample extract is reanalyzed to confirm. If the recoveries are still outside of limits the sample must be re-extracted and reanalyzed or the data flagged as “estimated concentration”.

9.1.5. Internal Standard: The internal standard (1-bromo-2-nitrobenzene) must elute within 30 seconds of and have an area response of 50 to 100% as compared to the most recent preceding calibration standard.

9.2. Instrument QC

9.2.1. GC System Performance Check

9.2.1.1. Endrin/4,4'-DDT Breakdown: Prior to performing any standards or sample analysis, a daily check is made on the chromatographic performance of the system. This performance check is made by injecting a standard of Endrin and DDT, each at a 250-ppb level (see Section 7.2), and calculating the percentage breakdown for each compound.

9.2.1.2. Ideally, only two peaks will be seen (one for Endrin and one for DDT). As a rule, this is not the case. It is normal to observe up to six peaks. Three peaks are attributable to Endrin and its degradation products: Endrin Aldehyde (EA) and Endrin Ketone (EK). Three peaks are attributable to DDT and its degradation products: DDE and DDD. Calculate the percentage breakdown as follows:

Endrin:

$$\frac{\text{(Areas of EA + EK)}}{\text{(Areas of EA + EK + Endrin)}} \times 100 = \%\ \text{breakdown Endrin}$$

DDT:

$$\frac{\text{(Areas of DDE + DDD)}}{\text{(Areas of DDE + DDD + DDT)}} \times 100 = \%\ \text{breakdown DDT}$$

9.2.1.3. If the percentage breakdown for either Endrin or DDT is greater than 15%, the system CANNOT be used for pesticide analysis. If the Endrin/DDT performance check fails, injection port/column maintenance must be performed. Usually, changing the glass wool/liner will cure most breakdown problems in the injection port. Depending upon the nature of the samples, the entire injection port will occasionally need to be cleaned. This cleaning is best done with 1:1 Acetone: Hexane. Another routine maintenance operation to improve column performance is the removal of the first 3 cm of the column. (Note: the septa should be changed each time the injection port is opened).

9.2.1.4. After injection port/column maintenance has been performed, and the columns have been given time to equilibrate (baseline back down to normal) the Endrin/DDT must be re-injected and the system performance re-evaluated.

9.2.2. Initial Calibration Range and Initial Calibration Verification (ICV)

9.2.2.1. ***Initial Calibration Range*:** Single component pesticides are calibrated using a five-point calibration range. Multi-component pesticides are calibrated using a single point calibration at the anticipated midpoint of the calibration range. Standards are prepared following the instructions in Section 7.2.

9.2.2.2. Single response Pesticide Calibration: All single component pesticides and two surrogates are calibrated with a minimum of 5 concentrations. Single component pesticides are analyzed at 10, (5 ppb for the 125 ml initial volume method) 5, 100, 250 and 500

ppb. Surrogate standards are analyzed at 25, 50, 100, 150 and 200 ppb. See Section 7.2 for details on standard prep.

9.2.2.3. Multi-response Pesticide Calibration: Chlordane (technical) and Toxaphene initial calibration is accomplished by analysis of a single point at 1000 ppb (see Section 7.2 for details on standard prep).

9.2.2.4. ***Initial Calibration Verification ICV*** An Initial Calibration Verification (ICV) standard is analyzed immediately after the Initial Calibration Range and before any samples are analyzed. The ICV is prepared as detailed in Section 7.2 and must be from a source separate from the standards used in the Initial Calibration Range.

9.2.3. Continuing Calibration Verification (CCV): For single component pesticides, a mid-point Continuing Calibration Verification (CCV) must be analyzed every 12-hours or 20 samples (whichever is more frequent). 'Samples' here are defined as field samples and batch QC (MB, LCS, MS, MSD) and do not include CCVs, PEMs, instrument blanks, etc… For multi-response pesticides a CCV must be analyzed within 12 hours of any multi-response pesticide detects.

9.2.3.1 Analysis of Replicate CCVs: Occasionally dual, sequential CCVs may (for a variety of reasons) be included in an analytical sequence. When such replicate CCVs are injected both must be evaluated as detailed in the table below (reference TestAmerica Document CA-Q-W-008, 'Technical Guidance on the Use and Evaluation of Replicate Continuing Calibration Verification (CCV)').

Dual CCV Evaluation Decision Tree		
Inejection	**QC Acceptance**	**Action**
CCV1 **CCV2**	**Pass** **Pass**	Continue analytical sequence. Data acceptable based on calibration.
CCV1 **CCV2**	**Pass** **Fail**	Analyses before CCV1 may be accepted. Re-analyze all samples that were analyzed after failed CCV2.
CCV1 **CCV2**	**Fail** **Pass**	Re-analyzed all samples that were analyzed before CCV1 and after the previous compliant CCV. Analyses after CCV2 may continue.
CCV1 **CCV2**	**Fail** **Fail**	Re-analyze all samples since the last acceptable CCV. Perform maintenance and/or recalibration prior to re-analysis.

9.2.3.2 In the event that replicate CCVs are included in a sample injection sequence, the acceptance of the associated sample analyses must be properly evaluated and the evaluation process must be documented. Under no circumstances is it allowable to accept sample data based on the evaluation of only 1 of the CCV replicates.

9.2.3.3 Clear documentation of the evaluation of the CCV must be included in the analytical sequence run log (i.e., 'Pass' or 'Fail' in the comment section for each CCV). Additionally, clear documentation of corrective action taken in the event of CCV failures must be included in the run log (see table above for actions that need documentation).

Note: It is acceptable to use the CCV solution as a primer at the beginning of an analytical sequence however the injection should be documented as a 'Primer' in the sequence rather than as a CCV.

9.2.4. Calibration Acceptance Summary

9.2.4.1. Retention Time Windows: Retention time windows must be established to compensate for minor shifts in absolute retention times as a result of sample loading and normal chromatographic variability (for more detail on the following procedures refer to TestAmerica Corporate Technical Services Policy No. CA-T-P-005, "Policy for Determining RT Windows for GC/ECD Tests" and TestAmerica Corporate Technical Services Work Instruction No. CA-T-WI-010, "Further Guidance on the RT Window Policy No. CA-T-P-005").

9.2.4.1.1. Initial determination of RT windows.

9.2.4.1.1.1. The center of the retention time (RT) window shall be updated based on the middle level in the initial calibration of the first CCV in the daily sequence, whichever is most recent.

9.2.4.1.1.2. Use a default RT window of 0.01 minutes. At the end of the batch evaluate all CCVs and LCSs in the batch. If necessary, widen the window such that all analytes fall within the RT window. Reprocess the batch using the new RT windows.

9.2.4.1.1.3. Multiply the maximum deviation by 1.5. This is the retention time window, unless the result is less than 0.01 minutes, in which case the window is set at 0.01 minutes. For example, if

the maximum RT deviation for a specific analyte is 0.008 minutes, then the RT window is set at ±0.012 minutes.

NOTE: For the multi-component analytes, for example Aroclors, Toxaphene and Technical Chlordane, the maximum deviation must be evaluated for each of the 3 to 6 major peaks used for sample calculations.

9.2.4.1.1.4. Retention time windows for analytes of interest must not overlap for GC analysis.

9.2.4.1.2. Ongoing evaluation of retention time windows

9.2.4.1.2.1. Evaluate the retention time windows on an ongoing basis. The center of the RT window is updated on the first CCV of the day. All analytes for all subsequent CCVs, LCS and matrix spikes must fall within the retention time window (except as discussed below).

9.2.4.1.2.2. Matrix spike analytes may fall outside of the retention time window if there is a large non-target peak coeluting with the analyte in the matrix spike.

9.2.4.1.2.3. If any analytes fall outside of the retention time window in CCVs, LCS or matrix spikes (except as discussed above for matrix spikes) then the RT windows for those analytes shall be widened to the minimum degree required for the analyte to fall within the RT window. All samples in the batch shall be reprocessed with the new RT window, and the wider RT window shall remain in place for subsequent batches.

9.2.4.1.2.4. Retention time windows should be reliably narrower than ±0.03 minutes. If RT windows wider that this are necessary, the instrument should be evaluated and maintenance performed as needed. Subsequent to maintenance, RT windows shall be narrowed to the extent that is consistent with the data obtained.

9.2.4.2. **Initial Calibration Range:** Internal standard calibration is employed for this method. A response factor is calculated for each analyte at each calibration concentration.

Response factor = $((A_x)\ (C_{is}))/((A_{is})\ (C_x))$

Where:
A_x = area of the compound
C_x = Concentration of the compound
A_{is} = area of the internal standard
C_{is} = Concentration of the internal standard

9.2.4.2.1. Calculate the percent Relative Standard Deviation of the response factors for each compound at each level:

% RSD = (RF Standard Deviation/RF Mean) X 100

Where:
RF = Response Factor

9.2.4.2.2. If the % RSD across the 5 point range is <20% for any given compound the calibration can be assumed to be linear and the average response factor can be used to calculate concentrations of target compounds in samples.

9.2.4.2.3. If the % RSD is >20% for any given compound, a first order linear regression may be applied to the data to calculate the calibration curve and determine sample concentration. If this method is employed, the r^1 (Correlation Coefficient) value must be $\geq$ 0.990 for the calibration to be acceptable. Calibration is checked every 12 hours or after every twenty (20) samples, whichever comes first, by injecting a calibration verification standard for all single component pesticide standards.

9.2.4.2.4. Chlordane and Toxaphene Calibration: Chlordane and Toxaphene are multiple response pesticides and are calibrated with a minimum of 5 points as required (i.e, within 12 hours of either analyte being detected in a sample). Three to eight peaks are used for calculation of response factors and the same criteria detailed above is applied to determine acceptability of calibration.

9.2.4.2.5. Resolution: All single component analyte peaks must exhibit at least 80% chromatographic resolution. The analyst performs a visual check of the mid-level initial calibration standard and all subsequent calibration checks (ICV/CCV). If the resolution requirement is not met instrument maintenance should be performed followed by re-calibration. Percent resolution can be calculated when necessary as follows:

% Resolution =V/H x 100

Where:

V= Depth of the valley between two peaks. The depth of the valley is measured along a vertical line from the level of the apex of the shorter peak to the floor of the valley between the two peaks

H = Height of the shorter of the adjacent peaks

9.2.4.3. Initial Calibration Verification (ICV): An ICV will consist of a second source standard at or near the midpoint of the Initial Calibration Range analyzed at the frequency specified in Section 9.2.2.4. The calculated concentration of the ICV must be within ±20%D of the expected concentration. Should the %D exceed 20% the analyst should take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the ICV. If the %D still exceeds 20% after a single ICV reinjection, a new Initial Calibration Range must be analyzed. All single component analyte peaks must exhibit 80% resolution (see Section 9.2.4.2.5)

9.2.4.4. Continuing Calibration Verification (CCV): A CCV will consist of a second source standard at or near the midpoint of the Initial Calibration Range analyzed at the frequency specified in Section 9.2.3.. The calculated concentration of the CCV must be within ±20%D of the expected concentration. Should the %D exceed 20% the analyst should take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the CCV. If the %D still exceeds 20% after a single CCV reinjection, a new Initial Calibration Range must be analyzed. All single component analyte peaks must exhibit 80% resolution (see Section 9.2.4.2.5).

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10. Procedure

10.1. Gas Chromatograph Operation

10.1.1. The sequence of events for GC analysis involves many steps. First the injection system and column performance and calibration must be verified. Maintenance operations are performed as needed. Then samples must be run on the instrument. Chromatograms and reports must be evaluated for content, integration and concentration. Re-runs and dilutions must be made based on the calibrations that were in effect at the time the sample was run. Lastly, a detailed analysis and calculations must be performed to determine the concentration of all the parameters for which the sample was analyzed.

10.1.2. General Operating Conditions

10.1.2.1. Injection System: A split/splitless injection port with electronic pressure control (EPC) is used. Thirty seconds after sample injection, the purge valve is turned on to facilitate the sweeping of any remaining residual solvent/sample from the injection port.

10.1.2.2. The EPC is used in the ramp pressure mode. The ramp pressure program is as follows:

Initial Pressure	InitialTime	Rate	Final Pressure	Hold
12 psi	2.5 min	7 psi/min	4 psi	1.50 min
		5 psi/min	9 psi	1.40 min
		9 psi/min	13 psi	2.00 min

10.1.2.3. For pesticide analysis the normal operating conditions of the injection port are as follows:

Injection port Temperature:	250^{o}C
Column flow:	12.3 ml/minute
Split vent flow:	5 ml/minute
EPC:	Pressure Ramp
Detector temperature	330C

10.1.2.4. In addition to the EPC, the injection port is also equipped with a siltek-coated glass double goose neck liner that contains a 1 cm glass wool plug. The plug of glass wool is located in the liner between the double goose neck.

10.1.2.5. This liner/glass wool combination provides many functions. The glass wool serves as a heat sink rapidly vaporizing solvent and samples resulting in higher response factors. The liner also protects the column head from accumulation of high boiling residuals and particulates.

10.1.2.6. The glass wool will be changed when changing the liner. The changing of the glass wool/liner is based upon the breakdown of an Endrin/DDT standard. This is covered in further detail in section 10.2.1.

10.1.2.7. Regular maintenance is performed on the injection port. When the glass wool/liner is changed, the septa also must be changed. Injection port, oven and detector temperatures are lowered to ambient prior to "cracking" the system. This is so as to introduce a minimum of damaging oxygen molecules into the system.

10.1.2.8. After the system has cooled, the old liner is removed. The injection port should be checked for particulate residues and cleaned as needed. A flashlight is usually required for this. After a new liner has been prepared it is placed into the injection port. A graphite seal is placed around the liner. The edges of the seal must be flat, not knife-edged, and free of nicks or burrs. If any of these conditions are not met, the graphite seal must be replaced as well. The graphite seal is critical to proper operation of the injection port. If in doubt, replace it.

10.1.2.9. The locking ring on the top of the injection port should be turned, with the wrench, about 1/8 turn past finger tight. The septum nut should never be tightened more than finger tight. After the injection port is reassembled, all column nuts inside the oven should be checked for leaks using Snoop (Supelco) or another suitable leak tester.

10.1.2.10. Once the signal from both detectors has stabilized, it is time to re-heat the zones. The zones should be heated in the order of detectors, oven and then injectors. This is to ensure that volatilized contaminants do not condense on the column or detector.

10.1.2.11. Oven: With the megabore columns installed, temperature programming is employed to achieve higher resolution of compounds and shorter run times than could be accomplished using isothermal methods.

10.1.2.11.1. A standard oven program for pesticide analysis is employed for all columns as follows:

Initial Temp	Hold Time1	Rate1	Temp1
160°C	0.62 min	30°/min	244°C
Hold Time2	**Rate 2**	**Final Temp**	**Final Time**
2.5min	21°/min	315°C	3.0min

10.1.2.12. Detectors: Detectors operate at 330°C and need to be supplied with 60 ml/min total flow. They are essentially maintenance free on a day-to-day basis. They are routinely baked out at 330°C to remove persistent contaminants. On occasion the detectors may be baked out at a higher temperature to remove contaminants with an extremely high boiling point (CAUTION: Do not exceed the maximum detector temperature of 380°C).

10.1.2.12.1. If the detectors are particularly contaminated, they must be sent to Agilent Technologies in Avondale, Pennsylvania for reconditioning. This should occur if the detector baseline is greater than 100 Hz. Detector reconditioning should be required at a maximum of biannually.

10.1.2.13. Chemstation: HP Chemstation software is used for automation of runs and data acquisition. The system is dedicated to a single GC and does not multitask. Therefore, data manipulation cannot be done while sample analysis is in progress. The data system acquires and stores all chromatographic data.

10.1.2.14. TestAmerica Chrom data processing software is used for the processing of the chromatography data files. Calibrations, verification standards and samples are processed and reviewed using this database. Chrom is integral to TestAmerica LIMS (TALS) which is used to generate all reports..

10.2. Analytical Sequence

10.2.1. The analytical sequence for performance checks, initial calibration, calibration verifications and sample analysis is described in the following sections.

10.2.2. Before calibration standards are analyzed the GC Performance Check Standard (see Section 9.2.2.1) must first be analyzed and evaluated to check the performance of the injection port and column with regard to catalytic active sites. The breakdown for both Endrin and DDT in the Performance Check Standard must be less than 15%. If the performance check fails this criteria system maintenance must be performed and the check successfully reanalyzed before proceeding with calibration.

10.2.3. A five point initial calibration (ICAL) is analyzed and evaluated for each of the 21 single response pesticides plus surrogates as described in Section 9.2.4.2. When needed a 5 point ICAL is also analyzed for Mirex.

10.2.4. Calibration for Technical Chlordane and Toxaphene: Chlordane and Toxaphene are multiple response pesticides containing at least 3-8 primary peaks each. A single point calibration check standard at a concentration of 1000ug/l is analyzed for Chlordane and Toxaphene. A full 5 point calibration range is analyzed for these compounds should they be detected in client samples..

10.2.5. A second source initial calibration verification (ICV) is analyzed and evaluated for each of the 21 single response pesticides as described in Section 9.2.4.3. When needed an ICV is also analyzed for Mirex. **NOTE**: The 12 hour time clock for Pesticides commences with the injection of the first Pesticide Calibration Standard or Verification.

10.2.6. GC Performance Check Standard is analyzed and the breakdown of Endrin/DDT is measured again before samples are analyzed and at the beginning of each subsequent 12 hour shift. If the breakdown check fails, then injection port/column maintenance is required

10.2.7. Client samples and QC samples may be analyzed after the analysis of the performance check. Sample analysis may proceed for up to 20 samples or 12 hours prior to analysis of another calibration verification (whichever is more frequent).

10.2.8. A Continuing Calibration Verification (CCV) for the 21 single response pesticides plus surrogates must be analyzed and evaluated every 12 hours or 20 samples (whichever is more frequent) as described in Section 9.2.4.4.. This is accomplished by running the midpoint calibration standard as a CCV (Pest Mix 100 ppb check standard and Mirex 100 ppb check standard; see Section 7.2.2.1). The calculated concentration for each compound in the CCV must be +/- 20 % of the expected concentration. Any samples analyzed after a failing CCV must be reanalyzed under a passing CCV. If, after performing instrument maintenance, the reanalysis of a CCV fails criteria, a new initial calibration range must be analyzed (see Section 9.2.4.2). Any data reported against a failing CCV must have a Non-Conformance Memo detailing the issue.

10.2.9. Analytical Sequence: The automation of GC runs is accomplished via the "SEQUENCE" macro of the Chemstation. The sequence file contains the name of Method file corresponding to the type of analysis to be performed, the range of samples to be run, and the number of injections per bottle. It is common practice to run the check standards, evaluate the instrument status, and then complete the Sample Table and Sequence File. If everything else is complete, the run is initiated using the START SEQUENCE soft-key of the SEQUENCE macro.

Example Analytical Sequence	
1. Hexane 2. Instrument Blank 3. Endrin/DDT Breakdown 4. Pesticide Mix 1 (2.5 ppb) 5. Pesticide Mix 2 (50 ppb) 6. Pesticide Mix 3 (100 ppb) 7. Pesticide Mix 4 (250 ppb) 8. Pesticide Mix 5 (500 ppb) 9. Chlordane (1000 ppb) 10. Toxaphene (1000 ppb) 11. Pesticide ICV (100 ppb)	12. Mirex 1 (10 ppb) (if needed) 13. Mirex 2 (20 ppb) (if needed) 14. Mirex 3 (100 ppb) (if needed) 15. Mirex 4 (250 ppb) (if needed) 16. Mirex 5 (500 ppb) (if needed) 17. Mirex ICV (100 ppb) (if needed) 18. Instrument Blank 19. Endrin/DDT Breakdown 20. Pest Mix 3 (100 ppb)…CCV 21. Mirex 3 (100 ppb) …CCV (if needed) 22. 20 or fewer samples or 12 hours 23. Instrument Blank 24. Endrin/DDT Breakdown 25. Pest Mix 3 (100 ppb)…CCV 26. Mirex 3 (100 ppb) …CCV (if needed) 27.20 or fewer samples or 12 hours

10.3. Dual Column Approach

10.3.1. The laboratory designates the rear column as the primary column and the front column as the secondary column. If the difference between the dual columns results in ≤40% RPD report the higher concentration.

10.3.2. The values are calculated from the chromatographic peaks that fall within the daily retention time windows. Retention times are updated using the retention times found in opening CCV for the most recent 12 hour period.

10.3.3. If the calculated values are greater than 40% RPD of each other, report the lower concentration regardless of whether that result is from the primary or secondary column. Report the result with a flag of P*. **Exception:** NJDEP DKQP protocols require reporting the higher concentration in all instances.

10.3.4. If the surrogates on one column are >40% RPD compared to the other column, this may be indicative of a bad injection or columnar blockage. The sample should be reanalyzed. If similar results are obtained following reanalysis, report the lower of the two numbers and describe the circumstances in the job summary and report case narrative.

10.3.5. If one of the columns fails CCV criteria (but the CCV is between 15%-40% greater than expected value), the sample results shall be reported from the compliant column. If the falls outside of acceptance criteria on the low side, reanalysis shall be performed.

10.3.6. If the CCV on one of the columns is more than 40% different from the correct value, it can be assumed that there has been significant drift on that column. The sample shall be reanalyzed against an acceptable calibration.

10.3.6.1. An exception to this requirement would be if the CCV recovery on one column fails on the high side and >40% RPD but the associated samples were non-detect for all target analytes on both columns. In this case the non-detect results may be reported from the compliant column.

10.3.7. In some cases where the sample chromatography is complex and has largely varying peaks concentrations, the chromatographic separation may not be sufficient on the 0.53mm ID columns. In this case a confirmatory analysis on an instrument with 0.32 ID columns may be required. The supplemental data produced using analysis on the 0.32mm ID 'microbore' column may minimize overlapping and baseline interference difficulties, and better resolves potential positive identifications. Use of this alternative chromatographic technique shall be noted in the job summary and report case narrative.

10.3.8. In summary, the flow chart in Attachment 1 presents a recommended rational approach to selecting the better number to report for dual column data. It shall be noted that these recommendations may be overridden by project specific requirements and that they cannot cover all eventualities. The complexity of some data set will require the final decision to be made utilizing the judgment of experienced analysts. In some cases further cleanup steps to remove interferences may be appropriate.

10.4. Extract Cleanups

10.4.1. Cleanup methods are dictated by the original sample matrix and the parameters being determined.

10.4.2. Cleanup of all water samples, if needed, is performed using Florisil (TestAmerica Edison SOP No. ED-ORP-020*: □□ri□i□□ □□n□□ □r □□□i□id□□□□□ □□□ □□□ E□r□□□□*, SW846 Method 3620B, most current revision) and TBA sulfite (TestAmerica Edison SOP No. ED-ORP-021: *T□□ □□□ □□□□□□E□□□ □n□□□□□□□r r□□ □□□i□id□□□□□ □□□ □□□ E□r□□□□□□□ □□□ □ □□□□d □□□□□□*most current revision). Blanks must also undergo cleanup following the same procedures as samples.

10.4.3. Cleanup of all soil samples is conducted using Florisil (TestAmerica Edison SOP No. ED-ORP-020*: □□ri□i□□ □□□n□□ □r □□□i□id□□□□□ □□□ □□□ E□r□□□□*, SW846 Method 3620B, most current revision) and, if needed, TBA sulfite (TestAmerica Edison SOP No. ED-ORP-021: *T□□ □□□ □□□□□□E□□□ □n□□□□ □□□□r r□□ □□□i□id□□□□□ □□□ □□□ E□r□□□□□□□ □□□ □ □□□□d □□□□□□*most current revision). Blanks must also undergo cleanup following the same procedures as samples.

10.4.4. Check Florisil prior to use to assure quantitative recovery of target analytes. (see Section 4.3.2 above and TestAmerica Edison SOP No. ED-ORP-020: □□ri□i□□ □□n□□ □r □□□i□id□□□□ □□□ □□ E□r□□□, SW846 Method 3620B,

10.5. Documentation

10.5.1. Before the analysis sequence is initiated the GC Maintenance logbook must be filled out. It should contain the following information: date, injector temp, oven temp, detector temp, injector flow, signal A, signal B, analysts initials, and notes for any necessary repairs.

10.5.2. After samples have been run, each standard and sample must be entered into the Instrument Run Log. The Instrument Run Log should contain the following information: run date, data file name, vial position, sample number, initial volume/weight, final volume, dilution factor, method, job number, QA number, extraction date, lab prep batch, Chrom batch signature of analyst at the bottom of each page, lot numbers for standards used, and result of run (O.K., dilution, non-inject, etc.).

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Calculation of Sample Amounts (Internal Standard Procedure)

11.3.1.1 Aqueous Samples

$$\text{Concentration } (\mu g/L) = \frac{(As)(Cis)(D)}{(Ais)(RF)(Vs)\ (Vi)\ (1000)}$$

Where:

As	=	Area of the target analyte peak in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.

Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the internal standard peak
RF	=	Average response factor from the initial calibration.
Vs	=	Volume of sample extracted (ml)

The 1000 in the denominator represents the number of ul in 1 ml.

11.3.1.2 Solid Samples

$$\text{Concentration } (\mu g/kg) = \frac{(As)(Cis)(D)(Vt)}{(Ais)(RF)(Ws)\ (Vi)\ (1000)}$$

Where:

As	=	Area of the target analyte peak in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the internal standard peak
RF	=	Average response factor from the initial calibration.
Vt	=	Volume of concentrated extract (ul)
Ws	=	Weight of sample (g)

The 1000 in the denominator represents the number of ul in 1 ml.

11.4. Relative Response Factors

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

A_x = Area of target analyte peak
Ais = Area of internal standard peak
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

11.5. Percent Relative Standard Deviation (% RSD):

$$\%\ RSD = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

11.6. Percent Difference (% D):

$$\% D = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} X\ 100$$

Where: RRFc = RRF from continuing calibration

$\overline{RRF_i}$ = Mean RRF from current initial calibration

11.7. Percent Recovery (% R): Surrogates and Spikes

$$\text{Recovery (\%)} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}} \times 100$$

11.8. Dry Weight Correction: All solid samples must be corrected for dry weight using the following formula for dry weight determination.

$$DW = \frac{Gd}{Gw} \times 100$$

Where:

DW	=	Percent % Dry Weight
Gd	=	Dry weight of selected sample aliquot
Gw	=	Wet weight of selected sample aliquot

Multiply the DW value times the wet weight of the sample extracted.

NOTE: All dry weight corrections are made in TALS at the time the final report is prepared.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 20 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, *Tr□inin□*, for the laboratory's training program.

13.0. Pollution Control

13.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

13.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage.

14.0. Waste Management

14.1. The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. 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. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.2. The following waste streams are generated as a result of this analysis:

- Expired Standards – The vials are collected in a 1 gallon polyethylene bucket. These vials are then transferred to an open top 55 gallon steel or polyethylene waste drum. These drums are transported to a waste facility for proper disposal.

- Auto sampler vials and expired standards: These vials are collected in satellite accumulation within the instrument laboratory. The vials are then placed into a 55 steel open top drum in the waste room. When the drums are full, the drum will be collected by the waste vendor for disposal. This waste is treated for incineration.

  Teris Profile Number: 50016652
  Onyx Profile WIP Number: 282493

- Mixed Solvent Waste: Mixed solvent waste is collected in a small beaker inside the bench top hood. This waste is then transferred into the satellite accumulation container in the Organic Prep. Lab. on a daily basis. This material is transferred into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.

  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

  Teris Profile Number (incineration): 50016710
  Onyx Profile Number: (stabilization) 402535

15.0. References / Cross-References

15.1. United States Environmental Protection Agency, "Method SW8000C: Determinative Chromatographic Separations,", Test Methods for Evaluating Solid Wastes, SW846, Revision 3, March 2003.

15.2. United States Environmental Protection Agency, "Method 8081B, Organochlorine Pesticide by Gas Chromatography", Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 2, February 2007.

15.3. TestAmerica Edison Document No. ED-QA-LQM, most current revision.

15.4. TestAmerica Edison SOP No. ED-ORP-014: most current revision.

15.5. TestAmerica Edison SOP No. ED-ORP-018: most current revision.

15.6. TestAmerica Edison SOP ED-ORP-044,

15.7. TestAmerica Edison SOP No. ED-ORP-019: most current revision.

15.8. TestAmerica Edison SOP No. ED-ORP-020: , SW846 Method 3620B, most current revision.

15.9. TestAmerica Edison SOP No. ED-ORP-021: *T□□ □□□ □□□□□□□E□□□ □n□□□□□□□□r □r□□ □□□i□id□□□□□ □□□ □□□ E□r□□□□□□□□ □□□ □ □□□□d □□□□□□*most current revision.

15.10. TestAmerica Edison SOP No. ED-GEN-022, *Tr□inin□□*most current revision.

15.11. Test America Corporate Quality SOP No. CA-Q-S-001 (□□□□□n□□ □□id □□□T□□□in□ □ □□□r□□□□), most current revision.

15.12. TestAmerica Edison SOP No. ED-GEN-023 (□□□□ □□□□□n□T□□□in□ □nd □□□r□□□□), most current revision.

15.13. TestAmerica Document CA-Q-W-008, 'Technical Guidance on the Use and Evaluation of Replicate Continuing Calibration Verification (CCV), most current version.

15.14. TestAmerica Corporate Quality SOP No. CA-Q-P-004, "Reporting Results for Methods that Require Second-Column Confirmation", most current revision.

15.15. TestAmerica Corporate Work Instruction No. CA-T-WI-008, "Minimum Requirements for Pesticide Analysis by SW846 Method 8081B", most current revision.

15.16. TestAmerica Corporate Policy CA-T-P-004, "Policy for Determining RT Windows for GC/ECD Tests", most current revision.

15.17. TestAmerica Corporate Technical Services Work Instruction No. CA-T-WI-010, "Further Guidance on the RT Window Policy No. CA-T-P-005", most current revision.

16.0. Method Modifications:

None

17.0. Attachments

Attachment 1: Dual Column Reporting Flowchart

18.0. Revision History

Revision 4, dated 09/13/2016:

- Section 9.2.4.1: completely rewrote this section to reflect requirements of TestAmerica Corporate Technical Services Policy No. CA-T-P-005, "Policy for Determining RT Windows for GC/ECD Tests" and TestAmerica Corporate Technical Services Work Instruction No. CA-T-WI-010
- Section 15 (References): added references to TestAmerica Corporate Technical Services Policy No. CA-T-P-005, "Policy for Determining RT Windows for GC/ECD

Tests" and TestAmerica Corporate Technical Services Work Instruction No. CA-T-WI-010, "Further Guidance on the RT Window Policy No. CA-T-P-005".

- Attachment 1: RT Windows for Single Analytes/Surrogates: DELETED and renamed subsequent attachments.

Revision 3, dated 01/05/2016:

- Section 2.2: expanded to include summary of internal standard calibration.
- Section 7.2.2: added 1-Bromo-2-nitrobenzene (internal standard) to the list of standards (Restek 32279). Components and concentration added to table.
- Added Section 7.2.2.4 which describes the preparation of the internal standard solution. All subsequent sections renumbered accordingly.
- Section 7.2.2.1.1: added footnote to ICAL standard prep table detailing requirement to spike each standard with internal standard solution.
- Section 7.2.2.4: describe internal standard spiking protocol (20 ul of 5.0 ug/ml solution into all standards, QC extracts and field sample extracts.
- Section 9.1: added internal standard to 'Sample QC' table.
- Section 9.1.4: added following minimum requirements for surrogate evaluation and reporting: a) Both surrogates must have reportable results that meet the acceptance criteria; b)reported surrogates must be from a column with a passing CCV; c)At least one surrogate must pass on any column from which target analytes are identified and reported. (per corp "Minimum Requirements" document).
- Section 9.1.5: added this section which describes acceptance criteria for internal standards (retention time and response).
- Section 9.2.3: Added the following sentence: "'Samples' here are defined as field samples and batch QC (MB, LCS, MS, MSD) and do not include CCVs, PEMs, instrument blanks, etc… ". Deleted the phrase 'and at the end of every sequence'. (both changes reflect 'Minimum Requirements for Pesticide Analysis' document).
- Added Section 9.2.4.1.3: "Updating absolute retention times: Update retention times with the retention times found in the opening CCV for that 12 hour period."
- Section 9.2.4.2: replaced 'external standard' with 'internal standard'. Added formula for calculation of response factors by internal standard method. Deleted text regarding calibration acceptance criteria from this section as it is duplicated in subsequent sections.
- Section 9.2.4.2.2: added phrase 'for any given compound.'
- Added section 9.2.4.2.5: describes resolution check.
- Sections 9.2.4.3 and 9.2.4.4: added statement concerning 80% resolution requirement.
- Section 9.2.4.4: changed 'ICV' to 'CCV' in last sentence.
- Section 10.1.2.13: Revised first sentence to read: ": HP Chemstation software is used for automation of runs and data acquisition."
- Section 10.2.2: corrected reference to Section 9.2.2.1 (was incorrectly listed as 7.2.2.1).
- Section 10.2.8: revised to clarify that CCV bracketing is not required and that only samples analyzed after a failing CCV must be reanalyzed (i.e., samples analyzed immediately prior to a failing CCV need not be reanalyzed).
- Section 10.3.2: added the following regarding updating retention times: "Retention times are updated using the retention times found in opening CCV for the most recent 12 hour period."

- Section 11: added formulas for calculation of sample concentrations as well as %RSD, %D, % Recovery, RRF and dry weight correction.
- Section 15: removed reference to SOP No. ED-ORP-016*: Automated Soxhlet Extraction of Solid Samples – Pesticides/PCBs, SW846 Method 3541* as it is no longer is use.
- Section 15: added reference to TestAmerica Corporate Quality SOP No. CA-Q-P-004, "Reporting Results for Methods that Require Second-Column Confirmation"
- Section 15: added reference to TestAmerica Corporate Work Instruction No. CA-T-WI-008, "Minimum Requirements for Pesticide Analysis by SW846 Method 8081B".
- Section 15: added reference to TestAmerica Corporate Policy Memorandum No. CA-Q-QM-006, "Technical Guidelines for Analysis of Complex GC/ECD Chromatograms".

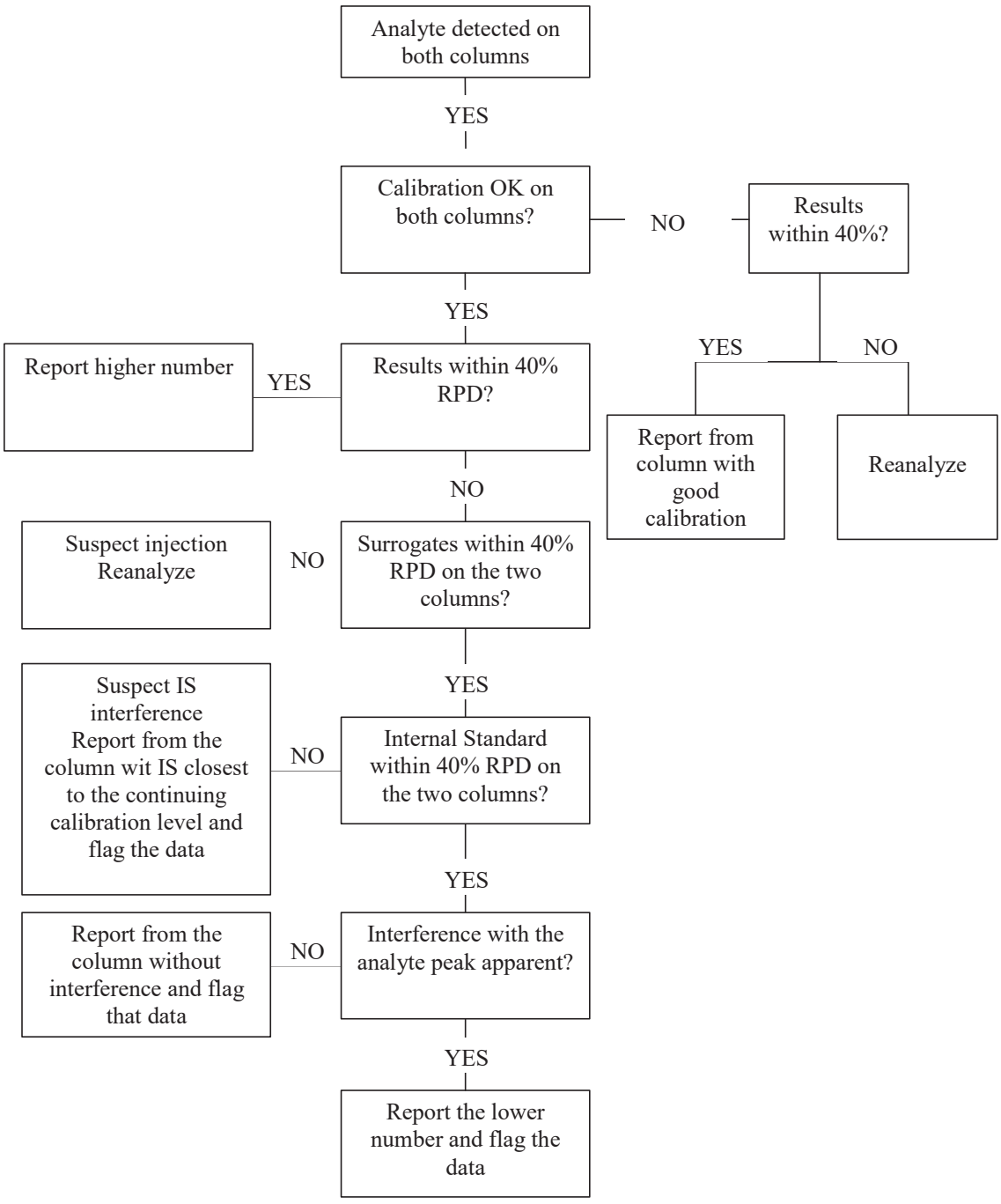
- Revision 2, dated 13 May 2015:
  - Section 1.1: updated RLs to reflect current lab practice.
  - Section 2.1 and throughout: revised initial aqueous/TCLP sample volumes to reflect current lab practice
  - Section 2.1.1: removed note describing option for Reduced Volume Extraction (RVE) as this is now standard lab practice.
  - Section 2.1.2 and throughout document: removed reference to prep by SW3541 as this method is no longer in use at Edison lab.
  - Section 4.3.2 and throughout: updated source of standards used for Florisil check.
  - Section 6.1.2.3.1: updated name of analytical GC columns currently in use.
  - Section 6.1.2.4.1: updated make-up gas to P-5 & Nitrogen.
  - Section 7.1.1: updated carrier gas to Hydrogen and make-up gas to P-5&Nitrogen.
  - Section 7.2.2 and throughout document: updated source and catalog numbers for analytical standards (Restek is now primary source of standards).
  - Section 7.2.2.1: updated standards prep instructions. Removed outdated references to standards and spiking mixes. Removed notes pertaining to RVE since this procedure is now standard and incorporated into instructions.
  - Section 7.2.2.10: added instructions for preparation of ICVs.
  - Section 8.1: adjusted water sample container from 1000ml to 250ml.
  - Section 9.2.2: removed references and instructions pertaining to the Pesticide Resolution Check (a CLP requirement we no longer perform).
  - Section 9.2.3.1: Added extensive discussion regarding the analysis of replicated CCVs.
  - Section 9.2.4.4: updated table to include new concentration for low standard (2.5 ppb).
  - Section 10.1.2.14 and throughout document: replaced references to Target with TestAmerica Chrom.
  - Section 10.2: made extensive revisions to discussion of the analytical sequence (removing duplicate entries and adjusting to reflect current procedures).
  - Section 10.3: added note concerning NJ DKQP requirements for reporting highest concentration of dual columns.
  - Section 15 (References): updated to include "Technical Guidance on the Use and Evaluation of Replicate Continuing Calibration Verification (CCV), most current version."
  - Added Sections 7.2.2.11 and 7.2.2.12 reflecting the addition and preparation of Internal standard

- Revision 1, dated 09 October 2012
  - Throughout: Revised LQM section number references to reflect the most current LQM revision.
  - Section 2.1.1: added description of reduced initial volume (125ml)/final volume (1ml) option.
  - Section 2.1.2: Added reference to TestAmerica Edison SOP ED-ORP-044, *Procedure for the Micro□a□e Extraction of Solids, SW846 3546.*
  - Section 7.2.2.1.1 added preparation of 5ppb standard for the reduced volume method.
  - Section 9.2.2.2 Added 5ppb standard for the reduced volume method
  - Section 9.2.4.4 Added 5 ppb standard to the initial calibration for the reduced volume method
  - Section 10.2.6 Added 5 ppb to the Analytical sequence for the low level method Section 15.0: Removed reference to TestAmerica Edison SOP EDS-GEN-019, □*r*□*anic Calculations,* most current revision.
  - Section 15.0: Added reference to TestAmerica Edison SOP ED-ORP-044, *Procedure for the Micro□a□e Extraction of Solids, SW846 3546*

- Revision 0, dated 02/17/2011: New

ATTACHMENT 1

Dual Column Reporting Flowchart
(Note: NJDEP DKQP requires reporting the higher concentration in all instances)



Title: SW846 Method SW8081B, Analysis of Organochlorine Pesticides by Gas Chromatography

Approvals (Signature/Date):

Catalina Dalangin SVOA GC Manager	9/13/16 Date	Dan Helfrich Health & Safety Manager	9/13/16 Date
Carl Armbruster Quality Assurance Manager	9/13/16 Date	Mark Acierno Laboratory Director	9/13/16 Date

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

Method 8081B is used to determine the concentrations of various organochlorine pesticides in extracts from solid and liquid matrices, using dual fused-silica, open-tubular, capillary columns with electron capture detectors (ECD). The list of analytes and their corresponding reporting limits are as follows:

Parameter		Soil	Water	Leachate
	CAS Registry No.	Reporting Limits (ug/Kg)	Reporting Limits (ug/L)	Reporting Limits (mg/L)
Aldrin	309-00-2	6.7	0.02	---
Alpha-BHC	319-84-6	2.0	0.02	---
Beta-BHC	319-85-7	2.0	0.02	---
Delta-BHC	319-86-8	2.0	0.02	---
Gamma-BHC (Lindane)	58-89-9	6.7	0.02	0.00050
Chlordane	57-74-9	67	0.50	0.0050
4,4' –DDD	72-54-8	6.7	0.02	---
4,4' –DDE	72-55-9	6.7	0.02	---
4.4' –DDT	50-29-3	6.7	0.02	---
Dieldrin	60-57-1	2.0	0.02	---
Endosulfan I	959-98-8	6.7	0.02	---
Endosulfan II	33213-65-9	6.7	0.02	---
Endosulfan sulfate	1031-07-8	6.7	0.02	---
Endrin	72-20-8	6.7	0.02	0.00050
Endrin aldehyde	7421-93-4	6.7	0.02	---
Endrin ketone	53494-70-5	6.7	0.02	---
Heptachlor	76-44-8	6.7	0.02	0.00050
Heptachlor epoxide	1024-57-3	6.7	0.02	0.00050
Methoxychlor	72-42-5	6.7	0.02	0.00050
Toxaphene	8001-35-2	67	.50	0.0050
Gamma- Chlordane	5103-74-2	6.7	0.02	---
Alpha-Chlordane	5103-71-9	6.7	0.02	---
Mirex	2385-85-5	6.7	0.010	---

The most current MDLs and RLs for this method can be found in the active TestAmerica LIMS (TALS) SW846 8081B Method Limit Group (MLG) database.

1.2 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work Request*) and Section 19 (*Test Methods and Method Validation)* of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1. Samples undergo a preparation step prior to analysis by SW846 Method 8081B. A measured volume or weight of sample (15g for soil, 1 g for waste, 250 ml for water, and 250 ml for TCLP) is extracted using the appropriate matrix-specific sample extraction technique. (Reference the applicable Organic Sample Prep SOPs listed below). The effective final volume is usually between 5 and 20 ml in hexane.

2.1.1. Aqueous samples are extracted using SW846 Method 3510C (SOP No. ED-ORP-014: *□□tra□tion of □esti□ides and □□□s in Water □□ □e□arator□ □unnel*).

2.1.2. Solid samples are extracted using SW846 Method 3550B: Sonication (*□□□ □o□□□□□R□□□□□□□□tra□tion of □esti□ides□□□s in □oil □sin□ □ow□ □evel □□tra□tion*) or SW846 Method 3546: Microwave (SOP No. ED-ORP-044: *□ro□edure for the Mi□rowave □□tra□tion of □olids□W□□□ □□□□)*□

2.1.3. Organic liquids are prepared using SW846 Method 3580A (*□□□ □o□□□□ □R□□□□□□Waste □ilution for □esti□ides and □□□s*).

2.1.4. Extract cleanup steps are employed depending on the nature of the matrix interferences. Suggested cleanups include SW846 Method 3620B (*□□□ □o□ □□□□R□□□□□□ □lorisil □leanu□ for □esti□ide□□□ □a□□le □□tra□ts*) and SW846 Method 3660B (*□□□ □o□ □□□□R□□□□□□ The Re□oval of □le□ental □ulfur fro□ □esti□ide□□□ □a□□le □□tra□ts*).

2.2. After cleanup, a small aliquot of the extract is injected into a gas chromatograph (GC) equipped with a capillary column. The GC is temperature programmed to separate the compounds which were recovered during the extraction step by boiling point. The effluent of the gas chromatograph is interfaced to an Electron Capture Detector (ECD) which is used to detect the compounds eluting from the GC.. Quantitation is accomplished by comparing the area response of each target analyte relative to an internal standard established through a five-point calibration (six points for second order regression). Specific calibration and quality control steps are included in the method that must be performed and must meet the specifications of SW846 Method 8081B.

2.3. For pesticide analysis a system performance check (DDT/Endrin breakdown) and a calibration verification standard must be run prior to analysis. Failure of either will generally indicate the need for injection port/column maintenance and/or recalibration.

2.4. Samples are analyzed after all the necessary checks have been performed. Samples analyzed for pesticides require an additional post analysis Quant Report to be printed and attached to the chromatographic report.

2.5. All samples are then manually reviewed. Secondary column confirmation of target compounds and quantitation are conducted by the analyst as required.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Interferences by phthalate esters introduced during sample preparation can pose a major problem in pesticide determinations.

4.1.1. Interferences from phthalate esters can be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

4.2. The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting organochlorine pesticides. Sulfur contamination should be expected with sediment samples. If sulfur is encountered, employ the sulfur removal procedures detailed in SW846 Method 3660B (□□□ □o□□□□□ R□□□□□□*The Re□oval of □le□ental □ulfur fro□ □esti□ide□□□□ □a□□le □□tra□ts*). Note that the recover of Endrin Aldehyde is adversely affected by the TBA cleanup procedure detailed in this method. Accordingly, this compound must be determined prior to sulfur cleanup.

4.3. Co-eluting chlorophenols are eliminated by using SW846 Method 3620B (□□□ □o□ □□□□R□□□□□□□*lorisil □leanu□ for □esti□ide□□□□ □a□□le □□tra□ts*).

4.3.1. Check Florisil prior to use to assure quantitative recovery of targeted analytes. Duplicate checks are required for each new lot or every three hundred samples whichever is more frequent.

4.3.2. Check Florisil by spiking 1ml of the Pest Std Mix A midpoint (Supelco Catalog No. 47977) and 0.5 ml of trichlorophenol (Absolute Standardsl Catalog No. 20024) onto the cartridge and concentrating to final volume of 1 ml. Inject 1 ul onto a capillary column, conducting the elution and analyzing the extract. Recovery is acceptable if all pesticides are recovered at 80 - 110% and the recovery of trichlorophenol is <5% and co-eluting interfering peaks are absent from the extract.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

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 MSDS 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 MSDS for each material before using it for the first time or when there are major changes to the MSDS.

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.
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.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation:

6.1.1. Gas Chromatograph: The system used is an HP and an Agilent Technologies (Avondale, PA) model 5890/6890 Gas Chromatograph (GC). Each GC is equipped for simultaneous quantitation and confirmation columns using two separate detector channels on dual megabore capillary columns that are suitable for the analysis of organochlorine pesticides. All operations are as automated as possible with the equipment utilized.

6.1.2. Injection system: Sample injection is accomplished by a single auto injector. The auto injector is serviced by a robot arm that shuttles samples between the sample tray and the injector turret.

6.1.2.1. The samples are injected into a split/splitless injection port equipped with electronic pressure control (EPC). The injection port is normally operated in splitless mode during injection. The EPC is operated in the ramp pressure mode.

6.1.2.2. Liners: The injection port is each fitted with replaceable, heavy-walled siltek-coated glass double gooseneck liner. The liner contains a plug of silanized glass wool approximately 1 cm in length. The glass wool is positioned in the liner between the double gooseneck. The liner is replaced on a regular maintenance schedule.

6.1.2.3. Oven and Columns: Temperature programmable gas chromatograph ovens are required, capable of integrated temperature control between 35^{O}C and 350^{O}C.

6.1.2.3.1. Two dissimilar columns are used for analysis. A Restek RtxCLPesticides, 30m x 0.53mm ID x 0.5um film thickness column is used for sample quantitation. The secondary confirmation column is a Restek RtxCLPesticides II, 30m x 0.53mm ID x 0.42um film thickness column.

6.1.2.4. Detectors: Sample detection is by electron capture. The GC is equipped with dual Electron Capture Detectors (ECD), one for each column.

6.1.2.4.1. Each detector is supplemented with make-up gas to provide sufficient detector flow for maintaining the electron plasma. This is in addition to the gas exiting the column. The make-up gas (P-5 & Nitrogen) is fed from a supply other than the injection port.

7. Reagents and Standards

7.1. Reagents

7.1.1. Gases: Hydrogen is used as the carrier and injection port purge gas. It is introduced to the GC at the injection port. P-5 and Nitrogen) is used as make-up gas. It is introduced to the GC via the make-up gas adapter at the end of the capillary column. Hydrogen is supplied via a Parker Balston H2 Generator. Nitrogen & P-5 is supplied by Air Gas

7.1.1.1. The gas streams are polished using three traps or filters before introduction to the GC The traps are as follows:

7.1.1.1.1. Hydrocarbon trap
7.1.1.1.2. H_2O trap
7.1.1.1.3. O_2 scrubber

7.1.1.2. Both the moisture trap and the Oxygen scrubber are of the indicating type. They require either replacement or reconditioning upon color change of the active agents. Refer to the instructions for the individual traps to determine if it is still active. The hydrocarbon trap is a simple activated carbon trap. With high quality gas, it should last for an extended period of time (1-yr. minimum).

7.1.2. Solvents used in the extraction and cleanup procedures include n-hexane, methylene chloride, and acetone that are exchanged to n-hexane prior to analysis.

7.1.3. Hexane is required in this procedure. All solvents must be pesticide quality or equivalent. Each lot of solvent is screened for contaminants before being used for analysis as detailed in TestAmerica Corporate Quality SOP No. CA-Q-S-001 (□*olvent* □ □□*id* □*ot Testin*□ □ □□□*roval*) and TestAmerica Edison SOP No. ED-GEN-023 (□*ulk* □*olvent Testin*□ *and* □□□*roval*).

7.2. Standards

7.2.1. Standards are purchased as concentrated solutions (see Section 7.2.2).

NOTE: Independent sources are used for quantitation standards and spiking standards

7.2.1.1. Most stock solutions are diluted (in volumetric glassware) to working concentration using hexane as the diluent as described in Section 7.2.2.1.

7.2.2. Standard mixes and sources *.

Standard Name	Source
Organochlorine Pesticide Mix AB #3	Restek Catalog No. 32415
Organochlorine Pesticide Mix AB #3.sec	Restek Catalog No. 32415.sec (second source)
Pesticide Surrogate Mix	Restek Catalog No. 32000
Endrin/DDT	Supelco Catalog No 48282
Chlordane	Restek Catalog No. 32021
Toxaphene	Restek Catalog No. 32005
Toxaphene (different pattern)	Restek Catalog No. 32071
Mirex	Accustandard Cat. No. P-066S-10X
1-Bromo-2-nitrobenzene (internal standard)	Restek Cat. No. 32279

*Suppliers with equivalent standards may be substituted..

The components of each standard mix are as follows (note: 'sec' indicates second source):

Parameter	Supplier	Catalog No.	Concentration of Standard (ug/ml)
Aldrin	Restek	32415 & 32415.sec	2000
Alpha-BHC	Restek	32415 & 32415.sec	2000
Beta-BHC	Restek	32415 & 32415.sec	2000
Delta-BHC	Restek	32415 & 32415.sec	2000
Gamma-BHC (Lindane)	Restek	32415 & 32415.sec	2000
Alpha -Chlordane	Restek	32415 & 32415.sec	2000
Gamma -Chlordane	Restek	32415 & 32415.sec	2000
Technical Chlordane	Restek	32021	1000
4,4' –DDD	Restek	32415 & 32415.sec	2000
4,4' –DDE	Restek	32415 & 32415.sec	2000
4.4' –DDT	Restek	32415 & 32415.sec	2000
Dieldrin	Restek	32415 & 32415.sec	2000
Endosulfan I	Restek	32415 & 32415.sec	2000
Endosulfan II	Restek	32415 & 32415.sec	2000
Endosulfan sulfate	Restek	32415 & 32415.sec	2000
Endrin	Restek	32415 & 32415.sec	2000
Endrin aldehyde	Restek	32415 & 32415.sec	2000
Endrin ketone	Restek	32415 & 32415.sec	2000
Heptachlor	Restek	32415 & 32415.sec	2000
Heptachlor epoxide	Restek	32415 & 32415.sec	2000
Methoxychlor	Restek	32415 & 32415.sec	2000
Toxaphene	Restek	32005	1000
Toxaphene	Restek	32071	5000
Mirex	Accustandard	861428-U	1000
4,4-DDT	Supelco	18282	500
Endrin	Supelco	48282	500
Decachlorobiphenyl (DCB)	Restek	32000	200
Tetrachloro-m-xylene (TCmX)	Restek	32000	200
1-Bromo-2-nitrobenzene (internal standard)	Restek	32279	1000

7.2.2.1. Standards Preparation

7.2.2.1.1. Calibration Mix (Organochlorine Pesticide Mix)

The 5 point calibration standards are prepared as detailed in the following table using volumetric glassware and hexane as the diluent:

Initial Calibration Standards Prep (Organochlorine Pesticide Mix)					
Stock Std	**2.5 ppb**	**50 ppb**	**100 ppb**	**250 ppb**	**500 ppb**
Organochlorine Mix AB#3 ***(2000 ug/ml)*** *Restek (Cat No..32415)in Hexane:Toluene*	Volume brought up to 200 ml hexane: *0.25 ul*	Volume brought up to 200 ml hexane: *5.0 ul*	Volume brought up to 500 ml hexane: *25 ul*	Volume brought up to 200 ml hexane: *25 ul*	Volume brought up to 200 ml hexane: *50 ul*
Initial Calibration Standards Prep (TCMX/DCB Surrogate Mix)					
Stock Std	**25 ppb***	**50 ppb**	**100 ppb**	**150 ppb**	**200 ppb**
Tetrachloro-m-xylene/ Decachlorobiphenyl Surrogates Mix (200 ug/ml) *Restek (Cat, No, 32000) in Acetone*	Volume brought up to 200 ml hexane: *6.25 ul*	Volume brought up to 200 ml hexane: *50 ul*	Volume brought up to 500 ml hexane: *250 ul*	Volume brought up to 200 ml hexane: *150 ul*	Volume brought up to 200 ml hexane: *200 ul*
Initial Calibration Standards Prep (Mirex)					
Stock Std	**2.5 ppb**	**50 ppb**	**100 ppb**	**250 ppb**	**500 ppb**
Mirex ***(1000 ug/ml)*** *Accustandard (Cat. No. P-066S-10x) in Methanol*	20x dilution of 50 ppb	10x dilution of 500 ppb	5x dilution of 500 ppb	2x dilution of 500 ppb	Volume brought up to 100 ml hexane: *50 ul*

Note: 20 ul of 5 ug/ml Internal Standard solution is added to all calibration standards prior to analysis.

7.2.2.2. **Pesticide Surrogate Spike Mix (10 ug/ml**) : The Pesticide 10ug/ml surrogate spiking solution is prepared by diluting 10 ml of 200 ug/ml of the Pesticide Surrogate Mix (Restek-32000) in to 200 ml of Acetone. For spiking instructions refer to the applicable prep SOP.

7.2.2.3. **Pesticide Surrogate Spike Mix (2 ug/ml) for the reduced volume extraction option:** prepare a 2 ug/ml spiking solution by diluting 10 ml of the 10 ug/ml stock standard described in 7.2.2.2 to a final volume of 50ml in acetone. For spiking instructions refer to the applicable prep SOP.

7.2.2.4. **Pesticide Internal Standard Spike Mix (5 ug/ml):** The Pesticide 5 ug/ml internal standard spike mix is prepared by

dilution 1 ml of 1000 ug/ml of the 1-Bromo-2-Nitrobenzene standard (Restek 32279) in to 200 ml of Hexane. 20 ul of this solution is added to all standards, QC samples and field sample extracts prior to analysis.

7.2.2.5. **Pesticide Spiking Standard (20 ug/ml):** The Pesticide Spiking Mix containing the single component pesticides is prepared at a final concentration of 20 ug/ml by diluting 500 ul of the Organochlorine Pesticide Mix AB#3 (Restek 32415 to a 50 ml final volume with acetone. For spiking instructions refer to the applicable prep SOP.

7.2.2.6. **Pesticide Spiking Standard (4 ug/ml) for the reduced volume extraction option**: prepare a 4 ug/ml spiking solution by diluting 10 ml of the 20ug/ml standard prepared in Section 7.2.2.4 above to a final volume of 50ml in acetone. For spiking instructions refer to the applicable prep SOP.

7.2.2.7. **System Performance Solution (Breakdown Check)** 3,3-DDT and Endrin at 0.25 ug/ml): The breakdown check is prepared by taking 250 ul of 500 ug/ml DDT/Endrin Mix (Supelco Catalog No 48282) and bringing it up to a volume of 500 ml with hexane

7.2.2.8. **Technical Chlordane Calibration Solution** (1.0 ug/ml solution w/ surrogates TCMX & DCB at 0.10 ug/ml): 100 ul of 1000 ug/ml Technical Chlordane Standard (Restek Catalog No 32021) and 50 ul of 200 ug/ml Surrogate Mix (Restek Catalog No. 32000). Dilute to 100 ml in Hexane. □□***T***□□ ICAL consists of single midpoint calibration standard for multiple responders Technical Chlordane and Toxaphene.

7.2.2.9. **Toxaphene Calibration Solution** (1.0 ug/ml solution w/surrogates TCMX & DCB at 0.1 ug/ml) : 20 ul of 5000 ug/ml Toxaphene stock (Restek Catalog No 32071) and 50 ul of 200 ug/ml Surrogate Mix (Restek Catalog No. 32000). Dilute to 100 ml in Hexane. □□***T***□□ ICAL consists of single midpoint calibration standard for multiple responders Technical Chlordane and Toxaphene.

7.2.2.10. **Initial Calibration Verification (ICV)** Preparation: follow the instructions above for midpoint standards substituting the second source standard (catalog no. suffix = sec) for the primary standards.

8. Sample Collection, Preservation, Shipment and Storage

8.1. Extracts must be stored under refrigeration in the dark and analyzed within 40 days of extraction.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Amber glass, 250 ml	250 ml	Cool 4 ± 2°C	7 days to extraction; Analyze within 40 days of extraction	SW846
Soils	Glass, 2 or 4 oz	100 g	Cool 4 ± 2°C	14 days to extraction; Analyze within 40 days of extraction	SW846

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

9. Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]
Internal Standard[1]	Every sample[3]	Response within -50% to +100% of most recent cal standard

LCS Duplicate (LCSD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into LIMS.

9.1.1. **Method Blanks** are extracted with each sample batch on each day that samples are extracted. The analytical results for the method blank must fall below the reporting limit for each compound of interest. If a target compound is detected in the blank at a concentration higher than the reporting limit, first the extract is reanalyzed for confirmation. If results are still outside of limits the entire batch of samples extracted with the affected blank must be re-extracted and reanalyzed.

9.1.2. Laboratory Control Sample (LCS): A Laboratory Control Sample (LCS) or blank spike must be extracted and analyzed for with each batch of 20 environmental samples. The recoveries of the LCS must fall within lab generated acceptance criteria (refer to the current TALS Method Limit Group database). If the LCS recovery results are outside of these limits, the extract is reanalyzed. If LCS recoveries are still outside of QC limits after extract reanalysis but recoveries for the Matrix Spike/ Matrix spike Duplicate (MS/MSD) are within QC limits, the data is reported and a Non Conformance Memo (NCM) is written.

9.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD): A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. MS/MSD recoveries are evaluated against lab generated limits (refer to the current TALS Method Limit Group database). If the MS/MSD recovery limits fall outside of lab limits the LCS recovery is evaluated. If the LCS recoveries meet criteria the data is reported and a Non-Conformance Memo (NCM) is written.

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with a 2 component surrogate standard mix containing TCMX & DCB (see Section 7.2). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (refer to the current TALS Method Limit Group database). Minimum requirements for surrogate evaluation:

- Both surrogates must have reportable results that meet the acceptance criteria;
- Reported surrogates must be from a column with a passing CCV;
- At least one surrogate must pass on any column from which target analytes are identified and reported.

If both TCMX and DCB recovery are outside of acceptance limits the sample extract is reanalyzed to confirm. If the recoveries are still outside of limits the sample must be re-extracted and reanalyzed or the data flagged as “estimated concentration”.

9.1.5. Internal Standard: The internal standard (1-bromo-2-nitrobenzene) must elute within 30 seconds of and have an area response of 50 to 100% as compared to the most recent preceding calibration standard.

9.2. Instrument QC

9.2.1. GC System Performance Check

9.2.1.1. Endrin/4,4'-DDT Breakdown: Prior to performing any standards or sample analysis, a daily check is made on the chromatographic performance of the system. This performance check is made by injecting a standard of Endrin and DDT, each at a 250-ppb level (see Section 7.2), and calculating the percentage breakdown for each compound.

9.2.1.2. Ideally, only two peaks will be seen (one for Endrin and one for DDT). As a rule, this is not the case. It is normal to observe up to six peaks. Three peaks are attributable to Endrin and its degradation products: Endrin Aldehyde (EA) and Endrin Ketone (EK). Three peaks are attributable to DDT and its degradation products: DDE and DDD. Calculate the percentage breakdown as follows:

□*ndrin:*

(Areas of EA + EK)_____ X 100 = % breakdown Endrin
(Areas of EA + EK + Endrin)

□□*T:*

(Areas of DDE + DDD)_____ X 100 = % breakdown DDT
(Areas of DDE + DDD + DDT)

9.2.1.3. If the percentage breakdown for either Endrin or DDT is greater than 15%, the system CANNOT be used for pesticide analysis. If the Endrin/DDT performance check fails, injection port/column maintenance must be performed. Usually, changing the glass wool/liner will cure most breakdown problems in the injection port. Depending upon the nature of the samples, the entire injection port will occasionally need to be cleaned. This cleaning is best done with 1:1 Acetone: Hexane. Another routine maintenance operation to improve column performance is the removal of the first 3 cm of the column. (Note: the septa should be changed each time the injection port is opened).

9.2.1.4. After injection port/column maintenance has been performed, and the columns have been given time to equilibrate (baseline back down to normal) the Endrin/DDT must be re-injected and the system performance re-evaluated.

9.2.2. Initial Calibration Range and Initial Calibration Verification (ICV)

9.2.2.1. □***nitial □alibration □ange*:** Single component pesticides are calibrated using a five-point calibration range. Multi-component pesticides are calibrated using a single point calibration at the anticipated midpoint of the calibration range. Standards are prepared following the instructions in Section 7.2.

9.2.2.2. Single response Pesticide Calibration: All single component pesticides and two surrogates are calibrated with a minimum of 5 concentrations. Single component pesticides are analyzed at 10, (5 ppb for the 125 ml initial volume method) 5, 100, 250 and 500

ppb. Surrogate standards are analyzed at 25, 50, 100, 150 and 200 ppb. See Section 7.2 for details on standard prep.

9.2.2.3. Multi-response Pesticide Calibration: Chlordane (technical) and Toxaphene initial calibration is accomplished by analysis of a single point at 1000 ppb (see Section 7.2 for details on standard prep).

9.2.2.4. ***Initial Calibration Verification (ICV):*** An Initial Calibration Verification (ICV) standard is analyzed immediately after the Initial Calibration Range and before any samples are analyzed. The ICV is prepared as detailed in Section 7.2 and must be from a source separate from the standards used in the Initial Calibration Range.

9.2.3. Continuing Calibration Verification (CCV): For single component pesticides, a mid-point Continuing Calibration Verification (CCV) must be analyzed every 12-hours or 20 samples (whichever is more frequent). 'Samples' here are defined as field samples and batch QC (MB, LCS, MS, MSD) and do not include CCVs, PEMs, instrument blanks, etc… For multi-response pesticides a CCV must be analyzed within 12 hours of any multi-response pesticide detects.

9.2.3.1 Analysis of Replicate CCVs: Occasionally dual, sequential CCVs may (for a variety of reasons) be included in an analytical sequence. When such replicate CCVs are injected both must be evaluated as detailed in the table below (reference TestAmerica Document CA-Q-W-008, 'Technical Guidance on the Use and Evaluation of Replicate Continuing Calibration Verification (CCV)').

Dual CCV Evaluation Decision Tree		
Inejection	**QC Acceptance**	**Action**
CCV1 **CCV2**	**Pass** **Pass**	Continue analytical sequence. Data acceptable based on calibration.
CCV1 **CCV2**	**Pass** **Fail**	Analyses before CCV1 may be accepted. Re-analyze all samples that were analyzed after failed CCV2.
CCV1 **CCV2**	**Fail** **Pass**	Re-analyzed all samples that were analyzed before CCV1 and after the previous compliant CCV. Analyses after CCV2 may continue.
CCV1 **CCV2**	**Fail** **Fail**	Re-analyze all samples since the last acceptable CCV. Perform maintenance and/or recalibration prior to re-analysis.

9.2.3.2 In the event that replicate CCVs are included in a sample injection sequence, the acceptance of the associated sample analyses must be properly evaluated and the evaluation process must be documented. Under no circumstances is it allowable to accept sample data based on the evaluation of only 1 of the CCV replicates.

9.2.3.3 Clear documentation of the evaluation of the CCV must be included in the analytical sequence run log (i.e., 'Pass' or 'Fail' in the comment section for each CCV). Additionally, clear documentation of corrective action taken in the event of CCV failures must be included in the run log (see table above for actions that need documentation).

Note: It is acceptable to use the CCV solution as a primer at the beginning of an analytical sequence however the injection should be documented as a 'Primer' in the sequence rather than as a CCV.

9.2.4. Calibration Acceptance Summary

9.2.4.1. Retention Time Windows: Retention time windows must be established to compensate for minor shifts in absolute retention times as a result of sample loading and normal chromatographic variability (for more detail on the following procedures refer to TestAmerica Corporate Technical Services Policy No. CA-T-P-005, "Policy for Determining RT Windows for GC/ECD Tests" and TestAmerica Corporate Technical Services Work Instruction No. CA-T-WI-010, "Further Guidance on the RT Window Policy No. CA-T-P-005").

9.2.4.1.1. Initial determination of RT windows.

9.2.4.1.1.1. The center of the retention time (RT) window shall be updated based on the middle level in the initial calibration of the first CCV in the daily sequence, whichever is most recent.

9.2.4.1.1.2. Use a default RT window of 0.01 minutes. At the end of the batch evaluate all CCVs and LCSs in the batch. If necessary, widen the window such that all analytes fall within the RT window. Reprocess the batch using the new RT windows.

9.2.4.1.1.3. Multiply the maximum deviation by 1.5. This is the retention time window, unless the result is less than 0.01 minutes, in which case the window is set at 0.01 minutes. For example, if

the maximum RT deviation for a specific analyte is 0.008 minutes, then the RT window is set at ±0.012 minutes.

NOTE: For the multi-component analytes, for example Aroclors, Toxaphene and Technical Chlordane, the maximum deviation must be evaluated for each of the 3 to 6 major peaks used for sample calculations.

9.2.4.1.1.4. Retention time windows for analytes of interest must not overlap for GC analysis.

9.2.4.1.2. Ongoing evaluation of retention time windows

9.2.4.1.2.1. Evaluate the retention time windows on an ongoing basis. The center of the RT window is updated on the first CCV of the day. All analytes for all subsequent CCVs, LCS and matrix spikes must fall within the retention time window (except as discussed below).

9.2.4.1.2.2. Matrix spike analytes may fall outside of the retention time window if there is a large non-target peak coeluting with the analyte in the matrix spike.

9.2.4.1.2.3. If any analytes fall outside of the retention time window in CCVs, LCS or matrix spikes (except as discussed above for matrix spikes) then the RT windows for those analytes shall be widened to the minimum degree required for the analyte to fall within the RT window. All samples in the batch shall be reprocessed with the new RT window, and the wider RT window shall remain in place for subsequent batches.

9.2.4.1.2.4. Retention time windows should be reliably narrower than ±0.03 minutes. If RT windows wider that this are necessary, the instrument should be evaluated and maintenance performed as needed. Subsequent to maintenance, RT windows shall be narrowed to the extent that is consistent with the data obtained.

9.2.4.2. **Initial Calibration Range:** Internal standard calibration is employed for this method. A response factor is calculated for each analyte at each calibration concentration.

Response factor = $((A_x)\ (C_{is}))/((A_{is})\ (C_x))$

Where:
A_x = area of the compound
C_x = Concentration of the compound
A_{is} = area of the internal standard
C_{is} = Concentration of the internal standard

9.2.4.2.1. Calculate the percent Relative Standard Deviation of the response factors for each compound at each level:

% RSD = (RF Standard Deviation/RF Mean) X 100

Where:
RF = Response Factor

9.2.4.2.2. If the % RSD across the 5 point range is <20% for any given compound the calibration can be assumed to be linear and the average response factor can be used to calculate concentrations of target compounds in samples.

9.2.4.2.3. If the % RSD is >20% for any given compound, a first order linear regression may be applied to the data to calculate the calibration curve and determine sample concentration. If this method is employed, the r^1 (Correlation Coefficient) value must be $\geq$ 0.990 for the calibration to be acceptable. Calibration is checked every 12 hours or after every twenty (20) samples, whichever comes first, by injecting a calibration verification standard for all single component pesticide standards.

9.2.4.2.4. Chlordane and Toxaphene Calibration: Chlordane and Toxaphene are multiple response pesticides and are calibrated with a minimum of 5 points as required (i.e, within 12 hours of either analyte being detected in a sample). Three to eight peaks are used for calculation of response factors and the same criteria detailed above is applied to determine acceptability of calibration.

9.2.4.2.5. Resolution: All single component analyte peaks must exhibit at least 80% chromatographic resolution. The analyst performs a visual check of the mid-level initial calibration standard and all subsequent calibration checks (ICV/CCV). If the resolution requirement is not met instrument maintenance should be performed followed by re-calibration. Percent resolution can be calculated when necessary as follows:

% Resolution =V/H x 100

Where:

V= Depth of the valley between two peaks. The depth of the valley is measured along a vertical line from the level of the apex of the shorter peak to the floor of the valley between the two peaks

H = Height of the shorter of the adjacent peaks

9.2.4.3. **Initial Calibration Verification (ICV):** An ICV will consist of a second source standard at or near the midpoint of the Initial Calibration Range analyzed at the frequency specified in Section 9.2.2.4. The calculated concentration of the ICV must be within ±20%D of the expected concentration. Should the %D exceed 20% the analyst should take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the ICV. If the %D still exceeds 20% after a single ICV reinjection, a new Initial Calibration Range must be analyzed. All single component analyte peaks must exhibit 80% resolution (see Section 9.2.4.2.5)

9.2.4.4. **Continuing Calibration Verification (CCV):** A CCV will consist of a second source standard at or near the midpoint of the Initial Calibration Range analyzed at the frequency specified in Section 9.2.3.. The calculated concentration of the CCV must be within ±20%D of the expected concentration. Should the %D exceed 20% the analyst should take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the CCV. If the %D still exceeds 20% after a single CCV reinjection, a new Initial Calibration Range must be analyzed. All single component analyte peaks must exhibit 80% resolution (see Section 9.2.4.2.5).

Step	***Stan□ar□s***	***Type***	***□ontrol □imit***	***□re□uency***
Method # 8081B				
GC System Performance Check	*Endrin/DDT, 250 ppb*	*Performance check*	*15% breakdown*	*At beginning of 12 hour clock and after system maintenance*
*Initial Calibration**	2.5, 50, 100, 250 and 500 ppb for single response, 1000 ppb for multi response	*Average response factor or 1st order linear regression*	*For average RF: <20%RSD all analytes. For linear regression: r ≥0.990*	*As required when ICV or CCV do not meet requirements*
ICV	*100 ppb*	*Average*	*± 20%D*	*Once after each initial calibration*

Step	*Standards*	*Type*	*Control Limit*	*Frequency*
CCV	*100 ppb*	*Average*	*± 20%D*	*Every 12 hrs or 20 samples, whichever is more frequent*

10. Procedure

10.1. Gas Chromatograph Operation

10.1.1. The sequence of events for GC analysis involves many steps. First the injection system and column performance and calibration must be verified. Maintenance operations are performed as needed. Then samples must be run on the instrument. Chromatograms and reports must be evaluated for content, integration and concentration. Re-runs and dilutions must be made based on the calibrations that were in effect at the time the sample was run. Lastly, a detailed analysis and calculations must be performed to determine the concentration of all the parameters for which the sample was analyzed.

10.1.2. General Operating Conditions

10.1.2.1. Injection System: A split/splitless injection port with electronic pressure control (EPC) is used. Thirty seconds after sample injection, the purge valve is turned on to facilitate the sweeping of any remaining residual solvent/sample from the injection port.

10.1.2.2. The EPC is used in the ramp pressure mode. The ramp pressure program is as follows:

Initial Pressure	InitialTime	Rate	Final Pressure	Hold
12 psi	2.5 min	7 psi/min	4 psi	1.50 min
		5 psi/min	9 psi	1.40 min
		9 psi/min	13 psi	2.00 min

10.1.2.3. For pesticide analysis the normal operating conditions of the injection port are as follows:

Injection port Temperature:	250^{o}C
Column flow:	12.3 ml/minute
Split vent flow:	5 ml/minute
EPC:	Pressure Ramp
Detector temperature	330C

10.1.2.4. In addition to the EPC, the injection port is also equipped with a siltek-coated glass double goose neck liner that contains a 1 cm glass wool plug. The plug of glass wool is located in the liner between the double goose neck.

10.1.2.5. This liner/glass wool combination provides many functions. The glass wool serves as a heat sink rapidly vaporizing solvent and samples resulting in higher response factors. The liner also protects the column head from accumulation of high boiling residuals and particulates.

10.1.2.6. The glass wool will be changed when changing the liner. The changing of the glass wool/liner is based upon the breakdown of an Endrin/DDT standard. This is covered in further detail in section 10.2.1.

10.1.2.7. Regular maintenance is performed on the injection port. When the glass wool/liner is changed, the septa also must be changed. Injection port, oven and detector temperatures are lowered to ambient prior to "cracking" the system. This is so as to introduce a minimum of damaging oxygen molecules into the system.

10.1.2.8. After the system has cooled, the old liner is removed. The injection port should be checked for particulate residues and cleaned as needed. A flashlight is usually required for this. After a new liner has been prepared it is placed into the injection port. A graphite seal is placed around the liner. The edges of the seal must be flat, not knife-edged, and free of nicks or burrs. If any of these conditions are not met, the graphite seal must be replaced as well. The graphite seal is critical to proper operation of the injection port. If in doubt, replace it.

10.1.2.9. The locking ring on the top of the injection port should be turned, with the wrench, about 1/8 turn past finger tight. The septum nut should never be tightened more than finger tight. After the injection port is reassembled, all column nuts inside the oven should be checked for leaks using Snoop (Supelco) or another suitable leak tester.

10.1.2.10. Once the signal from both detectors has stabilized, it is time to re-heat the zones. The zones should be heated in the order of detectors, oven and then injectors. This is to ensure that volatilized contaminants do not condense on the column or detector.

10.1.2.11. Oven: With the megabore columns installed, temperature programming is employed to achieve higher resolution of compounds and shorter run times than could be accomplished using isothermal methods.

10.1.2.11.1. A standard oven program for pesticide analysis is employed for all columns as follows:

Initial Temp	Hold Time1	Rate1	Temp1
160°C	0.62 min	30°/min	244°C
Hold Time2	**Rate 2**	**Final Temp**	**Final Time**
2.5min	21°/min	315°C	3.0min

10.1.2.12. Detectors: Detectors operate at 330°C and need to be supplied with 60 ml/min total flow. They are essentially maintenance free on a day-to-day basis. They are routinely baked out at 330°C to remove persistent contaminants. On occasion the detectors may be baked out at a higher temperature to remove contaminants with an extremely high boiling point (CAUTION: Do not exceed the maximum detector temperature of 380°C).

10.1.2.12.1. If the detectors are particularly contaminated, they must be sent to Agilent Technologies in Avondale, Pennsylvania for reconditioning. This should occur if the detector baseline is greater than 100 Hz. Detector reconditioning should be required at a maximum of biannually.

10.1.2.13. Chemstation: HP Chemstation software is used for automation of runs and data acquisition. The system is dedicated to a single GC and does not multitask. Therefore, data manipulation cannot be done while sample analysis is in progress. The data system acquires and stores all chromatographic data.

10.1.2.14. TestAmerica Chrom data processing software is used for the processing of the chromatography data files. Calibrations, verification standards and samples are processed and reviewed using this database. Chrom is integral to TestAmerica LIMS (TALS) which is used to generate all reports..

10.2. Analytical Sequence

10.2.1. The analytical sequence for performance checks, initial calibration, calibration verifications and sample analysis is described in the following sections.

10.2.2. Before calibration standards are analyzed the GC Performance Check Standard (see Section 9.2.2.1) must first be analyzed and evaluated to check the performance of the injection port and column with regard to catalytic active sites. The breakdown for both Endrin and DDT in the Performance Check Standard must be less than 15%. If the performance check fails this criteria system maintenance must be performed and the check successfully reanalyzed before proceeding with calibration.

10.2.3. A five point initial calibration (ICAL) is analyzed and evaluated for each of the 21 single response pesticides plus surrogates as described in Section 9.2.4.2. When needed a 5 point ICAL is also analyzed for Mirex.

10.2.4. Calibration for Technical Chlordane and Toxaphene: Chlordane and Toxaphene are multiple response pesticides containing at least 3-8 primary peaks each. A single point calibration check standard at a concentration of 1000ug/l is analyzed for Chlordane and Toxaphene. A full 5 point calibration range is analyzed for these compounds should they be detected in client samples..

10.2.5. A second source initial calibration verification (ICV) is analyzed and evaluated for each of the 21 single response pesticides as described in Section 9.2.4.3. When needed an ICV is also analyzed for Mirex. **NOTE**: The 12 hour time clock for Pesticides commences with the injection of the first Pesticide Calibration Standard or Verification.

10.2.6. GC Performance Check Standard is analyzed and the breakdown of Endrin/DDT is measured again before samples are analyzed and at the beginning of each subsequent 12 hour shift. If the breakdown check fails, then injection port/column maintenance is required

10.2.7. Client samples and QC samples may be analyzed after the analysis of the performance check. Sample analysis may proceed for up to 20 samples or 12 hours prior to analysis of another calibration verification (whichever is more frequent).

10.2.8. A Continuing Calibration Verification (CCV) for the 21 single response pesticides plus surrogates must be analyzed and evaluated every 12 hours or 20 samples (whichever is more frequent) as described in Section 9.2.4.4.. This is accomplished by running the midpoint calibration standard as a CCV (Pest Mix 100 ppb check standard and Mirex 100 ppb check standard; see Section 7.2.2.1). The calculated concentration for each compound in the CCV must be +/- 20 % of the expected concentration. Any samples analyzed after a failing CCV must be reanalyzed under a passing CCV. If, after performing instrument maintenance, the reanalysis of a CCV fails criteria, a new initial calibration range must be analyzed (see Section 9.2.4.2). Any data reported against a failing CCV must have a Non-Conformance Memo detailing the issue.

10.2.9. Analytical Sequence: The automation of GC runs is accomplished via the "SEQUENCE" macro of the Chemstation. The sequence file contains the name of Method file corresponding to the type of analysis to be performed, the range of samples to be run, and the number of injections per bottle. It is common practice to run the check standards, evaluate the instrument status, and then complete the Sample Table and Sequence File. If everything else is complete, the run is initiated using the START SEQUENCE soft-key of the SEQUENCE macro.

Example Analytical Sequence	
1. Hexane 2. Instrument Blank 3. Endrin/DDT Breakdown 4. Pesticide Mix 1 (2.5 ppb) 5. Pesticide Mix 2 (50 ppb) 6. Pesticide Mix 3 (100 ppb) 7. Pesticide Mix 4 (250 ppb) 8. Pesticide Mix 5 (500 ppb) 9. Chlordane (1000 ppb) 10. Toxaphene (1000 ppb) 11. Pesticide ICV (100 ppb)	12. Mirex 1 (10 ppb) (if needed) 13. Mirex 2 (20 ppb) (if needed) 14. Mirex 3 (100 ppb) (if needed) 15. Mirex 4 (250 ppb) (if needed) 16. Mirex 5 (500 ppb) (if needed) 17. Mirex ICV (100 ppb) (if needed) 18. Instrument Blank 19. Endrin/DDT Breakdown 20. Pest Mix 3 (100 ppb)…CCV 21. Mirex 3 (100 ppb) …CCV (if needed) 22. 20 or fewer samples or 12 hours 23. Instrument Blank 24. Endrin/DDT Breakdown 25. Pest Mix 3 (100 ppb)…CCV 26. Mirex 3 (100 ppb) …CCV (if needed) 27.20 or fewer samples or 12 hours

10.3. Dual Column Approach

10.3.1. The laboratory designates the rear column as the primary column and the front column as the secondary column. If the difference between the dual columns results in ≤40% RPD report the higher concentration.

10.3.2. The values are calculated from the chromatographic peaks that fall within the daily retention time windows. Retention times are updated using the retention times found in opening CCV for the most recent 12 hour period.

10.3.3. If the calculated values are greater than 40% RPD of each other, report the lower concentration regardless of whether that result is from the primary or secondary column. Report the result with a flag of P*. **Exception:** NJDEP DKQP protocols require reporting the higher concentration in all instances.

10.3.4. If the surrogates on one column are >40% RPD compared to the other column, this may be indicative of a bad injection or columnar blockage. The sample should be reanalyzed. If similar results are obtained following reanalysis, report the lower of the two numbers and describe the circumstances in the job summary and report case narrative.

10.3.5. If one of the columns fails CCV criteria (but the CCV is between 15%-40% greater than expected value), the sample results shall be reported from the compliant column. If the falls outside of acceptance criteria on the low side, reanalysis shall be performed.

10.3.6. If the CCV on one of the columns is more than 40% different from the correct value, it can be assumed that there has been significant drift on that column. The sample shall be reanalyzed against an acceptable calibration.

10.3.6.1. An exception to this requirement would be if the CCV recovery on one column fails on the high side and >40% RPD but the associated samples were non-detect for all target analytes on both columns. In this case the non-detect results may be reported from the compliant column.

10.3.7. In some cases where the sample chromatography is complex and has largely varying peaks concentrations, the chromatographic separation may not be sufficient on the 0.53mm ID columns. In this case a confirmatory analysis on an instrument with 0.32 ID columns may be required. The supplemental data produced using analysis on the 0.32mm ID 'microbore' column may minimize overlapping and baseline interference difficulties, and better resolves potential positive identifications. Use of this alternative chromatographic technique shall be noted in the job summary and report case narrative.

10.3.8. In summary, the flow chart in Attachment 1 presents a recommended rational approach to selecting the better number to report for dual column data. It shall be noted that these recommendations may be overridden by project specific requirements and that they cannot cover all eventualities. The complexity of some data set will require the final decision to be made utilizing the judgment of experienced analysts. In some cases further cleanup steps to remove interferences may be appropriate.

10.4. Extract Cleanups

10.4.1. Cleanup methods are dictated by the original sample matrix and the parameters being determined.

10.4.2. Cleanup of all water samples, if needed, is performed using Florisil (TestAmerica Edison SOP No. ED-ORP-020*: Florisil Cleanup for Pesticide/PCB Sample E□tracts*, SW846 Method 3620B, most current revision) and TBA sulfite (TestAmerica Edison SOP No. ED-ORP-021: *The Removal of Elemental Sulfur from Pesticide/PCB Sample E□tracts, S□ 8□□ Method □□□0B,* most current revision). Blanks must also undergo cleanup following the same procedures as samples.

10.4.3. Cleanup of all soil samples is conducted using Florisil (TestAmerica Edison SOP No. ED-ORP-020*: Florisil Cleanup for Pesticide/PCB Sample E□tracts*, SW846 Method 3620B, most current revision) and, if needed, TBA sulfite (TestAmerica Edison SOP No. ED-ORP-021: *The Removal of Elemental Sulfur from Pesticide/PCB Sample E□tracts, S□ 8□□ Method □□□0B,* most current revision). Blanks must also undergo cleanup following the same procedures as samples.

10.4.4. Check Florisil prior to use to assure quantitative recovery of target analytes. (see Section 4.3.2 above and TestAmerica Edison SOP No. ED-ORP-020*: Florisil Cleanup for Pesticide/PCB Sample E□tracts*, SW846 Method 3620B,

10.5. Documentation

10.5.1. Before the analysis sequence is initiated the GC Maintenance logbook must be filled out. It should contain the following information: date, injector temp, oven temp, detector temp, injector flow, signal A, signal B, analysts initials, and notes for any necessary repairs.

10.5.2. After samples have been run, each standard and sample must be entered into the Instrument Run Log. The Instrument Run Log should contain the following information: run date, data file name, vial position, sample number, initial volume/weight, final volume, dilution factor, method, job number, QA number, extraction date, lab prep batch, Chrom batch signature of analyst at the bottom of each page, lot numbers for standards used, and result of run (O.K., dilution, non-inject, etc.).

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Calculation of Sample Amounts (Internal Standard Procedure)

11.3.1.1 Aqueous Samples

$$\text{Concentration } (\mu g/L) = \frac{(As)(Cis)(D)}{(Ais)(RF)(Vs)\ (Vi)\ (1000)}$$

Where:

As	=	Area of the target analyte peak in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.

Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the internal standard peak
RF	=	Average response factor from the initial calibration.
Vs	=	Volume of sample extracted (ml)

The 1000 in the denominator represents the number of ul in 1 ml.

11.3.1.2 Solid Samples

$$\text{Concentration } (\mu g/kg) = \frac{(As)(Cis)(D)(Vt)}{(Ais)(RF)(Ws)\ (Vi)\ (1000)}$$

Where:

As	=	Area of the target analyte peak in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the internal standard peak
RF	=	Average response factor from the initial calibration.
Vt	=	Volume of concentrated extract (ul)
Ws	=	Weight of sample (g)

The 1000 in the denominator represents the number of ul in 1 ml.

11.4. Relative Response Factors

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

A_x = Area of target analyte peak
Ais = Area of internal standard peak
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

11.5. Percent Relative Standard Deviation (% RSD):

$$\%\ RSD = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

11.6. Percent Difference (% D):

$$\% D = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} X\ 100$$

Where: RRFc = RRF from continuing calibration

$\overline{RRF_i}$ = Mean RRF from current initial calibration

11.7. Percent Recovery (% R): Surrogates and Spikes

$$\text{Recovery (\%)} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}} \times 100$$

11.8. Dry Weight Correction: All solid samples must be corrected for dry weight using the following formula for dry weight determination.

$$DW = \frac{Gd}{Gw} \times 100$$

Where:

DW	=	Percent % Dry Weight
Gd	=	Dry weight of selected sample aliquot
Gw	=	Wet weight of selected sample aliquot

Multiply the DW value times the wet weight of the sample extracted.

NOTE: All dry weight corrections are made in TALS at the time the final report is prepared.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 20 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, *Training*, for the laboratory's training program.

13.0. Pollution Control

13.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

13.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage.

14.0. Waste Management

14.1. The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. 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. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.2. The following waste streams are generated as a result of this analysis:

- Expired Standards – The vials are collected in a 1 gallon polyethylene bucket. These vials are then transferred to an open top 55 gallon steel or polyethylene waste drum. These drums are transported to a waste facility for proper disposal.

- Auto sampler vials and expired standards: These vials are collected in satellite accumulation within the instrument laboratory. The vials are then placed into a 55 steel open top drum in the waste room. When the drums are full, the drum will be collected by the waste vendor for disposal. This waste is treated for incineration.

  Teris Profile Number: 50016652
  Onyx Profile WIP Number: 282493

- Mixed Solvent Waste: Mixed solvent waste is collected in a small beaker inside the bench top hood. This waste is then transferred into the satellite accumulation container in the Organic Prep. Lab. on a daily basis. This material is transferred into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.

  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

  Teris Profile Number (incineration): 50016710
  Onyx Profile Number: (stabilization) 402535

15.0. References / Cross-References

15.1. United States Environmental Protection Agency, "Method SW8000C: Determinative Chromatographic Separations,", Test Methods for Evaluating Solid Wastes, SW846, Revision 3, March 2003.

15.2. United States Environmental Protection Agency, "Method 8081B, Organochlorine Pesticide by Gas Chromatography", Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 2, February 2007.

15.3. TestAmerica Edison Document No. ED-QA-LQM, □*aboratory* □*uality Manual*, most current revision.

15.4. TestAmerica Edison SOP No. ED-ORP-014: *E□traction of Pesticides and PCBs in* □ *ater by Separatory Funnel, S□ 8□□ Method □510C,* most current revision.

15.5. TestAmerica Edison SOP No. ED-ORP-018*: E□traction of Pesticides/PCBs in Soil* □*sing □ow□□evel E□traction, S□ 8□□ Method □550B,* most current revision.

15.6. TestAmerica Edison SOP ED-ORP-044, *Procedure for the Microwave E□traction of Solids, S□ 8□□ □5□□*

15.7. TestAmerica Edison SOP No. ED-ORP-019*: □ aste Dilution for Pesticides and PCBs, S□ 8□□ Method □580A,* most current revision.

15.8. TestAmerica Edison SOP No. ED-ORP-020*: Florisil Cleanup for Pesticide/PCB Sample E□tracts*, SW846 Method 3620B, most current revision.

15.9. TestAmerica Edison SOP No. ED-ORP-021: *The Removal of Elemental Sulfur from Pesticide/PCB Sample E□tracts, S□ 8□□ Method □□□0B,* most current revision.

15.10. TestAmerica Edison SOP No. ED-GEN-022, *Training,* most current revision.

15.11. Test America Corporate Quality SOP No. CA-Q-S-001 (*Solvent □ Acid □ot Testing □ Approval*), most current revision.

15.12. TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*), most current revision.

15.13. TestAmerica Document CA-Q-W-008, 'Technical Guidance on the Use and Evaluation of Replicate Continuing Calibration Verification (CCV), most current version.

15.14. TestAmerica Corporate Quality SOP No. CA-Q-P-004, "Reporting Results for Methods that Require Second-Column Confirmation", most current revision.

15.15. TestAmerica Corporate Work Instruction No. CA-T-WI-008, "Minimum Requirements for Pesticide Analysis by SW846 Method 8081B", most current revision.

15.16. TestAmerica Corporate Policy CA-T-P-004, "Policy for Determining RT Windows for GC/ECD Tests", most current revision.

15.17. TestAmerica Corporate Technical Services Work Instruction No. CA-T-WI-010, "Further Guidance on the RT Window Policy No. CA-T-P-005", most current revision.

16.0. Method Modifications:

None

17.0. Attachments

Attachment 1: Dual Column Reporting Flowchart

18.0. Revision History

Revision 4, dated 09/13/2016:

- Section 9.2.4.1: completely rewrote this section to reflect requirements of TestAmerica Corporate Technical Services Policy No. CA-T-P-005, "Policy for Determining RT Windows for GC/ECD Tests" and TestAmerica Corporate Technical Services Work Instruction No. CA-T-WI-010
- Section 15 (References): added references to TestAmerica Corporate Technical Services Policy No. CA-T-P-005, "Policy for Determining RT Windows for GC/ECD

Tests" and TestAmerica Corporate Technical Services Work Instruction No. CA-T-WI-010, "Further Guidance on the RT Window Policy No. CA-T-P-005".

- Attachment 1: RT Windows for Single Analytes/Surrogates: DELETED and renamed subsequent attachments.

Revision 3, dated 01/05/2016:

- Section 2.2: expanded to include summary of internal standard calibration.
- Section 7.2.2: added 1-Bromo-2-nitrobenzene (internal standard) to the list of standards (Restek 32279). Components and concentration added to table.
- Added Section 7.2.2.4 which describes the preparation of the internal standard solution. All subsequent sections renumbered accordingly.
- Section 7.2.2.1.1: added footnote to ICAL standard prep table detailing requirement to spike each standard with internal standard solution.
- Section 7.2.2.4: describe internal standard spiking protocol (20 ul of 5.0 ug/ml solution into all standards, QC extracts and field sample extracts.
- Section 9.1: added internal standard to 'Sample QC' table.
- Section 9.1.4: added following minimum requirements for surrogate evaluation and reporting: a) Both surrogates must have reportable results that meet the acceptance criteria; b)reported surrogates must be from a column with a passing CCV; c)At least one surrogate must pass on any column from which target analytes are identified and reported. (per corp "Minimum Requirements" document).
- Section 9.1.5: added this section which describes acceptance criteria for internal standards (retention time and response).
- Section 9.2.3: Added the following sentence: "'Samples' here are defined as field samples and batch QC (MB, LCS, MS, MSD) and do not include CCVs, PEMs, instrument blanks, etc… ". Deleted the phrase 'and at the end of every sequence'. (both changes reflect 'Minimum Requirements for Pesticide Analysis' document).
- Added Section 9.2.4.1.3: "Updating absolute retention times: Update retention times with the retention times found in the opening CCV for that 12 hour period."
- Section 9.2.4.2: replaced 'external standard' with 'internal standard'. Added formula for calculation of response factors by internal standard method. Deleted text regarding calibration acceptance criteria from this section as it is duplicated in subsequent sections.
- Section 9.2.4.2.2: added phrase 'for any given compound.'
- Added section 9.2.4.2.5: describes resolution check.
- Sections 9.2.4.3 and 9.2.4.4: added statement concerning 80% resolution requirement.
- Section 9.2.4.4: changed 'ICV' to 'CCV' in last sentence.
- Section 10.1.2.13: Revised first sentence to read: ": HP Chemstation software is used for automation of runs and data acquisition."
- Section 10.2.2: corrected reference to Section 9.2.2.1 (was incorrectly listed as 7.2.2.1).
- Section 10.2.8: revised to clarify that CCV bracketing is not required and that only samples analyzed after a failing CCV must be reanalyzed (i.e., samples analyzed immediately prior to a failing CCV need not be reanalyzed).
- Section 10.3.2: added the following regarding updating retention times: "Retention times are updated using the retention times found in opening CCV for the most recent 12 hour period."

- Section 11: added formulas for calculation of sample concentrations as well as %RSD, %D, % Recovery, RRF and dry weight correction.
- Section 15: removed reference to SOP No. ED-ORP-016*: Automated So□hlet E□traction of Solid Samples □ Pesticides/PCBs, S□ 8□□ Method □5□1* as it is no longer is use.
- Section 15: added reference to TestAmerica Corporate Quality SOP No. CA-Q-P-004, "Reporting Results for Methods that Require Second-Column Confirmation"
- Section 15: added reference to TestAmerica Corporate Work Instruction No. CA-T-WI-008, "Minimum Requirements for Pesticide Analysis by SW846 Method 8081B".
- Section 15: added reference to TestAmerica Corporate Policy Memorandum No. CA-Q-QM-006, "Technical Guidelines for Analysis of Complex GC/ECD Chromatograms".

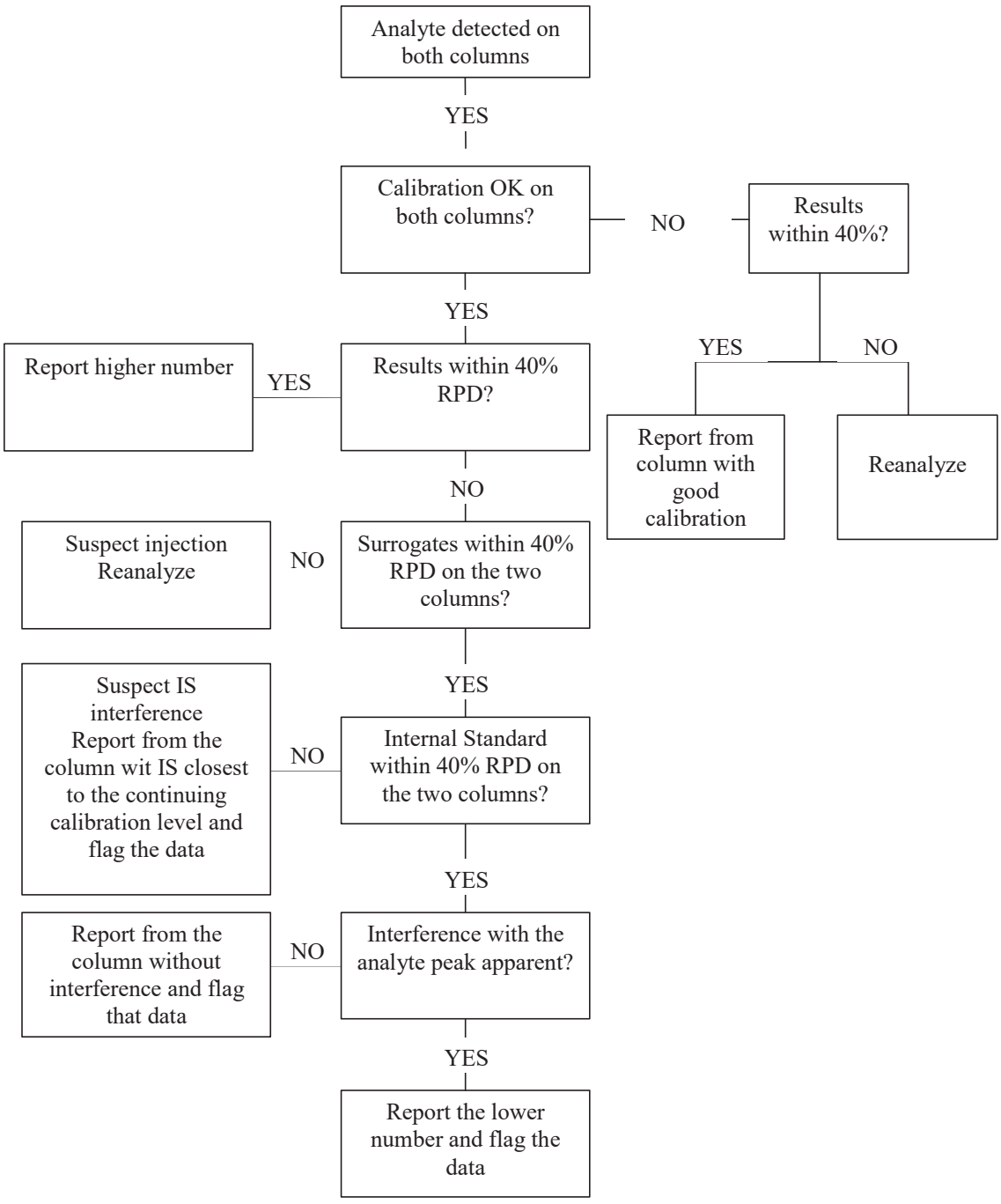
- Revision 2, dated 13 May 2015:
  - Section 1.1: updated RLs to reflect current lab practice.
  - Section 2.1 and throughout: revised initial aqueous/TCLP sample volumes to reflect current lab practice
  - Section 2.1.1: removed note describing option for Reduced Volume Extraction (RVE) as this is now standard lab practice.
  - Section 2.1.2 and throughout document: removed reference to prep by SW3541 as this method is no longer in use at Edison lab.
  - Section 4.3.2 and throughout: updated source of standards used for Florisil check.
  - Section 6.1.2.3.1: updated name of analytical GC columns currently in use.
  - Section 6.1.2.4.1: updated make-up gas to P-5 & Nitrogen.
  - Section 7.1.1: updated carrier gas to Hydrogen and make-up gas to P-5&Nitrogen.
  - Section 7.2.2 and throughout document: updated source and catalog numbers for analytical standards (Restek is now primary source of standards).
  - Section 7.2.2.1: updated standards prep instructions. Removed outdated references to standards and spiking mixes. Removed notes pertaining to RVE since this procedure is now standard and incorporated into instructions.
  - Section 7.2.2.10: added instructions for preparation of ICVs.
  - Section 8.1: adjusted water sample container from 1000ml to 250ml.
  - Section 9.2.2: removed references and instructions pertaining to the Pesticide Resolution Check (a CLP requirement we no longer perform).
  - Section 9.2.3.1: Added extensive discussion regarding the analysis of replicated CCVs.
  - Section 9.2.4.4: updated table to include new concentration for low standard (2.5 ppb).
  - Section 10.1.2.14 and throughout document: replaced references to Target with TestAmerica Chrom.
  - Section 10.2: made extensive revisions to discussion of the analytical sequence (removing duplicate entries and adjusting to reflect current procedures).
  - Section 10.3: added note concerning NJ DKQP requirements for reporting highest concentration of dual columns.
  - Section 15 (References): updated to include "Technical Guidance on the Use and Evaluation of Replicate Continuing Calibration Verification (CCV), most current version."
  - Added Sections 7.2.2.11 and 7.2.2.12 reflecting the addition and preparation of Internal standard

- Revision 1, dated 09 October 2012
  - Throughout: Revised LQM section number references to reflect the most current LQM revision.
  - Section 2.1.1: added description of reduced initial volume (125ml)/final volume (1ml) option.
  - Section 2.1.2: Added reference to TestAmerica Edison SOP ED-ORP-044, *Procedure for the Microwave E□traction of Solids, S□ 8□□ □5□□*
  - Section 7.2.2.1.1 added preparation of 5ppb standard for the reduced volume method.
  - Section 9.2.2.2 Added 5ppb standard for the reduced volume method
  - Section 9.2.4.4 Added 5 ppb standard to the initial calibration for the reduced volume method
  - Section 10.2.6 Added 5 ppb to the Analytical sequence for the low level method Section 15.0: Removed reference to TestAmerica Edison SOP EDS-GEN-019, *Organic Calculations,* most current revision.
  - Section 15.0: Added reference to TestAmerica Edison SOP ED-ORP-044, *Procedure for the Microwave E□traction of Solids, S□ 8□□ □5□□*

- Revision 0, dated 02/17/2011: New

ATTACHMENT 1

Dual Column Reporting Flowchart
(Note: NJDEP DKQP requires reporting the higher concentration in all instances)



Title: Analysis of Total and Amenable Cyanide in Water, Drinking water, Wastewater, Soil, and Wipes- Automated by Method EPA SW846 9012B; 335.4 and Standard Method 4500 CN^-C, G

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Jasmine Parillo 10/23/2017	Dan Helfrich 10/23/2017
Jasmine Parillo Date	Dan Helfrich Date
Department Manager	Health & Safety Manager /Coordinator
Carl Armbruster 10/23/2017	10/23/2017
Carl Armbruster Date	Mark Acierno Date
Quality Assurance Manager	Laboratory Director

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 Methods EPA 335.4, SW846 9012B, Lachat 10-204-00-1-A and SM 4500 CN^-C, G are described in this SOP. The SOP is applicable to water, drinking water, wastewater, soil, waste, and wipe samples requiring cyanide determination. The method detects inorganic cyanides that are present as either soluble salts or complexes. It is used to determine values for both total cyanide, free cyanide and cyanide amenable to chlorination.

1.1.2 The automated method has a detector that is sensitive to approximately to 0.005 mg CN-/L. The laboratory's reporting limit are as follows:

Analyte	Water Reporting Limit (mg/L)	Soil Reporting Limit (mg/Kg) MIDI Distillation	Soil Reporting Limit (mg/Kg) Micro Distillation	Wipes Reporting limit (mg/wipe)
Cyanide	**0.01**	**0.50**	**0.24**	**0.0005**

1.1.3 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

Cyanide is extracted by means of reflux distillation (EASY-Dist distillation or MICRO DIST apparatus). Sample is distilled in a strongly acidic solution which breaks down cyanide complexes and converts the cyanide to hydrocyanic acid (HCN). HCN is absorbed into a sodium hydroxide absorber solution and the cyanide ion in the absorbing solution is determined by automated UV colorimetry at 570nm.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 Interferences can be reduced or eliminated by using the distillation procedure.

4.2 The presence of sulfide in the sample matrix adversely effects the determination of total cyanide. Samples that contain hydrogen sulfide, metals sulfides or other compounds that may produce hydrogen sulfide during distillation should be treated.

4.2.1 Place a drop of the sample on lead acetate test paper (which has been pre-moistened with pH 4 acetate buffer solution) to detect the presence of sulfides. If sulfides are present (test strip turns black), the sample volume required for the cyanide determination should be increased by 25 milliliters (ml).

4.2.2 The total volume of sample should be treated with powdered cadmium carbonate or lead carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide.

4.2.3 Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper

4.2.4 Filter the solution through a dry filter paper into a dry beaker, and from the filtrate measure the sample to be used for analysis.

NOTE: Avoid a large excess of cadmium carbonate and a long contact time in order to minimize a loss of cyanide on the precipitated material.

4.3 Oxidizing agents such as chlorine decompose most cyanides.

4.3.1 To determine if oxidizing agents are present test a drop of sample with potassium iodine-starch paper. If a blue color appears that indicates the need for treatment. Add ascorbic acid, a few crystals at a time, to the sample until a drop of sample produces no blue color on the indicator paper. Add an additional 0.6g of ascorbic acid for each liter of sample volume.

4.4 Nitrates and nitrites may give high biased results. During distillation they form nitrous acid, which will react with some organic compounds to form oximes. These compounds once formed will decompose to generate HCN. They are interferences when present above 10 mg/L with other specific organic compounds.

4.4.1 Nitrate and nitrite interferences are eliminated by treatment with sulfamic acid just before distillation.

4.5 Thiocyanate is reported to be an interference when present at very high levels. Levels of 10 mg/L were not found to interfere.

4.6 Fatty acids, detergents, surfactants may cause foaming during the distillation process, making the endpoint difficult to detect. If this occurs, samples can be acidified with acetic acid (1.6M) to pH 6.0-7.0.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the

assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Ensure cooling water is turned on to the distillation unit. Otherwise the samples may boil over and come into contact with the heating plates. Potassium Cyanide will give off Hydrogen Cyanide (HCN) gas if combined with strong acids. Inhalation of CN gas can cause irritation, dizziness, nausea, unconsciousness and potentially death.

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 MSDS 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 MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Phosphoric Acid	Corrosive	1 Mg/M3 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.
Sodium Hydroxide	Corrosive	2 Mg/M3-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.
Pyridine	Flammable Irritant	5 ppm-TWA	Inhalation causes severe irritation to the respiratory tract. Symptoms of overexposure include headache, dizziness, nausea, and shortness of breath. Causes severe irritation possibly burns, to the skin. Symptoms include redness and severe pain. Absorption through the skin may occur, resulting in toxic effects similar to inhalation. May act as a photosensitizer. Vapors cause eye irritation. Splashes cause severe irritation, possible corneal burns and eye damage.
Barbituric Acid	Irritant	Not established	Limited information. Inhalation may irritate respiratory tract. Causes skin and eye irritation. Should be treated as potential health hazard; do not ingest.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	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.
Potassium Hydroxide	Poison Corrosive Reactive	2 mg/m3 – Ceiling	Inhalation symptoms may include coughing, sneezing, damage to the nasal or respiratory tract. High concentrations can cause lung damage. Swallowing may cause severe burns of mouth, throat and stomach. Other symptoms may include vomiting, diarrhea. Severe scarring of tissue and death may result. Contact with skin can cause irritation or severe burns and scarring. Causes irritation of eyes with tearing, redness, swelling. Greater exposures cause severe burns with possible blindness.
Potassium Cyanide	Poison Corrosive	5 Mg/M3 TWA as CN	This material will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death. Corrosive to the respiratory tract. May cause headache, weakness, dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heartbeat, unconsciousness, convulsions, coma and death. Solutions are corrosive to the skin and eyes, and may cause deep ulcers, which heal slowly. May be absorbed through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred vision, and eye damage.
Sodium Cyanide	Poison Corrosive	5 mg/m3 TWA as CN (skin)	This material will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death. Corrosive to the respiratory tract. May cause headache, weakness, dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heartbeat, unconsciousness, convulsions, coma and death. Solutions are corrosive to the skin and eyes, and may cause deep ulcers, which heal slowly. May be absorbed through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred vision, and eye damage.
Silver Nitrate	Poison Corrosive Oxidizer	0.01mg/m3 (TWA) for silver metal dust and fume as Ag	Inhalation symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting. May be absorbed into the body following inhalation. Swallowing can cause severe burns of the mouth, throat and stomach. Can cause sore throat, vomiting, and diarrhea. Poison. Symptoms include pain and burning in the mouth, blackening of the skin and mucous membranes, throat, and abdomen, salivation, vomiting of black material, diarrhea, collapse, shock, coma and death. Skin contact can cause redness, pain and severe burns. Eye contact can cause blurred vision, redness, pain, severe tissue burns and eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

- EASY- Dist distillation apparatus with all associated glassware (Westco Scientific Instruments)
- MICRO DIST distillation apparatus with sample distillation tube press (Hach).
- Lachat 8000 and 8500 series: Flow injection analysis equipment-designed to deliver and react sample and reagents in the required order and ratios. Lachat 8000 series
  - Multichannel proportioning pump
  - Reaction manifold
  - Colorimetric detector
  - Data system
  - Heating Unit

6.2. Supplies

- Lachat Micro Distillation Tubing, p/n A17117 (collector tubes, caps, membranes, and sample tubes) or equivalent, disposable
- Various Class A volumetric flask and pipettors
- Micro-porous boiling chips
- 120 ml snap seal sample cups
- 24/40 clips
- pH-indicator strips (pH 0-14)
- Potassium Iodine-starch paper
- Lead-Acetate paper (pre-moistened with pH 4 acetate buffer solution)
- Analytical balance
- Vacuum pump
- Specimen cups
- Transfer pipettes
- Graduated cylinder (class A)

- Micro-buret

7. Reagents and Standards

7.1. Reagents

7.1.1. Deionized water-18 megohm reagent grade Type II water.

7.1.2. Acetone – store at room temperature; for stability information refer to manufacturer's instructions.

7.1.3. Rhodanine indicator - p-dimethyl-aminobenzalrhodanine: Place 20 mg of p-dimethyl-aminobenzalrhodanine in a 100ml volumetric flask and dilute to the mark with acetone. Store at room temperature. Stable for six months.

7.1.4. Acetate Buffer - Dissolve 82.0 gram of $NaC_2H_3O_2$ x $3H_2O$ in 100ml DI water. Add sufficient glacial acetic acid and adjust pH to 4.5 (approx. 10 drops). This solution is stable for 6 months, store at 4ºC.

7.1.5. Cadmium Carbonate (powder) ACS reagent –store at room temperature; discard after 5 years from date receipt or earlier if necessary.

7.1.6. Reagents for cyanides amenable to chlorination.

7.1.6.1. Calcium hypochlorite solution, (0.35M), $Ca(OCl)_2$. Dissolve 5 g of calcium hypochlorite in 100 ml of deionized water. Solution is stable for 6 months, store at room temperature.

7.1.6.2. 0.25 N NaOH. Dissolve 10.0 g NaOH into 1 L of deionized water. Dilute to mark with deionized water and invert to mix. Solution is stable for 6 months, store at room temperature.

7.1.6.3. Ascorbic acid, $C_6H_8O_6$ - store at room temperature, for stability information, refer to manufacturer's instructions.

7.1.7. Reagents for cyanide distillation.

7.1.7.1. 0.25 M NaOH. See section 7.1.6.2

7.1.7.2. Sulfamic Acid Solution: Dissolve 9.6 g. H_2NSO_3H into 100ml of deionized water. Solution is stable for 6 months, store at room temperature.

7.1.7.3. Micro Distillation Releasing Agent (7.11 M sulfuric acid/ 0.79M magnesium chloride): In a hood, place a 500ml beaker on a top-loading balance and tare. Then add 110.8g DI water. Then add and dissolve completely 32.2g magnesium chloride hexahydrate ($MgCl_2$ x $6H_2O$) in this water. Slowly add 139g concentrated sulfuric acid a little at a time, swirling and allowing to cool. Solution is stable for 6 months, store at room temperature.

7.1.7.4. Bismuth nitrate Solution: Dissolve 3 g of $Bi(NO)_3 \bullet 5H_2O$ in 10 mls DEIONIZED WATER , add 25 mls glacial acetic acid while stirring and dilute to a 100 ml final volume.

7.1.8. Reagents for automated colorimetric determination.

7.1.8.1. Pyridine-Barbituric acid Reagent: In a fume hood, place 15.0 g barbituric acid in a 1 L beaker and add 100 ml DEIONIZED WATER, rinsing down the sides of the beaker to wet the barbituric. Add 75 ml pyridine (C_5H_5N) while stirring and mix until barbituric acid dissolves. Add 15 ml conc. HCl (12 M HCl) and mix. Transfer to a 1 L volumetric flask, dilute to mark and invert to mix. Prepare weekly, store solution in amber bottle and at room temperature.

7.1.8.2. Chloramine-T: Dissolve 2.0g chloramine-T hydrate in 500 ml deionized water. Prepare fresh daily. Store at room temperature.

7.1.8.3. Phosphate buffer: In a 1 L volumetric flask, dissolve 97.0 g potassium phosphate, monobasic, anhydrous, (KH_2PO_4) in approximately 800 ml of DEIONIZED WATER. Dilute to mark and invert to mix. Solution is stable for one month. Store at room temperature.

7.1.8.4. 0.25 M NaOH (carrier) See section 7.1.6.2.

7.2. Standards

7.2.1. Standard 0.0192N Silver nitrate solution (1 ml = 1 mg CN): Prepared by weighing 3.2647 g of dried $AgNO_3$, dissolved in deionized water and dilute to 1000ml. Solution is stable for 6 months or when QC check is outside the acceptable limits, whichever comes first, store at 4ºC.

7.2.2. Primary (Simple) Cyanide stock solution, 1000 mg/L: This standard is used for the preparation of calibration standards (Sec 10.2). Purchased commercially from Accustandard (catalog number WC-CN-10X-5). Follow manufacturer's instructions for storage and stability. Standardize the solution against 0.0192N silver nitrate solution quarterly.

If prepared in house, prepare as follows: Primary Cyanide stock solution, 1000 mg/L: Dissolve 2.51 gm of KCN and 2.0 gm of KOH in 900 ml of deionized water. Standardize the solution against 0.0192N silver nitrate solution weekly and dilute to the appropriate concentration so that 1ml=1mg CN. Solution is stable for 6 months, store at 4ºC. Standardize the solution as follows::

- Take 5.0 ml of the primary stock cyanide solution (Sec. 7.2.2) and dilute it to 50ml using the 0.25 M NaOH. Add 10-12 drops of the rhodanine indicator (Sec. 7.1.3) to produce a bright yellow color.

- Using the 10 ml micro buret, titrate the stock cyanide solution from a

bright yellow color to a salmon hue endpoint. Record the volume of silver nitrate solution used for the titration in the Standardization logbook.

- Titrate a blank using 50mls of 0.25N NaOH, 10-12 drops of rhodanine indicator. Record the volume of silver nitrate solution used for the titration in the Standardization logbook.

- Calculate CN concentration as follows:

Based on the following: $Ag^{+} + 2\ CN \rightarrow [Ag(CN)_2]^{-}$

$$CN^{-}(mg/L) = \frac{A-B}{C} \times N \times \frac{2\ mol\ CN}{1\ eq\ AgNO_3} \times \frac{26.02g\ CN}{1\ mol\ CN} \times \frac{1000mg}{1\ g}$$

Where:
A= ml $AgNO_3$ for titration of sample
B=ml $AgNO_3$ for titration of blank
C= ml or g of sample
N= normality of $AgNO_3$

- Record the preparation in the reagent TALS. Record final concentration in the Standardization logbook.

- **Note** [illegible]

7.2.3. Secondary (Simple) Cyanide Stock Solution, 1000mg/L: The secondary cyanide stock solution must come from a source different from the primary cyanide stock solution (ERA, cat# 997 or Accustandard, cat# WC-CN-10X-5). Follow manufacturer's instructions for storage and stability. Standardize the solution against 0.0192N silver nitrate solution quarterly. This standard is used for the preparation of ICV and CCV standards (Sec 9.2.1 and 9.2.2).

If prepared in house, prepare as follows: Dissolve 2.51 gm of KCN and 2.0 gm of KOH in 900 ml of deionized water. Note: the secondary cyanide stock solution must be prepared from a different source than the primary cyanide stock solution. Standardize the solution with 0.0192N silver nitrate solution weekly and dilute to the appropriate concentration so that 1ml=1mg CN. The solution is stable for 6 months, store at 4ºC. Follow the standardization procedure in 7.2.2.

Note: [illegible]

[redacted]

7.2.4. Primary Working Solution, 6 mg/L: Take 0.6 mL of the Primary Cyanide Stock Solution (Sec. 7.2.2) and dilute to a final volume of 100ml with 0.25N NaOH. Prepare fresh daily.

7.2.5. Secondary Working Solution, 6 mg/L: Take 0.6 mL of the Secondary Cyanide Stock Solution (Sec. 7.2.3) and dilute to a final volume of 100ml with 0.25N NaOH. Prepare fresh daily.

7.2.5.1. Secondary Working Solution for Easy-Dist: 10mg/L: Take 1.0 mL of the Secondary Cyanide Stock Solution (Sec. 7.2.3) and dilute to a final volume of 100ml with 0.25N NaOH. Prepare fresh daily.

7.2.6. Complex Cyanide stock solution (Potassium Ferrocyanide Trihydrate in 0.5 NAOH), 1000 mg/L: Purchased commercially from ERA (catalog number 998). Follow manufacturer's instructions for storage and stability. This standard is used for the preparation of QC samples which are distilled prior to analysis (LCS and MS/MSD).

7.2.7. Complex Cyanide Working solution, 6 mg/L: Take 0.6 mL of the Complex Cyanide stock solution (Sec 7.2.6) and dilute to a final volume of 100 ml with 0.25N NaOH. Prepare fresh daily.

7.2.7.1. Complex Cyanide Working solution for Easy-Dist, 10 mg/L: Take 1.0 mL of the Complex Cyanide stock solution (Sec 7.2.6) and dilute to a final volume of 100 ml with 0.25N NaOH. Prepare fresh daily.

7.2.8. Soil Standard Reference Materia (LCSSRM): Purchased commercially from ERA or Phenova. Follow manufacturer's instructions for storage and stability. This standard is used for the preparation of QC samples which are distilled prior to analysis.

8. Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference

Waters	Glass or plastic	200 ml	NaOH, pH > 12; Cool 4 ± 2°C	14 Days	Method 335.4; SW846 Method 9012B
Soils	Glass	5.0 grams	Cool 4 ± 2°C	14 Days	Method 335.4; SW846 Method 9012B
Wipes	Glass	1 wipe	Cool 4 ± 2°C	14 Days	SW846 Meth od 9012B

[1] Inclusive of digestion and analysis.

9. Quality Control

9.1. Sample QC: Waters, soils, and wipes are separated into different QC batches.

9.1.1. Method Blank (MB): 0.25N NaOH is used for the blank and is carried through the entire sample preparation and analytical process. Add 6ml of 0.25 N NaOH to a disposable distillation sample tube. Results must be less than the reporting limit, or the batch must be redistilled and reanalyzed. One method blank must be analyzed with each set up of samples or every 20 samples, whichever is more frequent. Note: For *wipe* sample batches, the method blank consists of a blank wipe plus 50 ml of 0.25N NaOH.

9.1.2. Laboratory Control Sample (LCS): Prepare the LCS according to the distillation method. Prepare fresh daily.

9.1.2.1. **MICRO DIST:** For every aqueous batch, a LCS (Laboratory Control Sample) must be analyzed with each distillation batch or every 20 samples, whichever is more frequent. For every soil batch, a soil LCSSRM and a LCS (Laboratory Control Sample) must be analyzed at a frequency of 1 per 20 samples.

- **MICRO DIST***: Laboratory Control Sample (LCS),* 0.10 mg/L: Add 0.1 mL of the 6 mg/L Complex CN working solution (Sec. 7.2.7) and 5mL of 0.25 NaOH. The results must be within the control limits; if not, the batch must be redistilled and reanalyzed. Control limits: for 9012B ±15% of the true value; for 335.4 ±10% of the true value; and for SM4500CN ±10% of the true value.

- **MICRO DIST**: *Soil - Laboratory Control Sample Standard Reference Material (LCSSRM),* weigh out 0.25g of the standard reference material, add 5.0 ml DI water to the disposable distillation tube. The results must be within vendor specified QC limits or the batch must be redistilled and reanalyzed.

9.1.2.2. **EASY DIST:** Two LCS (Laboratory Control Sample-Low) and one HLCS (Laboratory Control Sample –High) must be run with each distillation batch or every 20 samples whichever is more frequent. .

For every soil batch, a soil LCSSRM must be analyzed at a frequency of 1 per 20 samples.

- **EASY DIST**: *Laboratory Control Sample-Low (LLCS),* 0.100 mg/L**:** Add 50ml of 0.25N NaOH to the boiling tube then add 0.50 ml of the 10 mg/L Complex CN working solution (Sec. 7.2.7). The results must be within 90-110% of the true value or the batch must be redistilled and reanalyzed.
- **EASY DIST***Laboratory Control Sample i (LCS)**:* Add 50ml of 0.25N NaOH to the boiling tube then add 1.0 ml of the 10 mg/L Complex CN working solution (Sec. 7.2.7). The results must be within 90-110% of the true value or the batch must be redistilled and reanalyzed.
- **EASY DIST**, *Soil Laboratory Control Sample Standard Reference Material (LCSSRM)*- weigh out 1.0gm of the standard reference material into the boiling tube and add 50 mL deionized water. The results must be within vendor specified QC limits or the batch must be redistilled and reanalyzed.

- Note: For *wipe* batches, prepare an LCS/LCSD at 0.2 mg/L. Add a blank wipe to the boiling tube and follow the preparation procedures above.

9.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD) One MS/MSD pair must be analyzed for every 10 samples.

- Aqueous samples (drinking water and wastewater): **MICRO DIST, 0.2 mg/L:** two portions of the same sample (matrix spike and matrix spike duplicate), each 6 ml, are added to separate disposable distillation tubes then add 0.2 mL of the 6 mg/L Complex CN primary working solution (Sec 7.2.7) into each sample. **EASY DIST, 0.2 mg/l:** two portions of the same sample (matrix spike and matrix spike duplicate), each 50 ml, are added to separate boiling tubes then add 1.0 ml of the 10 mg/L Complex CN primary working solution (Sec 7.2.7) into each sample; the spiking concentration is 0.20mg/L. The results must be within 90-110 %..

- Soil samples: **MICRO DIST, 4.8 mg/Kg:** add 0.25g of the sample to two separate disposable distillation tubes (matrix spike and matrix spike duplicate) and add 5.0 ml of deionized water. Spike these samples with 0.2 mL of the 6 mg/L Complex CN primary working solution (Sec. 7.2.7). **EASY DIST, 10 mg/kg**: add 1g of the sample to two separate boiling tubes (matrix spike and matrix spike duplicate) and add 50 ml of deionized water. Spike these samples with 1.0 ml of the 10 mg/L Complex CN primary working solution (Sec 7.2.7). The results must be within laboratory generated limits.

- Wipe samples: matrix spikes are not required.

- Amenable to chlorination: MS/MSD – For aqueous samples, measure 50 ml of sample for both the MS and MSD and add to a 100 ml beaker.

Add 1.0 ml of the 10 mg/L Complex CN primary working solution (Sec 7.2.7) into each sample; the spiking concentration is 0.20mg/L. For soils samples, weigh out 1g of sample and add 50ml of 0.25N NaOH then follow spiking procedure for aqueous samples. The spike must be added before the chlorination step begins. The form of cyanide used for spiking is completely amenable to decomposition with chlorine and therefore, the recovery of the spikes is expected to be 0%. If cyanide remains following the chlorination step, it is evident that the chlorination was not complete.

9.2. Instrument QC

9.2.1. Initial Calibration Verification (ICV), 0.20 ppm: The ICV must be analyzed immediately after an acceptable calibration and before any samples are analyzed. The determined concentration must be within the ICV control limits; if not the analysis should be terminated until the source of the problem is identified and corrected. ICV control limits for 9012B is ±15% of the true value; for 335.4 ±10% of the true value and for SM4500CN ±15% of the true value.

- **Micro Dist:** The ICV will be prepared from the secondary Simple CN source (Sec 7.2.3). See Sec 10.2.3.3 for the ICV preparation instructions by Micro Dist.

- **Easy-Dist**: The ICV is prepared by taking 2ml of the 10ppm secondary working solution (Sec. 7.2.5) and diluted up to a final volume of 100ml with 0.25N NaOH solution. .

9.2.2. Continuing Calibration Verification (CCV), 0.20 ppm: A CCV is analyzed after every 10 samples and at the end of the sample run. The CCV will be prepared from the same source as the ICAL. CCV control limits for 9012B is ±15% of the true value; for 335.4 ±10% of the true value and for SM4500CN ±10% of the true value. If the result exceeds the limit, the analysis should be terminated until the source of the problem is identified and corrected and all samples following the last acceptable CCV must be reanalyzed.

9.2.3. Initial Calibration Blank (ICB): An ICB is analyzed immediately after the ICV to verify calibration and acceptable instrument performance. The results must be less than the reporting limit. Use 0.25N NaOH for the ICB.

9.2.4. Continuing Calibration Blank (CCB): A CCB must be analyzed after every 10 samples and at the end of the sample run. The results must be less than the reporting limit. If the CCB result exceeds the reporting limit, all samples following the last acceptable CCB must be reanalyzed. Use 0.25N NaOH for the CCB.

10. Procedure

10.1. Sample Preparation

10.1.1. Each sample matrix must be spot tested for the presence of interfering ions.

10.1.1.1. Check that pH of each sample is >12 using the pH indicator strip.

10.1.1.2. Check for the presence of chlorine using KI-Starch paper. See section 4.3.1.

10.1.1.3. Check for the presence of sulfide using lead acetate paper (pre-moistened with pH4 acetate buffer solution). See section 4.2.1. – 4.2.4. Method 9012B: If positive add bismuth nitrate to the sample. At the same time add bismuth nitrate to all standards before distillation, see Section 10.2.3.

10.1.2. Procedure for the pretreatment of cyanides amenable to chlorination.

10.1.2.1. Two aliquots of the sample are used to determine cyanides amenable to chlorination. For aqueous samples, measure 50 ml of sample to a 100 ml beaker. For soils samples, weigh out 1g of sample and add 50ml of 0.25N NaOH. Add calcium hypochlorite solution (Section 7.1.6.1) drop-wise while agitating the mixture using a magnetic stir plate. Maintain the pH at between 11 and 12 with sodium hydroxide solution (Section 7.1.6.2)

Note: A MS/MSD must be performed for samples requiring cyanide amenable to chlorination as a negative quality control; see Sec 9.1.3.

CAUTION: Toxic cyanogen chloride is the initial reaction product during an alkaline chlorination. This reaction should take place in a fume hood.

10.1.2.2. Residual chlorine must be maintained in the solution for 1 hour. Monitor pH with pH test paper and chlorine with KI-starch paper. A distinct blue color on the paper indicates sufficient chlorine in solution.

10.1.2.3. Following one hour, add ascorbic acid crystals on 0.5 g portions until a test with the KI-Starch paper shows no residual chlorine. Add an excess of 0.5 g of ascorbic acid crystals to insure that no residual chlorine is present as a reducing agent.

10.1.2.4. Test for total cyanide in both a chlorinated and unchlorinated aliquot of the sample. Cyanide amenable to chlorination is determined by the difference between the total cyanide result of the un-chlorinated aliquot and the chlorinated aliquot.

10.1.3. EASY-Dist Distillation Procedure

10.1.3.1. For *a□□eo□□* samples, add 50 ml sample into the boiling tube. For *□olid* samples, weigh 1.0 gram of sample into the boiling tube and add 50 ml deionized water. For *wipe* samples, add 1 wipe into the boiling tube, add 50 ml of deionized water and record 1 wipe as the initial amount in TALS prep batch worksheet.

10.1.3.2. Add a few micro-porous boiling chips to the boiling tube.

THE HEATER BLOCK TEMPERATURE MUST BE 70℃ OR LOWER TO LOAD BOILING TUBES. DO NOT PRE-HEAT BLOCK ABOVE 70℃. Lo ading tubes containing sample into a hot block will cause super-heating of samples and a potential boil-over of tube contents.

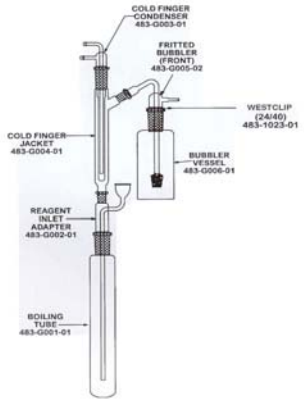


Figure 1: EASY-Dist Cyanide Distillation Apparatus

10.1.3.3. Add 50 mls of 0.25 M NaOH in bubbler vessel. Place the fritted bubbler (front) in the bubbler vessel and seal the joint with a size 24/40 clip. This is the NaOH absorber.

10.1.3.4. Connect the reagent inlet adapter to the boiling tube. Connect the cold finger jacket to the reagent inlet adapter. Attach the cold finger condenser to the cold finger jacket.

10.1.3.5. Attach the sealed NaOH absorber to the cold finger condenser. Turn the condenser water on.

10.1.3.6. Put approximately 500 mls of NaOH in the excess cyanide trap.

10.1.3.7. Attach the black vacuum line from the EASY-Dist gas manifold port to the inlet of the NaOH absorber.

10.1.3.8. Turn vacuum on and adjust the individual air flow rate using the control knob at each manifold port. Adjust the flow such that two to five bubbles per second are exiting the base of the reagent inlet adapter. When properly adjusted, the NaOH absorber solution will be actively foaming without bubbling over into the vacuum line.

10.1.3.9. If the sample is sludgy or the analyst suspects sulfide, add 5 mls bismuth nitrate solution to sample before adding the sulfamic acid.

10.1.3.10. Add 5 mls sulfamic acid solution to the boiling tube through the reagent inlet adapter and let mix for three minutes.

10.1.3.11. Add 5 mls 1:1 H_2SO_4 to the boiling tube through the reagent inlet adapter. Rinse the acid down the inlet with small volume of distilled water and allow content to mix for three minutes.

10.1.3.12. Add 2 mls magnesium chloride solution to the boiling tube through the reagent inlet adapter and rinse down with small volume of distilled water.

10.1.3.13. Turn on heating block. Heater block is set at 2.0 hour. Temperature should reach 125 +/- 3℃ in approx. 20 minutes. The 1.5 hour reflux starts after the programmed temperature set point is reached. Heating block will automatically shut off after 1.5 hour.

10.1.3.14. Periodically monitor the vacuum and heating to avoid excessive bubbling in the bubbler vessel. Adequate heating is indicated by a constant reflux of condensed vapor off of the condenser. Discontinue heating but maintain vacuum flow. Cool glassware apparatus for 15 minutes.

10.1.3.15. Turn off vacuum pump. Remove bubbler vessel and transfer its contents into a 125ml snap seal cups. DO NOT DILUTE.

10.1.3.16. The solution is now ready for semi-automated colorimetry.

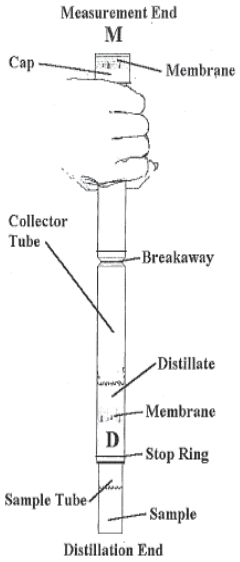
10.1.4. MICRO DIST Distillation Procedure

10.1.4.1. Set the controller to 120 deg C. Allow the block to warm up which takes approximately 40 minutes.

10.1.4.2. With the ‘M’ end or ‘measuring end’ up, place the collector tubes for the samples into the tube rack. Add 2 mL of 0.25N NaOH solution and add a membrane and cap. Label the top of the tube with the sample ID. Up to 21 samples tubes can fit on one block.

10.1.4.3. In the ‘D’ end (or ‘distillation end’), pipet 6.0ml of aqueous sample into each sample tube or weigh out 0.25 to 0.30 grams of sample plus 5.0 ml of DI water for soil samples. Add 0.25ml of the sulfamic acid solution and 0.75ml of the releasing agent to all tubes. Label the tube with the sample ID.

***NOTE**: Homogenize soil samples properly prior to taking an aliquot for analysis.



10.1.4.4. Immediately push the 'D' end or 'distillation or discarded' end of the collector tube over the open end of each sample tube to start the seal.

10.1.4.5. Place the assembly into the press, putting the sample tube through the hole in the white base. Before pressing, the user should hold the collector tube firmly at the breakaway point to ensure the tube does not shift during the pressing procedure.

10.1.4.6. Press down on the press handle until the stop ring on the sample tube hits the 'D' end of the collector tube.

10.1.4.7. Put on heat resistant gloves and push the sample tube and 'D' end of each tube into the preheated block so that the collector tube stop ring touches the block.

10.1.4.8. Set timer for 30 minutes. Do not pull the tubes out before the 30 minutes to check them. This will cause the sample to suck-back.

10.1.4.9. After the 30 minutes have passed, put on heat resistant gloves. Remove the first tube from the block and immediately pull off its sample tube using a downward, twisting motion. Important: Remove the sample tube within 4 seconds of removing it from the block or suck-back of the sample will occur, which may cause low recoveries. Dispose of the sample tube and remaining hot solution by dropping it into the sink or waste bucket.

Note: Do not pull out all the samples at once. Each sample needs to be separated immediately upon removal from the block.

10.1.4.10. Place the tubes in the collector tube rack with the 'M' end up. Parafilm the 'D' end of the tube and allow the tubes to cool for at least 10 minutes.

10.1.4.11. For each collector tube, hold the tube horizontally and rinse the walls with the distillate in order to homogenize it. Slowly roll the distillate around the tube to gather any droplets clinging to the walls. Then slowly return the collector tube to an upright position with the 'D' end up.

10.1.4.12. With the 'D' end up, break the collector tube in half by pulling the 'D' end hard towards the user to break it and twisting and tearing off the 'D' end. Rinse the 'D' end with a small volume of 0.25N NaOH and pour into the 'M' end. The 'D' end can be discarded.

10.1.4.13. Do not invert the sample. With the 'M' end down, place the tube in the collector tube rack. Dilute to the mark with deionized water. Seal the open end with parafilm if the sample is not going to be analyzed immediately.

10.1.4.14. The solution is now ready for semi-automated colorimetry.

10.2. Calibration

10.2.1. Prepare fresh calibration standards everyday or before each analysis.

10.2.2. For Micro-Dist only: All standards (Calibration Standards, calibration blank, ICV/CCV) must be distilled in the same manner as samples.

10.2.3. Preparation of Standards for samples without sulfide:

10.2.3.1. Use the primary working solution (Sec. 7.2.4) to prepare the calibration standards below. Note: For wipe batches, the on-instrument concentrations are expressed in mg/L. Final results are calculated to mg/wipe when data is imported into TALS.

10.2.3.2. Prepare calibration standards and CCV for Micro Distillation batch as follows; the final volume in collector tube is 6.0 ml.

Volume (mL) of 6 mg/L CN primary working solution	Volume (mL) of 0.25N NaOH added to each Distillation Sample Tube	Concentration CN-(mg/L)
0.40	5	0.40
0.20	5	0.20

Volume (mL) of 6 mg/L CN primary working solution	Volume (mL) of 0.25N NaOH added to each Distillation Sample Tube	Concentration CN-(mg/L)
0.10	5	0.10
0.05	5	0.05
0.025	5	0.025
0.010	5	0.010
0	5	0.0
CCV	5	0.20

Note: [1] Add a calibration standard of 0.005 CN mg/l as directed on special projects.
[2] Prepare CCV with each distillation batch.
[3] Due to limited volume of the distillate, it may be necessary to prepare and distill two or three CCVs in the batch.

10.2.3.3. Prepare ICV using a secondary Simple CN source (Sec 7.2.3) as follows:

Volume (mL) of 6 mg/L CN secondary working solution (ICV)	Volume (mL) of 0.25N NaOH added to each Distillation Sample Tube	Concentration CN-(mg/L)
0.20	5	0.20

10.2.3.4. Prepare Calibration standards for Easy Dist batches as follows:

Volume (ml) of 10mg/L CN primary working solution	Final Volume (ml) using 0.25N NaOH	Concentration CN-(mg/L)	Concentration CN-(mg/wipe)
2.0	50	0.40	0.02
1.0	50	0.20	0.01
0.5	50	0.10	0.005
0.25	50	0.05	0.0025
0.25	100	0.025	0.00125
2.5 **	50	0.010	0.0005
0	50	0.0	0.0

** For the 0.01 mg/L standard, take 2.5 ml of the 0.20 mg/L standard and dilute to 50 ml with 0.25N NaOH.

Note: Add a calibration standard of 0.005 CN mg/l as directed on special projects.

10.2.3.5. Correlation coefficient of 0.995 or greater must be obtained.

10.2.4. Method 9012B: Preparation of Standards for samples with sulfide:

10.2.4.1. From the 6 mg/L primary working standard prepare five standards as above to be distilled in the same manner as the samples using the method of standard additions. Note: Bismuth Nitrate must also be added to the standards. Standards distilled

in this way will give a linear curve at lower concentrations but as concentration increased the recovery decreases.

10.2.4.2. Correlation coefficient of 0.995 or greater must be obtained.

10.3. Sample Analysis

10.3.1. System startup:

10.3.1.1. Follow manifold scheme for this method.

10.3.1.2. Turn on heating unit at 60° C for 15 minutes before analysis.

10.3.1.3. Put all reagent lines into deionized water and turn pump on. Check for leaks and slow flow rate.

10.3.1.4. Switch over reagent lines to proper reagent. Let reagent run through manifold for 15 minutes before analysis to allow baseline to stabilize.

10.3.2. Set up tray table with all appropriate QC checks. CCV/CCB every 10 samples and at the end of the run. The following analytical sequence must be used:

Instrument Calibration
CCV (IPC)
CCB
ICV (QCS)
ICB
Method Blank
LCS (LLCS for Easy Dist)
HLCS (for Easy Dist only)
LCSSRM (soil batches)
6 Samples with Analytical Spikes
CCV (IPC)
CCB
10 samples
CCV (IPC)
CCB
Repeat until run is complete
CCV (IPC)
CCB

Note: An LCS/LCSD will be substituted for the LLCS/HLCS in the analytical sequence for wipes.

10.3.3. Place standards and samples into autosampler cups.

10.3.4. Run tray.

10.3.5. Dilute and reanalyze samples that exceed the calibration curve using the distilled calibration blank or CCB.

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Final results calculation in aqueous samples:

Concentration = mg/L

Concentration (mg/L) from instrument is imported to TALS
Samples with dilution factor are calculated in TALS for final results

11.4. Final results calculation in solid samples:

$$\text{Concentration mg/kg} = \frac{C \times V \times D}{W}$$

Where:
C = sample concentration in extract (mg/L)
V = Volume of extract (mL)
D = Dilution Factor
W = Weight/Volume of sample aliquot extracted (grams)

NOTE: All dry weight corrections are made in TALS.

11.5. Final results calculation in wipe samples:

$$\text{Concentration mg/wipe} = \frac{C \times V \times D}{1000}$$

Where:
C= sample concentration in extract (mg/L)
V=Volume of extract (mL)
D=Dilution factor (if present)

11.6. Final results calculation amenable to chlorination:

mg CN^- amenable to chlorination/L = A - B

where:
A: mg CN^-/L: found in unchlorinated portion of sample
B: mg CN^-/L: found in chlorinated portion of sample

11.7. Data Reduction

11.7.1. Cyanide soil and waters are batched separately in TALS Analyst Desktop. Update Cyanide prep batches as follows:.

11.7.1.1. For soils, enter in 0.25-0.30g under the 'initial amount' column and 6ml under the 'final amount' column for each sample including the MB and LCSSRM.

11.7.1.2. For waters, enter 6ml under the columns 'initial amount' and 'final amount' for each sample including the MB and LCS. Under the columns 'chlorinechk' and 'sulfidechk' record the results from the test strip paper. 'N' designates the interference was not present. Record 'pH>12' under the column 'DistillpHchk.'

11.7.1.3. Since the calibration standards are distilled, they will be recorded in the prep batch. Under the column 'Lab ID,' type in "IC' for 7 rows. Enter 6ml under the columns 'initial amount' and 'final amount' for each standard. Enter how many milliliters of the 6ppm primary working solution were used for each calibration standard following the chart in Sec. 10.2.2.1.

11.7.1.4. For both soil and water batches, record reagent information including reagent ID and expiration date on the batch information page. Record the actual time the samples were distilling under 'batch comments.' This should be designated as distillation start and end time.

11.7.2. Instrument data is imported to TALS by following these steps:

11.7.2.1. After the run has finished, click the data icon at the top of the screen. Choose the correct file. Go to 'File" and choose "Export Data' from the drop down menu. A screen will pop up, click 'ok' and then another screen will pop up, click 'ok.'

11.7.3. From the desktop, open the 'LIMSdata' icon on the Lachat 8000 and the 'Exports' icon on the Lachat 8500. Select the correct file, and then right click. Go to 'Send to-> LimsImport ->' Choose the correct method. The data has now been imported to TALS.

11.7.4. All reagent information is recorded in the "Batch Information" page. Use the 'Worksheet tab' if additional pages are necessary.

11.7.5. Record special notes and observations in the "worksheet" tab (i.e. sample appearance and notes on why samples were rejected or diluted).

11.7.6. All raw data is attached as a pdf file. The raw data includes the instrument report and calibration curve.

11.7.7. Analyst must fill out the Wet Chem Data Review checklist (WI# EDS-WI-008) during the first level review. The batch is second level reviewed and the checklist is filed in wetchem department.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. 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 TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out:

- Pyridine/Hydroxide Solution: Hazardous Waste Liquid, n.o.s. This material is collected at satellite accumulation in 30 gallon Poly containers. When the container is full, the container is transferred to the waste room and held in secondary containment until a waste vendor is called for a pick up.

  Teris Profile Number: 50016717
  Onyx Profile WIP Number: 584650

15.0. References / Cross-References

15.1. Methods for the Determination of Inorganic Substances in Environmental Samples, EMSL-Cincinnati, EPA/600/R-93/100, August 1993, Method - 335.4 Determination of Total Cyanide by Semi-Automated Colorimetry

15.2. Standard Methods for the Examination of Water and Wastewater , 18th Edition, American Public Health Association, Baltimore Maryland, 1992, SM 4500-CN^- C.

15.3. Standard Methods for the Examination of Water and Wastewater , 22nd Edition, American Public Health Association, Washington, DC 2011, Editorial Revision 2011, SM 4500-CN C, G-11.

15.4. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd ed., U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. U.S. Government Printing Office: Washington, DC, 1995; SW846 Method 9012B.

15.5. QuikChem Method 10-204-00-1-A, Determination of Cyanide in Waters.

15.6. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory □□ality Man□al*, most current revision.

15.7. TestAmerica Edison SOP ED-GEN-022, □*rainin*□, most current revision.

15.8. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory □ a□te □i□po□al □roced□re*□, current revision.

15.9. TestAmerica Edison Work Instruction # EDS-WI-008, Wetchem Data Review Checklist, most current revision.

15.10. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

16.0. Method Modifications:

Item	Method no.	Modification
1	335.4	Sec 4.3.1: This SOP referenced method 335.4 (not SW846) when

		treating samples suspected to contain chlorine and nitrates/nitrites.
2	QuikChem Method 10-204-001-41-A	Sec 7.1.8.3: Preparation procedure for the Phosphate buffer follows Quikchem method 10-204-001-41-1 (not Method 335.4).
3	SM 4500 CN^- C	Sec 10.1.3.10: Sulfamic acid solution is added to all samples during distillation. Addition of Sulfamic acid is a procedure described in Standard methods 18th ed, Method 4500 CN^- C. Methods SW846 9012B and EPA 335.4 require the addition of Sulfamic acid for samples which are known to contain NO3 and/or NO2.
4	SW846 9012B	Throughout (as applicable): revised to include the preparation and analysis of wipe samples.
5	335.4; SW9012B	Sec 7.1.7.2-7.1.7.3; 10.1.4.2; 10.1.4.3- Modified procedure for reagent preparation and amount of reagent added in sample while keeping the reagent sample volume ratio and concentration the same.
6	335.4; SW9012B	□□□□ □ □□□□□□t i□ *recommended t□at at lea□t two □tandard□ (a □i□□ and a low) be di□tilled and compared to □imilar □al□e□on t□e c□r□e to en□□re t□at t□e di□tillation tec□ni□□e i□ reliable*□Sec 9.1.2: For Micro Distillation, one LCS (0.10 mg/L) will be prepared for each distillation batch; however calibration standards, ICV and CCV will be distilled in the same manner as the samples.
7	335.4; SW9012B	*Met□od □□□□□□S□ □□□□□□□i□till □tandard□ for □ample□ wit□ S□lfide*□This SOP follows the procedure described in the Hach Micro Dist User Manual, Method Number 10-204-00-1-X when distilling samples by Micro Dist- □*t i□ imperati□e t□at t□e □tandard□ be di□tilled wit□ t□e □ample*□□
8	Micro Dist (Method No. 10-204-00-1-X)	Add between 0.50-1.0g of soil/sludge to 5 ml DI water. This SOP will use 0.25g of soil sample to 5 ml DI water. Improved accuracy with the soil LCS when using 0.25g rather than 0.50g sample weight.

17.0. Attachments

N/A

18.0. Revision History

- Revision 13, dated 23 October 2017
  - Sec. 9.1.2: Corrected preparation instructions for LCS from 0.20 mg/L to 0.10 mg/L. Updated control limits for Method 9012B to +/-15%.
  - Sec. 9.2.1: Updated the ICV control limits for Method 9012B.
  - Sec. 9.2.2: Updated the CCV control limits for Method 9012B; removed instructions to prepare a CCV with each Micro-Distillation batch.
  - Sec. 10.3.2: Updated the example analytical sequence for Micro Dist and Easy Dist.
  - Sec 16, Item 6 of Method Modification – Deleted text- CCV (0.20 mg/L) will be distilled with each distillation batch.

- Revision 12, dated 26 September 2017
  - Sec. 1.1.2: Updated the Soil RL to 0.24 mg/Kg to reflect the initial soil sample weight change from 0.5g to 0.25g.

- Sec. 6.2: Added Lachat Micro Distillation tubes and pH test strips to list of supplies.
- Sec. 7.1.7.2: Revised preparation instructions for sulfamic acid solution; revised to use a double strength concentration sulfamic acid solution, however the amount added in sample is reduced in half.
- Sec. 7.1.7.3: Replaced 2.5M MgCl2 and 1:1 H2SO4 solution with Microdistillation releasing agent (7.11 M sulfuric acid/ 0.79M magnesium chloride) to be used instead of the separate H2SO4 and MgCl2 solution; equivalent reagent concentration is added in sample.
- Sec. 7.2.3: Added ERA cat# 997 as an alternative source for a Simple CN secondary standard.
- Sec 7.2.4 & 7.2.5: Added preparation instructions for the Micro Dist Primary/Secondary Working standards.
- Sec 7.2.6: Deleted LCSH
- Sec 7.2.7: Updated preparation instructions for the Complex CN Working Solution (Micro Dist).
- Sec 7.2.8: Added soil standard reference material.
- Sec 9.1.1: Updated preparation instructions for MB from deionized water to 0.25 N NaOH to reflect actual laboratory practices.
- Sec 9.1.2: Updated preparation instructions for LCS needed for Micro-Dist; removed LCSH prep for Micro Distillation. Revised the LCS sample weight to use from 0.5g to 0.25g. Added Easy Dist prep instructions for LCSSRM.
- Sec 9.1.3: Updated the matrix spike preparation instructions for Micro Dist for soil and water.
- Sec 9.2.1: Updated the ICV preparation instructions for Micro-Dist;
- Sec 9.2.2: Revised the standard source used for preparing CCV; source will be the same source as the calibration standards and will be distilled with each Micro Dist distillation batch.
- Sec 10.1.1.1: Added instructions to check pH of each sample.
- Sec 10.1.4.2: Revised the volume to add in collector tube to 2.0ml of 0.25N NaOH.
- Sec 10.1.4.3: Revised the reagents and amount of reagents added into the sample/distillation tube; Revised the initial weight used for solid samples from 0.5g to 0.25-0.30g. Added note to homogenize soil sample prior to taking aliquot.
- Sec 10.1.4.12: Added instructions to rinse the D-end and add rinsate to the M-end.
- Sec 10.2.2: Added instructions to distill all standards used for the analysis of CN by Micro Distillation.
- Sec 10.2.3.2 & 10.2.3.3: Added preparation instructions for the calibration standards and check standards (ICV,CCV) by Micro Distillation. Added note that CCV must be distilled with each distillation batch.
- Sec 10.3.2: Clarified analytical sequence for Micro Dist and Easy Dist.
- Sec 10.3.5: Updated instructions for sample dilutions.
- Sec 11.7.1: Added instructions for completing Batch information page in TALS.
- Sec 16: Method modification items 5-8 added.

- Revision 11, dated 11 August 2014
  - Sec. 7.2.2 & 7.2.3: Clarified procedure as to when to apply the standardized value of the CN stock solution.

- Sec. 9.1.2: LLCS and HLCS will be analyzed for both methods SW9012 and EPA335.4 Deleted text: *'Distillation of two LCSs (LLCS and HLCS) is not a method requirement for Method 335.4. Use HLCS as the QC sample for Method 335.4.*

- Revision 10, dated 12 March 2014
  - Deleted Method 9012A reference throughout the SOP; replaced SW method reference with 9012B.
  - Sec 15.3 Added reference to Standard Methods Editorial revision 2011; subsequent sections adjusted accordingly.

- Revision 9, dated 12 November 2012
  - Sec 1.1.2: Revised RL for solids from 0.12 mg/kg to 0.10 mg/kg; sample final volume is 5 ml.
  - Sec 9.1.2 & 9.1.3: Deleted text: *After micro-distillation, volume is brought up to 6 ml with deionized water.* The final volume of the distillate is not adjusted.
  - Deleted Sec 10.1.4.13, final distillate will not to be diluted to 6 ml; subsequent sections adjusted accordingly.
  - Sec 10.1.4.13 (formerly Sec 10.1.4.14): deleted text: *Shake the tube with a gentle whipping motion to mix in diluent water.* Added text: *With the 'M' end down, place the tube in the collector tube rack.*

- Revision 8, dated 25 October 2012
  - Deleted Sec 7.2.4 (5 mg/L primary working solution), standard not applicable; subsequent sections adjusted accordingly.
  - Sec 7.2.2, 7.2.3 & 7.2.6: Clarified to include which QC samples these standards are applicable to.
  - Sec 7.2.6 & 7.2.7: Added complex cyanide standard, 1000 mg/L stock standard and 10 mg/L working solution to be used for distilled QC samples (LCS and MS/MSD).
  - Sec 9.1.2: Added note: *Distillaton of two LCSs (LLCS and HLCS) is not a method requirement for Method 335.4. Use HLCS as the QC sample for Method 335.4.*
  - Sec 9.1.2: Revised the LLCS concentration to 0.10 mg/L; revised to reflect the correct spiking amount (ml) and source standard (complex CN working solution) in the preparation procedure.
  - Sec 9.1.3: Revised the spiking solution from simple to complex CN.

- Revision 7, dated 13 September 2012
  - Sec 1: Revised to include the RL for microdistillation in soils.
  - Sec. 2.0: Revised to include 'MICRO DIST' as one of the ways to distill samples.
  - Sec. 6.0: Updated list of equipment (added MICRO DIST and Lachat 8000 and 8500 series).
  - Sec. 9.1.1: Changed the frequency for the Method Blank analysis from 1 per 10 samples to 1 per 20 samples.
  - Sec. 9.1.2: Changed the frequency for the LLCS/HLCS analysis from 1 per 10 samples to 1 per 20 samples. Also, included the micro distillation preparation procedure for LLCS/HLCS.
  - Sec. 9.1.3: Clarified the requirement of one MS/MSD per 10 samples. Also, included the micro distillation preparation procedure for MS/MSD.
  - Sec. 10.1.4: Added procedure for micro distillation of samples.

- Revision 6, dated 22 March 2012
  - Sec. 1.1.1: added method 4500 CN G to the list of methods.
  - Sec 1.1.3, 12.1 & 12.2: Revised QAM references to reflect the most recent QAM revision.
  - Sec. 7.2.2/7.2.3: Added information on commercially purchased CN stock standard.
  - Sec 9.1.3: Added MS/MSD requirement for cyanide amenable to chlorination under sample QC.
  - Sec. 10.1.2.1: Added prep for soil samples requiring cyanide amenable to chlorination.
  - Sec 11.6: Added calculation formula for CN amenable to chlorination; subsequent sections adjusted accordingly.
  - Sec 15.9: Added applicable reference.

- Revision 5, dated 22 November 2010
  - Sec. 1.1.1: Added wipes to list of applicable matrices.
  - Sec. 1.1.2: Added reporting limit for wipes.
  - Sec. 6.2: Added specimen cups, transfer pipettes, graduated cylinder and micro-burets to the list of supplies.
  - Sec. 7.1.4: Changed the amount of acetic acid added from “approximately 100 ml” to “approximately 10 drops” to reflect actual laboratory practices.
  - Sec. 7.1.6.2: Clarified reagent prep
  - Sec. 8.0: Added wipes to section.
  - Sec. 9.1: Added sample QC requirements for wipe batches.
  - Sec. 9.1.1: Included the prep of the method blank for wipe batches
  - Sec. 9.1.2: Revised the prep of the LLCS and HLCS to reflect actual laboratory practices. Also, included prep of the LCS/LCSD for wipe batches.
  - Sec. 9.1.3: Revised MS/MSD prep for aqueous and soil.
  - Sec. 9.2.2: Deleted text “If the CCV is not within the control limits, a second analysis should be performed.”
  - Sec 9.2.3 & 9.2.4: Clarified the type of solution utilized for the ICB and CCB.
  - Sec. 10.1.3.1: Revised step of the distillation procedure to reflect actual laboratory practices.
  - Sec. 10.2.2.1: Revised calibration standards prep Table.
  - Sec. 10.3.2: Added comment on how to set up the analytical sequence for wipe samples.
  - Sec. 11.5: Included calculation for wipe samples.
  - Sec. 11.6: Added steps to import data to TALS.
  - Section 16: Added method revision for addition of wipe matrix to SW846 9012A/9012B.

- Revision 4, dated 28 September 2009
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Incorporated SOP ED-WET-007 (Analysis of Total Cyanide in Drinking water-Automated, Method 335.4) into this SOP; retired SOP ED-WET-007 at the effective date of this SOP.

- Sec. 7.2.2: Added information for the standardization of the Cyanide Stock. Replace frequency of the standardization from monthly to weekly to comply with Standard methods.
- Sec 7.2.3 – 7.2.6: Added standards and preparation instructions.
- Sec 9.1.3: Clarified % recovery limits for water and solid samples.
- Sec 9.2.1 & 9.2.2: Clarified corrective actions to be taken if QCS/ICV and IPC/CCV results are outside the acceptable limits.
- Sec 10.3.2: Revised sample analytical sequence to comply with method 335.4.
- Sec. 11.5: Revised new data reduction procedures in accordance with new TALS.
- Sec. 15: Replace reference 335.2 and 335.3 with the EPA 335.4 reference; added applicable reference.
- Sec 16: Added Method modification to specify actual source of reagent information.

- Revision 3b, dated 13 July 2009
  - Sec 2.1: .For each batch of ten samples or less, a matrix spike and matrix spike duplicate will be distilled and analyzed. (Replaced 20 samples to 10 samples per batch).
  - Sec 9.5.1.3 & 9.5.2.2.: Revised Correlation coefficient from 0.997 to 0.995 as per corporate SOP on Calibration curves.
  - Sec 10.3: Up to 10 samples can be put in a batch. .Revised number of samples in a batch from 20 to 10 samples.

- Revision 3a, dated 13 March 2009
  - Sec 1.1: Delete Methods 335.1/.3; Add Method Lachat 10-204-00-1-A (Methods deleted in response to MUR).
  - Sec 3.6: Delete text: If this occurs, a Dow Corning 544 can be added to prevent the foam from collecting in the condenser. Replace the above procedure with: *If this occurs, samples can be acidified with acetic acid (6M) to pH 6.*
  - Sec 5.2: Add: Solution is stable for 6 months or when QC check is outside the acceptable limits, whichever comes first, store at 4ºC.
  - Sec 5.4: Acetate Buffer-Dissolve 82.0g of NaC2H3O2 x 3 H2O in 100ml DI water. Add glacial acetic acid drop by drop until the pH is 4.5. This solution is stable for 6 months, store at 4ºC. (Changed amount of acetic acid added to reflect actual laboratory practices).
  - Sec 5.6: Remove: Dow Corning 544 antifoam agent. Lab does not use this reagent , samples will be acidified with acetic acid.
  - Sec. 5.7.1, 5.8.2, 5.8.3, 5.8.4: Add: Solution is stable for 6 months, store at room temperature.
  - Sec 5.7.2: 0.25M NaOH. Add: Dissolve 10.0g NaOH into 1L deionized water. Solution is stable for 6 months, store at room temperature.
  - Sec. 5.9.1 & 5.9.2: Add: Store at room temperature.
  - Sec. 5.9.3: Phosphate buffer: In a 1L volumetric flask, dissolve 97g of potassium phosphate, monobasic, anhydrous (KH2PO4) in approximately 800ml of deionized water. Dilute to the mark. Solution is stable for one month. Store at room temperature. (Changed reagent preparation to comply with the Lachat method).
  - Sec 6.3: Add: Laboratory Control Sample (LCS): Prepare a 5ppm and 10ppm CN standard by diluting the 1000ppm Primary CN stock. Prepare fresh daily.
    - LLCS: Take 0.25ml of the 5ppm CN standard and bring up to a 50ml final volume with 0.25N NaOH. The true value is 0.025ppm.

- HLCS: Take 1.0ml of the 10ppm CN standard and bring up to a 50ml final volume with 0.25N NaOH. The true value is 0.2ppm

- Sec 9.10: Delete text: “Free Cyanide is estimated using the weak acid dissociable procedure following the colorimetric procedure without distillation. For reference refer to SM 4500-CN-A.” Revise the above text to: “*□ree C□anide will be treated following the procedure for the C□anide Amenable to chlorination and therefore must be distilled.□See Section □.□*
- Sec. 10.7: MS and MSD Recovery: Delete: The recovery must be within laboratory generated limits. Revise the above text to: *□he recover□ must be within □□-□□□□ (*Revise MS/MSD recovery to comply with method 335.4).
- Sec. 15.1: References: Replace 335.2 & 335.3 reference with EPA 335.4 reference

Title: PCB Preparation for Analysis by HRGC/HRMS [Method 1668A & EPA 1668C]

Approvals (Signature/Date):

Robert Hrabak, Technical Director — 7/26/18	Joe Schairer, Health & Safety Manager / Coordinator — 7/30/18
Lisa Stafford, Quality Assurance Manager — 7/25/2018	Chris Williams, Laboratory Director — 8/1/18

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1. SCOPE AND APPLICATION

1.1. This SOP is appropriate for the preparation of samples for analysis of mono- through deca-chlorinated biphenyls in a variety of matrices at low part-per-trillion to part-per-billion levels using high resolution gas chromatography and high resolution mass spectrometry, by Methods 1668A and 1668C.

1.2. The calibration range of the procedure for 1 L water is 20 to 20000 ppq, 2 to 2000 ppt for 10.0 g soil, sediment or tissue, and 40-40,000 pg/train (assuming 1/2 sample to 20 uL F.V.) for air train samples for mono-deca PCBs. Analysis of dilutions of aliquots of the sample will permit measurements of concentrations above the upper method calibration limit. The practical limits of detection and quantitation may be different from the lower method calibration limit, depending on the complexity of the matrix and the level of PCB contamination of the reagent and absorbent used in the extraction and cleanup procedure.

1.3. All PCB congener labeling in this document is consistent with IUPAC naming conventions.

1.4. 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.

1.5. The detection limits and quantitation limits in this method are driven more by the level of interference and laboratory background than by absolute instrument sensitivity. Reporting limits are discussed in the analysis SOP WS-ID-0013.

2. SUMMARY OF METHOD

2.1. This procedure uses matrix specific extraction and analyte specific cleanup techniques.

2.2. An aliquot of a matrix (water, soil, sediment, XAD Resin, filter) is spiked with the solution containing 27 isotopically 13C-labeled PCBs listed in Table I. The sample is then extracted according to matrix specific extraction procedures.

2.3. After extraction, surrogate standards are spiked into the extract prior to performing cleanups. Available cleanup processes include silica gel, and gel permeation chromatography. Acid and/or base back extraction may also be used.

2.4. The preparation of the final extract for the instrumental analysis is accomplished by adding 5 isotopically ($^{13}C_{12}$) labeled internal standards (Table I, “Recovery Standard Solution”). Quantitation and analysis is performed as described in the analytical method SOP WS-ID-0013.

2.5. After internal standards are added and the extract is concentrated to 20u□, the extract is then analy□ed according to SOP WS-ID-0013.

2.□ Quantitative analysis is performed differently for different congeners□

2.□1. The Toxic and □OC PCBs are determined using isotope dilution against a 5 point calibration.

2.□2. □or all congeners excluding those in Section 2.□1 quantitation is carried out by internal standard technique using a single calibration point.

□. DEF□□□T□O□S

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance □anual (QA□).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QA□.

□. □□TERFERE□□ES

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 interferences under the conditions of analysis by running laboratory method blanks. Analysts shall not use P□C gloves.

4.2. The use of high-purity reagents and solvents helps minimi□e interference problems. Purification of solvents by distillation in all-glass systems may be necessary.

4.3. Reuse of glassware is to be minimi□ed to avoid the risk of contamination. If samples are known or suspected of containing high analyte levels glassware should be segregated and isolated until analytical results are available. Additional cleaning or disposal of glassware may be necessary dependent upon analyte levels. □lassware cleaning details are covered in SOP WS-OP-0011.

4.4. Interferences co-extracted from the sample will vary considerably from matrix to matrix. PCBs are often associated with other interfering chlorinated substances such as polychlorinated dioxins/furans (PCDD/PCD□), polychlorinated diphenyl ethers (PCDP□s), polychlorinated naphthalenes, methoxy biphenyl hydroxydiphenyl ethers, ben□ylphenyl ethers, brominated diphenyl ethers, polynuclear aromatics and pesticides 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 (30 m SPB Octyl) is used to resolve as many PCB isomers as possible□however, no single column is known to resolve all isomers.

□. SAFETY

□mployees must abide by the policies and procedures in the Corporate □nvironmental □ealth and Safety □ anual (CW-□-□ -001), the Sacramento Addendum to the Corporate □□□S □ anual (WS-P□□S-0002), and this document. This procedure may involve ha□ardous 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 ha□ardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. □ye protection that satisfies A□SI □□7.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. □atex and vinyl gloves provide no protection against most of the organic solvents used in this method. □itrile or similar gloves must be used. □atex gloves may be used for methanol.

5.1.2. □xposure 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. □aboratory 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 in□uries. □aboratory associates performing these procedures are in the best position to reali□e when they are at risk for these types of in□uries. 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 □□□S staff. The task will be analy□ed to determine a better means of accomplishing it.

5.1.4. □earing protection must be worn when using mechanical systems to grind tissue samples.

5.1.5. When dissecting crawfish abdomens with a scalpel, cut from the hand holding the abdomen toward the tail (away from you).

5.1.□ □inely divided dry soils contaminated with PCBs are particularly ha□ardous 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.

5.1.7. Assembly and disassembly of glassware creates a risk of breakage and cuts. All staff members shall wear □evlar or similar cut-resistant gloves over chemically resistant gloves when assembling and disassembling glassware.

5.1.□ The use of separatory funnels to extract aqueous samples with methylene chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the sample container has been sealed and inverted. □ent 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.

5.1.□ 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. □lass 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.

5.1.10. □ ercury is a highly toxic compound that must be handled with care. The analyst must be aware of the handling and cleanup techniques before handling this material. Spilled mercury requires that special cleanup tools and procedures be used.

5.1.11. When dispensing methylene chloride or any other solvent from the Cycletainer, do not lock the dispenser into the open position and walk away to perform any other tasks. While you may lock the dispenser open for steady flow to reduce the ergonomic impact, you must stay there with the dispenser and container being filled to prevent it from overflowing.

5.2. Primary □ aterials □sed

The following is a list of the materials used in this method, which have a serious or significant ha□ard rating. □**OTE □T** □□□ □□□□ □□□□ □□□□□□□□□□ □□□□ □□□□□□□□ □□□□ □□ □□□ □ □□□□□. **T** □□ □□□□□ □□□□□ □□□□□□□□□□ □□□ □ □□□ □□□□□ □□□□ □□□ □□□□□□□□□□□□ □□ □□□ **SDS** □□□ □□□□ □□□□□ □ □□□□□□□□□□□□ □□ □□□ □□□□□. A complete list of materials used in the method can be found in the reagents and materials section. □mployees must review the information in the SDS for each material before using it for the first time or when there are ma□or changes to the SDS.

Table 1: Materials used in this SOP and their Hazards			
Material	**Hazards**	**Exposure Limit (2)**	**Signs and symptoms of exposure**
Sulfuric Acid (1)	Corrosive Oxidizer Dehydradator	1 mg/m3	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.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m3	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.
Mercury	Poison	0.1 Mg/M3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
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.
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.
Iso-octane (2,2,4-trimethylpentane, 2,4,4-trimethylpentane)	Flammable	500 PPM TWA	Causes eye or respiratory tract irritation. Repeated/ prolonged exposure can cause defatting of skin. High concentrations can produce drowsiness
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.
Dodecane	Flammable	None listed	May cause respiratory tract, skin or eye irritation.

Table 1: Materials used in this SOP and their Hazards			
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.
Nonane	Flammable	200 ppm	Primary hazard is flammability. May also cause skin irritation, drowsiness and dizziness if inhaled.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

□. E□U□□ME□T A□D SU□□□□ES

□1. □quipment for sample preparation

□1.1. □aboratory fume hood of sufficient si□e to contain the sample preparation equipment listed below.

□1.2. □love box (optional)

□1.3. □ eat grinder - □obart, or equivalent, with 3-5 mm holes in inner plate.

□1.4. Analytical Balance - Capable of weighing 0.1 mg

□1.5. Top □oading Balance- Capable of weighing 10 mg.

□2. □xtraction Apparatus

□2.1. Water Samples

□2.1.1. □raduated cylinder, 1□ capacity

□2.1.2. 1□ filtration flasks with side arm, for use in vacuum filtration of water samples

□2.1.3. Separatory funnels - 250, 500 and 2000 m□, with Teflon stopcocks.

□2.1.4. □lass fiber filters, Whatman □□-D, □□-□, □□ □-150 or equivalent

□2.2. Soxhlet/Dean-Stark (SDS) extractor

□2.2.1. Soxhlet □50 mm i.d., 200 m□ capacity with 500 m□ flask or 70 mm x 240 mm with 1000 m□ round bottom flask.

□2.2.2. Thimble - 30 x 100 or 43 x 123 mm □lass fiber

□2.2.3. □ oisture trap - Dean-Stark or Barret.

□2.2.4. □eating mantle - hemispherical, to fit 500 m□ or 1000 m□ round bottom flask (Cal-□lass □□-□□01-112, or equivalent).

□2.2.5. □ariable transformer - Powerstat (or equivalent), 110 volts, 10 amps.

□2.3. Spatulas - Stainless Steel or disposable tongue compressors.

□2.4. Soxtherm extraction apparatus (□erhardt S□41 □or equivalent), including glass thimble holders, glass beakers, and gaskets.

□3. □iltration Apparatus

□3.1. Sodium sulfate, granular anhydrous

□3.2. Pyrex glass wool. (pre-rinsed with methylene chloride)

□3.3. □lass stemless filter funnel - 125 - 250 m□.

□3.4. □lass fiber filter paper (Whatman □□/D, or equivalent).

□3.5. Buechner funnel, 15 cm.

□3.□ Pressure filtration apparatus - □acuum filtration

□4. Centrifuge Apparatus

□4.1. Centrifuge - Capable of rotating 500 m□ centrifuge bottles or 15 m□ centrifuge tubes at 5000 rpm minimum

□4.2. Centrifuge tubes - 12-15 m□, with screw caps, to fit centrifuge

□5. Concentration Apparatus

□5.1. Rotary evaporator □(B□chi R210 or equivalent) equipped with a variable temperature water bath.

□5.1.1. A vacuum source is required for use of the rotary evaporator. It must be equipped with a shutoff valve at the evaporator, and preferably, have a vacuum gauge.

□5.1.2. A recirculation water pump and chiller are recommended, as the use of tap water for cooling the evaporator wastes large volumes of water, and can lead to inconsistent performance because water temperatures and pressures vary.

□5.1.3. Round bottom flasks - 500 m□ and 1000 m□ or larger, with ground glass fitting compatible with the rotary evaporator.

□5.2. Turbo evaporator with a temperature controlled water bath and nitrogen pressure control.

□5.3. □itrogen blow down apparatus - installed in a fume hood.

□5.4. Sample vials - 2-5 m□ with Teflon-lined screw cap and 200 u□ auto-in□ector insert vials with septum lined screw caps.

□. REA□E□TS A□D STA□DARDS

7.1. Reagents for extraction procedures.

All reagents must be ACS reagent grade or better unless otherwise specified.

7.1.1. p□ ad□ustment and back extraction

7.1.1.1. 10□ sodium hydroxide □Reagent grade

7.1.1.2. Sulfuric acid - Reagent grade (specific gravity 1.□4)

7.2. Solution drying and evaporation

7.2.1. Solution drying - Sodium sulfate, reagent grade, granular anhydrous (Baker 3375, or equivalent), rinsed with methylene chloride (20 m□/g) and stored in a pre-cleaned glass bottle with a screw cap that prevents moisture from entering.

7.2.2. Prepurified nitrogen, reagent grade

7.3. □xtraction Solvents□Acetone, toluene, hexane, dodecane, iso-octane, nonane, methanol and methylene chloride (DC□).

7.4. Absorbents for sample cleanup

7.4.1. Silica gel

7.4.1.1. Activated silica gel - Silica □el □0-□isher S□25-212, 230-400 mesh, rinsed with methylene chloride, baked at 1□5□C-1□5 □C for at least 12 hours before use. Store at 125 □C-145 □C in a covered flask or bottle.

7.4.1.2. Acid silica gel (44 percent w/w) - Thoroughly mix 24 m□ (44g) of concentrated sulfuric acid with 5□0 g of activated silica gel in a clean container. Break up aggregates with a stirring rod until a uniform mixture is obtained. Store in a screw-capped bottle with a Teflon-lined cap.

7.4.2. Alumina□ the standard alumina cleanup procedure employs acidic alumina only. The same type of alumina must be used for all samples, including those used to demonstrate ongoing precision and accuracy (□CS).

7.4.2.1. Acid Alumina - IC□ Super A. This alumina is purchased activated and can be used directly from the bottle. A column profile is performed for each new vendor□otherwise a laboratory control sample is used to re-verify new material from an existing vendor.

7.4.3. □ ercury□triple distilled. (□ allinckrodt Baker, Inc. or equivalent)

7.5. Reference matrices

7.5.1. Reagent water - Water in which the PCBs and interfering compounds are not detected at above □ the reporting limit by this method. The lab is currently using □P□C water.

7.5.2. □igh solids reference matrix □sodium sulfate.

7.5.3. □lass fiber filter paper or extraction thimble - □lass fiber filter papers or extraction thimbles in which the PCBs and interfering compounds are not detected by this method.

7.5.4. Other matrices - This method may be verified on any matrix if the following criteria are met. Ideally, the matrix should be free of the PCBs, but in no case shall the background level of the PCBs in the reference matrix exceed three times the minimum levels given in Table 2 (CC1). If low background levels of the PCBs are present in the reference matrix, the spike level of the analytes used in Table III should be increased to provide a spike-to-background ratio in the range of 1□1 to 5□1.

7.5.5. Tissue reference matrix □ □egetable oil (or equivalent) and sodium sulfate.

7.□ Standard solutions - Purchased as solutions or mixtures with certification to their purity, concentration and authenticity, or prepared from materials of known purity and composition. If compound purity is □□percent or greater, the weight may be used without correction to compute the concentration of the standard. When not being used, standards are stored in the dark in screw-capped vials with Teflon-lined caps in a refrigerator at 3 ± 3 degrees C. A mark is placed on the vial at the level of the solution so that solvent evaporation loss can be detected. If solvent loss has occurred, the solution should be replaced.

7.□1. Sealed ampoules may be used until the manufacturer□s expiration date is exceeded.

7.□1.1. If no expiration date is provided, then the expiration date will be 10 years from the date the ampoule is opened.

7.□1.2. The solvent level should be monitored prior to each use to assure there has been no concentration of the standard over time.

7.□2. All calibration, daily isotope dilution analytes, daily clean up isotope dilution analytes, daily internal standard and daily spiking solutions are stable for one year from preparation.

7.□2.1. After one 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.□2.2. The re-verification must be performed using an unexpired, not previously re-verified solution from a second lot or second vendor.

7.7. Stock solutions

7.7.1. Preparation - Prepare in isooctane or equivalent solvent per the steps below or purchase as dilute solutions (Cambridge Isotope □aboratories, Cambridge, □ A, or equivalent).

7.7.2. Stock standard solutions are prepared from dilutions of neat solutions. Dilutions are performed in volumetric flasks and transferred to a clean vial or amber glass bottle with Teflon-lined cap.

7.□ Stock standard solutions should be checked for signs of degradation prior to the preparation of calibration of performance test standards. Reference standards that can be used to determine the accuracy of calibration standards are available from Cambridge Isotope □aboratories.

7.□ Secondary standard - □sing stock solutions, prepare secondary standard solutions containing the compounds and concentrations shown in Table 1 in dodecane.

7.10. □abeled compound stock standard - □rom stock standard solutions prepared as above, or from purchased mixtures, prepare this standard to contain the labeled compounds at the concentrations shown in Table 1 in iso-octane.

7.11. Isotope Dilution Analytes (IDA) - Prepare at the concentration shown in Table 1 in iso-octane.

7.12. Surrogate Standard (S□) - Prepare at the concentration shown in Table 1 in iso-octane.

7.13. Internal Standard (IS) - Prepare at the concentration shown in Table 1 in dodecane.

7.14. □ative spike solution (TA) - □sed for determination of ongoing precision and accuracy, in the form of a laboratory control sample (□CS). This solution contains the analytes and labeled compounds at the concentrations listed in Tables 1 and 3 in isooctane.

7.15. Standard solutions will be periodically assayed against reference standards. Continued use of standard solutions past the initially indicated expiration date is acceptable if concentrations are verified versus the reference standard. □pon acceptable verification, a new expiration/evaluation date will be noted in the standards logbook.

7. SAMPLE COLLECTION, PRESERVATION AND STORAGE

7.1. The sample collection, shipping, handling, and chain-of-custody procedures are not described in the document. The complexity of the method is such that, in order to obtain reliable results, testers should be trained and experienced with sampling and preservation procedures.

7.2. There are no demonstrated technical holding times associated with the PCBs in aqueous, solid, semi-solid, tissues, or other matrices. If stored in the dark at 0-6°C, without freezing, and preserved if required, aqueous samples may be stored for up the one year. 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.

7.3. All extracts must be stored capped and in the dark at ≤-10.

8. QUALITY CONTROL

8.1. One method blank must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The method blank is an aliquot of reference 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 acceptance limit or when surrogate recoveries are outside control limits. The associated samples will be evaluated for adverse impact, and flagged or qualified as appropriate. Re-extraction of the blank, other batch QC, and the affected samples are required when the method blank is deemed unacceptable. For any analyte detected in the blank in any of the homologous series of PCBs, the detection limit in the samples analyzed with that blank for the specific isomer or the total for that homologous series is increased to 5 times of the contamination level. Alternatively, both QC and sample results may be reported and qualified as necessary.

8.1.1. Certain programs, such as those under the 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 reporting limit.

8.2. 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. reagent water, Ottawa sand, sodium sulfate, extraction thimble, filter paper, etc.) spiked with 100 uL of Isotope Dilution Analyte fortification solution (Table 1) and 100 uL of Target Analyte Standard fortification solution (Table 3). 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 50-150%, or the control

limits provided on the □I□ S, or by the client. The associated samples will be evaluated for adverse impact, and flagged or qualified as appropriate. Re-extraction of the blank, other batch QC, and the affected samples are required when the □CS is deemed unacceptable

□3. A matrix spike/matrix spike duplicate (□ S/□ SD or □ S/SD) pair may be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. □ S/□ SD are analy□ed at the request of the client. An □ S/□ SD pair is aliquots of a selected field sample spiked with analytes of known identity and concentration. The □ S/□ SD pair must be processed in the same manner and at the same time as the associated samples. Spiked analytes must be within control limits 50-150□, or the control limits provided on the □I□ S, or by the client. The result obtained from □ S and □ SD samples analysis should agree within 50 percent relative difference. Corrective actions must be documented on a □on-Conformance memo. Outliers with recoveries or precision outside control limits will be flagged and narrated as appropriate.

□□. □A□□□RAT□O□

10.1. On a daily basis, calibrate any auto-pipettes to be used in accordance with SOP WS-QA-0004.

10.2. On a daily basis, calibrate any balances to be used in accordance with SOP WS-QA-0041.

□□. □RO□EDURE

One time procedural variations are allowed only if deemed necessary in the professional □udgment of a supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample si□e, or other parameters. Any variation in procedure shall be completely documented using a □on-Conformance □emo and is approved by a Technical Specialist and QA □anager. If contractually required, the client shall be notified. The □on-Conformance □emo shall be filed in the pro□ect file. Any unauthori□ed deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.1. □rinding or Blending of Tissue Samples.

If not otherwise specified by the client, the whole fish (fro□en) 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 si□e 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. Please refer to SOP WS-WI-001□ for further grinding procedure.

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11.2. Phase Separation

Phase separation on very wet (≥25 percent water) soil and sediment samples may be accomplished as follows: Place a 50 g portion in a suitable centrifuge bottle and centrifuge for 30 minutes at 2,000 rpm. Remove the bottle from the centrifuge and mark the interface level on the bottle. Estimate the relative volume of each phase. With a disposable pipette, transfer the liquid layer into a clean bottle. Mix the solid with a stainless steel spatula or tongue compressor and remove a portion to be weighed and analyzed (percent moisture determination, extraction). Return the remaining solid portion to the original sample bottle (if empty) or to a clean sample bottle that is properly labeled, and store it as appropriate. Analyze the solid phase by using only the soil and sediment method. Take note of the estimated volume of liquid before disposing of the liquid as a liquid waste.

CAUTION: For samples containing high levels of volatile water miscible organics, the analyst must ensure, during the preparation step, that the pH of the sample remains neutral or basic. Acidic conditions will cause the loss of volatile water miscible organics.

11.3. Soxhlet Extraction (Solids, Tissues, Sludges, and Wipes).

11.3.1. Percent moisture is determined in accordance with SOP WS-OP-0013.

11.3.2. Isotope Dilution Analyte Standard. A 100 µL aliquot, or 2 ng equivalent, of the internal standard mixture is added to all samples, regardless of sample size.

CAUTION: Only use cooling water that is not chilled. Condensers that are cooled with chilled water can cause water to condense on the outside of the condenser and drip into the sample. Check condensers prior to 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.

11.3.3. Sludges. Paper pulp sludges are generally air-dried and ground. Because of the drying procedure, a Dean-Stark water separator may or may not be used for extraction. Extraction is generally done by Soxhlet with 200-300 mL of toluene.

11.3.3.1. Non-Paper pulp sludges are extracted with 200-300 mL of toluene.

11.3.3.2. Soxhlet sample for a minimum of 16 hours. Cool the sample, then filter the toluene extract, if needed, through a glass-fiber filter or equivalent into a round-bottom flask. Rinse the filter with 10 mL toluene, and combine the extract and rinsate. Concentrate the toluene extract solutions to near dryness on a rotary evaporator at 80°C and 25 pounds vacuum at 120-150 RPM Use of an inert gas to

concentrate the extract is also permitted. Proceed with Section 11. as necessary.

11.3.3.3. Still-Bottom uel il. ll organic liquids, fuel oils, and solids that will dissolve in a solvent and are treated as a solvent dilution will be prepared by dissolving a nominal 1g of sample in 0mLs of oluene or an appropriate solvent. ake a mL aliquot and spike with appropriate D solution and proceed with Section as necessary.

11.3. ly sh. eigh a 2-10 g sample aliquot into a glass fiber extraction thimble. dd 100 uL of the D mixture. he samples are Soxhlet extracted with toluene for a minimum of 16 hours. 100uL of dodecane can be used as a keeper in the samples, if the lower column cleanup is planned. Concentrate the toluene extract solutions to near dryness on a rotary evaporator at 80°C and 25 pounds vacuum at 120-150 RPM. Proceed with Section 11. as necessary.

***Note**: As an option, sodium sulfate may be mixed with the samples to remove moisture before sample extraction.*

11.3.5. Solids/Tissues -- Weigh a 2-10 g sample aliquot into a glass fiber extraction thimble. Add 100 uL of IDA solution. Extract the samples in a Soxhlet with a Dean-Stark apparatus with toluene for a minimum of 16 hours. The solvent must cycle completely through the system at least five times per hour. Concentrate by rotary evaporator at 80ºC and 25 psi vacuum at 120-150 RPM.

11.3.5.1. If tissues requiring % Lipids are to be extracted, for each sample weigh a 20 g aliquot for tissues into a glass fiber extraction thimble, as percent lipids will be determined as well from this extraction. Add 200 uL of IDA solution and extract for at least 16 hours. Concentrate by rotary evaporator at 80ºC and 25 psi vacuum at 120-150 RPM until ~10mLs of solvent remains. Proceed to 11.5 for % Lipids determination.

***Note**: The matrix for the tissue method blank consists of 9 g of sodium sulfate and 0.25 g of canola oil.*

11.4. Soxtherm Extraction (Solids, Tissues, Sludges, and Wipes)

11.4.1. Prior to loading samples, run the system through 2 cleaning cycles (approximately one hour each).

11.4.2. After pre-extraction, cool the system, disassemble the apparatus, and rinse with acetone.

11.4.2.1. Weigh a well-mixed aliquot of each sample (10 g, unless otherwise specified) into a clean Soxhlet thimble. Record the mass to the

nearest 0.01g. □se sodium sulfate for the batch □C (M□, LCS) for solids, and a mixture of □g sodium sulfate and 0.25 g vegetable oil for the batch □C for tissue matrices.

11.4.2.2. If tissues requiring % Lipids are to be extracted, for each sample weigh a 20 g aliquot for tissues into a glass fiber extraction thimble, as percent lipids will be determined as well from this extraction.

11.4.2.3. In the case of wipes, place the entire wipe sample into the Soxhlet apparatus (no thimble needed), including any liquid present with the sample. □se pre-cleaned wipes for the batch □C samples.

11.4.3. Place the thimble into the Soxtherm apparatus.

11.4.4. Spike all samples with 100 uL of IDA solution (2 ng), for a final concentration of 200 pg/g (based on a 10 g sample). If % lipid is needed or samples are wipes, add 200 uL of IDA solution.

11.4.5. Reassemble the pre-extracted apparatus and add a fresh charge (150 mL) of toluene to the apparatus and boiling chips.

11.4.6. Program the system to boil for 1 hour, and reduce the toluene volume by □0-□0 mL (volume □volume of the thimble).

11.4.□ Continue the extraction for one hour fifteen minutes, reducing the toluene volume by another 15 mL.

11.4.8. After refluxing, allow the apparatus to cool.

11.4.□ Pour the samples into round bottom flasks.

11.4.□1. If % lipid is required, bring samples up to 10mLs with □exane and split in half. % lipid portion will proceed to Section 5 and the other half will proceed to Section 11.□(Cleanups) as necessary.

11.4.□2. If % lipid is not required, and an alumina column cleanup (Section 11.□4) is planned, add 100uL of dodecane to the sample as a solvent keeper.

11.4.□3. If extracts were wipe samples, bring samples up to 10mLs in □exane and split samples in half. □alf will be archived and the other half will proceed to Section 11.□(Cleanups) as necessary.

11.4.10. Proceed with Section 11.□(Cleanups) as necessary.

11.5. □ish Tissue Lipid Content Determination

The percent lipid of fish samples is determined as follows□

11.5.1. Concentrate the extract from Sections 11.3.5 or11.4 on a rotary evaporator

until volume is low enough to transfer to a culture tube (approx. 5-10mLs) that has been pre-weighed with label. Transfer the sample into the culture tube and continue blowing down the sample until constant weight is attained. The percent lipid is calculated using the following expression□

Equation 1

$$\text{Percent Lipid} = \frac{FinalTubeMass - InitialTubeMass\ (g)}{1/2\,EntireSampleMassExtracted\ (g)} \times 100$$

11.6. Aqueous Samples.

11.6.1. Determine the sample volume by placing the sample bottle on a top-loading balance and record/transfer the weight into TALS. If there is no headspace in the bottle, carefully decant 5-10 mL of the sample before taking the weight.

11.6.1.1. 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.

11.6.2. Add 100 uL of IDA solution to the sample in the bottle. Each aliquot of spike mixture is added similarly. Shake the bottle for 30 seconds and allow to sit for at least one hour prior to extraction.

11.6.3. Create a method blank and LCS by adding 1 L of laboratory reagent water into two 1 L amber glass bottles. Add 100 uL of IDA solution to each sample and 100 uL of native spike solution to the LCS.

11.6.4. When ready for extraction, pour the entire sample (approximately 1 L) into a 2 L separatory funnel.

11.6.5. Reweigh the empty bottle and record/transfer the sample weight into TALS. Record the sample weight to the nearest 1 gram.

11.6.6. Add 100 mL methylene chloride (DCM) to the sample bottle, seal, and shake for 30 seconds to rinse the inner surface. Transfer the solvent to the separatory funnel.

11.6.7. Extract the sample by shaking the funnel for two minutes with periodic venting. Allow the organic layer to separate from the water phase for a minimum of 10 minutes.

WARNING: Methylene chloride creates excessive pressure very rapidly! Therefore, initial venting should be done immediately after the sample container has been sealed and inverted. Vent into hood away from analysts and other samples.

WARNING: Separatory funnel extraction is a high risk activity. Analyst will wear a face shield over safety glasses/goggles for this extraction. Alternatively, the extraction can take place behind a closed hood sash.

11.6.8. 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.

***Note**: Mechanical techniques could include any or all of the following: running sample through glass-wool, centrifuge, add 10mLs of 10N NaOH, or let sit longer for separation. See section 11.6 for more details.*

11.6.9. Drain the DCM phase back into the sample bottle.

11.6.10. Extraction is repeated two additional times with 100 mL each of methylene chloride. Drain the DCM after each extraction back into the sample bottle.

11.6.11. After the third extraction, dry the extract with sodium sulfate: Place glass wool in a pre-cleaned filter funnel. Then add in Na2SO4. Rinse glass wool and Na2SO4 with DCM. Pour extract through Na2SO4 to remove any water. Collect the extract into a 500 mL round bottom flask.

11.6.12. Rinse the Na2SO4 in the filter funnel with fresh DCM and collect into round bottom flask.

11.6.12.1. Concentrate on a rotary evaporator at 75ºC and no vacuum at 120-150 RPM. Proceed to Section 11.9 for cleanup options as needed.

***Note**: If no lower column cleanup is required do not add dodecane as a solvent keeper.*

11.6.13. Reweigh the empty bottle and record the sample weight. Record the sample weight to the nearest 1 gram.

11.7. Air train sample – based on Method CARB 428 (Option 1).

There are two options for the preparation of air train sample. The first option is all fractions combined for a single analysis, and the second is to prepare and analyze separate front and back-half extracts. Below is Option 1, the Single composite of all air train fractions.

11.7.1. Preparation of Extraction Thimble

Place a glass extraction thimble into the Soxhlet apparatus, charge the apparatus with toluene, and reflux for a minimum of 3 hours. Remove the toluene and discard it. Remove the extraction thimble system and place it in a glass beaker for air drying and for catching rinses. Alternatively, the thimbles may be used directly from the box if the thimbles have been shown to be free

of PCBs or interfering compounds at or above the project required reporting limits.

11.7.2. Preparation of Container No. 1 (filter)

Place the filter and all particulate matter from Container No. 1 into the cleaned extraction thimble.

11.7.3. Preparation of Adsorbent Cartridge

Suspend the adsorbent module directly over the extraction thimble containing the associated filter. The glass frit of the module should be positioned up. Add a small amount of acetone to the module and discharge the XAD-2 resin into the thimble by using a nitrogen blowing apparatus. Rinse the walls of the module with acetone to remove any excess resin, collect the rinse in the thimble. Rinse the module with toluene and collect the rinses in the thimble containing the XAD-2 resin and filter.

11.7.4. Preparation of Container No. 2 (Front and Back-half Acetone and Methylene Chloride rinses)

Transfer the solvent to a round bottom flask. Rinse the sample container 3 times with methylene chloride and add these to the round bottom flask. Concentrate the solvent to a volume of about 1-5 mL using a rotary evaporator, at a temperature of 60ºC and <10 inches of vacuum at 120-150 RPM.

11.7.5. Preparation of Container No. 3 (Front and Back-half Toluene rinses)

This step can be done one of two ways as dictated by the client:

11.7.5.1. Option 1- Add the toluene rinse to the round bottom flask previously used to concentrate Container No. 2. Rinse the samples container three times with toluene and add the rinses to the round bottom flask. Concentrate the solvent to about 1-5 mL using a rotary evaporator at a temperature of 70ºC and 25 inches of vacuum. Add the rinses to the Soxhlet containing the corresponding filter and XAD resin portions.

11.7.5.2. Option 2- Add the toluene rinse to a pre-cleaned round bottom flask. Rinse the sample container three times with toluene and collect rinses in the round bottom flask. Concentrate the solvent to about 1-5 mL using a rotary evaporator at a temperature of 70°C and 25 inches of vacuum. Do not add the concentrated solvent to the Soxhlet containing the corresponding filter and XAD, instead add 100 uL of Isotope Dilution Anayltes (IDA) and analyze as a separate sample.

11.7.6. Extraction and Concentration.

11.7.6.1. Surrogate compounds should have been added to the adsorbent cartridge prior to sample collection.

11.7.6.2. Place the extraction thimble containing the filter, XAD resin, and solvent rinses into the Soxhlet extractor. Rinse the beaker three times with toluene and add the rinses to the Soxhlet. Add toluene to the round bottom flask until it is about 2□3 full. Add several Teflon boiling chips to the round bottom flask and assemble the Soxhlet apparatus.

11.7.6.3. Add 100 uL of IDA standard to the thimble, connect the condenser, and adjust the heat source to cause the extractor to cycle approximately three times per hour for a minimum of 16 hours.

***Note**: Additional 100 uL volume of IDA standard may be added per split (i.e. ½ archive = 200 uL total; 1/3 archive, 1/3 Method 0023A, 1/3 1668A = 300 uL; etc.)*

11.7.6.4. Allow the Soxhlet to cool and 100 uL of dodecane may be added as a solvent keeper if extracts will go through acid alumina cleanup.

11.7.6.5. Concentrate the extract to near dryness using a rotary evaporator at 70ºC and 25 inches of vacuum. Proceed with clean up or splitting of the samples based on client requirements.

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.

Option 2- Separate Front and Back-half fractions. The Front-half consists of the filter and front-half solvent rinses, the Back-half consists of the XAD resin and Back-half rinses.

11.7.7. Preparation of Container No. 1 (filter)

Place the filter and all particulate matter from Container No. 1 into the cleaned extraction thimble.

11.7.8. Preparation of the Front-half acetone and methylene chloride rinse

Transfer the solvent to a round bottom flask. Rinse the sample container 3 times with methylene chloride and add these to the round bottom flask. Concentrate the solvent to a volume of about 1-5 mL, using a rotary, at a temperature of 60ºC and no vacuum at 120-150 RPM. Quantitatively transfer the solvent to the Soxhlet apparatus containing the corresponding filter fraction.

11.7.9. Preparation of the Front-half toluene rinse

Add the toluene rinse to the round bottom flask previously used to concentrate the acetone□methylene chloride rinses. Rinse the samples container three times with toluene and add the rinses to the round bottom flask. Concentrate the solvent to about 1-5 mL using a rotary evaporator at a temperature of 70ºC and 25 inches of vacuum at 120-150 RPM. Add the rinses to the Soxhlet containing the corresponding filter and acetone□methylene chloride fractions.

11.7.10. Preparation of Adsorbent Cartridge

Suspend the adsorbent module directly over a pre-cleaned extraction thimble. The glass frit of the module should be positioned up. Add a small amount of acetone to the module and discharge the XAD-2 resin into the thimble by using a nitrogen blowing apparatus. Rinse the walls of the module with acetone to remove any excess resin, collect the rinse in the thimble. Rinse the module with toluene and collect the rinses in the thimble containing the XAD-2 resin.

11.7.11. Preparation of the Back-half acetone and methylene chloride rinse.

Transfer the solvent to a round bottom flask. Rinse the sample container 3 times with methylene chloride and add these to the round bottom flask. Concentrate the solvent to a volume of about 1-5 mL, using a rotary, at a temperature of 60ºC and no vacuum at 120-150 RPM. Quantitatively transfer the solvent to the Soxhlet apparatus containing the corresponding XAD resin fraction.

11.7.12. Preparation of the Back-half toluene rinse

Add the toluene rinse to the round bottom flask previously used to concentrate the acetone□methylene chloride rinses. Rinse the samples container three times with toluene and add the rinses to the round bottom flask. Concentrate the solvent to about 1-5 mL using a rotary evaporator at a temperature of 70ºC and 25 inches of vacuum at 120-150 RPM. Add the rinses to the Soxhlet containing the corresponding XAD resin and acetone□methylene chloride fractions.

11.8. There are several useful methods to decrease or eliminate emulsion in a□ueous 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 1□1 Na□□□□$_2$□ solution to change the p□ enough to disrupt the emulsion phase, which works 90□ of the time.

11.8.1. □sing the 10N Na□□ solution to decrease emulsions□

11.8.1.1. Add 10 mL of 10N Na□□ to the extract in the sample container. Secure the lid to the bottle and shake the container for 5-10

seconds. If emulsion is dissolved proceed to section 11.6.1.2. If the emulsion persists add additional 10 mL aliquots of 10N NaOH until the emulsions are dissolved. Pour the solution with DCM back into the same 2.0 L separatory funnel and drain the DCM phase through Na_2SO_4 into a 500mL round bottom. Empty the aqueous waste into the LL waste drum.

11.8.1.2. Concentrate the extract on a rotary evaporator at 65ºC and no vacuum at 120-150 RPM. Proceed to Section 11.9 for cleanup options as needed.

11.9. Cleanup: Depending on the complexity of the matrix, the following clean-up procedures may be employed as necessary or per client requirements.

11.9.1. Add 100 uL of Surrogate Standard to each extract.

11.9.2. Option C (Acid only) (Required for all solid and tissue matrices):

11.9.2.1. 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 sulfuric acid layer (bottom). Repeat the acid washing until no color is visible in the acid layer (perform a maximum of four acid washings).

11.9.2.2. Neutralize the extract with 10 mL of DI water. Shake for two minutes. Remove and discard the aqueous layer (bottom).

11.9.2.3. Dry the extract with sodium sulfate: Place glass wool in a pre-cleaned filter funnel. Then add in Na_2SO_4. Rinse glass wool and Na_2SO_4 with Hexane. Pour extract through Na_2SO_4 to remove any water. Collect the extract into a round bottom flask.

11.9.2.4 Reduce the volume to about 2 mL on a rotovap and proceed with any needed cleanups

***Note**: Additional rinses may be performed against sodium hydroxide or reagent water if deemed necessary by the laboratory management.*

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.

11.9.3. Silica Gel Column Cleanup. **(Required for all samples)**

11.9.3.1. Add a plug of glass wool to a 20 mm ID disposable column and add 2 grams of activated silica gel, 4 grams of acidic silica gel, 2 grams of activated silica gel, and 1 cm of sodium sulfate.

11.9.3.2. Pre-rinse the column with at least 20 mL of methylene chloride followed by at least 20 mL of hexane.

11.9.3.3. Add the sample extract to the column, and rinse the sample container with 2 x 2 mL hexane rinses. Add the rinses to the column.

11.9.3.4. Elute the column with hexane and collect a total of 90 mL of hexane.

11.9.3.5 Reduce the volume to about 2 mL under nitrogen and proceed with alumina column cleanup if deemed necessary.

11.9.4. Alumina Column Cleanup. **(Optional)**

11.9.4.1. Put a small plug of glass wool in a 10 mm ID column and add 10 grams of acid alumina followed by 1 cm of sodium sulfate.

11.9.4.2. Pre-rinse the column at least 20 mL of hexane.

11.9.4.3. Transfer the sample (in 2 mL hexane) to the acid alumina column. Rinse sample flask with 2 x 2 mL hexane, transfer to acid alumina column.

11.9.4.4. Elute the column with 10 mL of hexane and discard.

11.9.4.5 Elute the column with 30 mL of 1:1 methylene chloride:hexane and collect the extract into a culture tube.

11.9.4.6 Reduce the volume to about 2 mL under nitrogen and proceed with any needed cleanups or proceed to Section 11.10.

11.9.5 Sulfur Removal with Elemental Mercury. **(Optional Cleanup)** The entire sample is used for this process. If the sulfur concentration is such that crystallization occurs in the concentrated extract, residual rings of sulfur appear, centrifuge the extract to settle the crystals, and carefully draw off the sample extract with a disposable pipette, leaving the excess sulfur in the centrifuge tube. Transfer the extract to a clean concentrator tube before proceeding with further sulfur cleanup.

***Note**: Use mercury sparingly in order to minimize exposure and disposal costs.*

11.9.5.1. Transfer of sample extract into a clean concentrator tube or Teflon sealed vial.

11.9.5.2. Add one to three drops (about 0.25mL) of mercury to the extract vial and seal.

WARNING: Only nitrile gloves may be used when working with mercury and organic solvents at the same time.

11.9.5.3. Shake well for 15-30 seconds.

11.9.5.4. If black precipitate forms, sulfur was present. Centrifuge, then transfer the supernatant to a clean test tube and repeat steps 11.9.5.2 and 11.9.5.3. Do this until relatively little precipitate remains, or a maximum of five sulfur cleanups has been performed.

WARNING: □o NO□ centri□uge glass tubes containing mercury. □ue to the density o□the mercury□the test tube could break in the centri□uge.

11.9.5.1. If no precipitate is present, remove the extract from the mercury using a disposable pipette and transfer to a clean vial. □otate the mercury waste out of the laboratory to the □a□ardous □ aste storage area for lab pack disposal.

11.9.5.2. □roceed to Section 11.10 with the extract.

11.10. □inal □olume

11.10.1. □educe the sample volume under nitrogen to approximately 1 mL.

11.10.2. Add 20 uL of internal standard in dodecane to a 4 mL vial.

11.10.3. Transfer the sample contents to the vial containing the internal standard and rinse 2□ with 1ml hexane.

11.10.4. □educe under nitrogen to 20 uL.

11.10.5. Transfer to an in□ection vial and cap.

11.10.□ If analysis is not to be performed the same day store the vials in the dark at less than -10°C.

11.10.□ The extract is ready for analysis by □ S-ID-0013.

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13.1. □ ethod Detection Limit

□ach laboratory must generate a valid method detection limit for each analyte of interest. The □ DL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 C□□ □art 13□, Appendix □, and further defined in □ S-□A-000□. □ DL are available in the □uality Assurance department.

13.2. Initial Demonstration

□ach analyst must make a one time initial demonstration of capability for each individual method. Demonstration of capability for both soils and water matrices is re□uired. This re□uires analysis of □C check samples containing all of the standard analytes for the method. □or some tests it may be necessary to use more than one □C check mix to cover all analytes of interest.

13.2.1. □our ali□uots of the □C check sample are analy□ed using the same procedures used to analy□e samples, including sample preparation. The concentration of the □C check sample should be e□uivalent to a mid level calibration standard.

13.2.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these results with the historical acceptance criteria.

13.2.3. If any analyte does not meet the acceptance criteria, the test must be repeated. □nly those analytes that did not meet criteria in the first test need to be evaluated. □epeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3. Training □ualification

The group□team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the re□uired experience.

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It is TestAmerica□s policy to evaluate each method and look for opportunities to minimi□e waste generated (i.e., examine recycling options, ordering chemicals based on □uantity needed, preparation of reagents based on anticipated usage and reagent stability). □mployees must abide by the policies in Section 13 of the Corporate □nvironmental □ealth and Safety □ anual (C□ -□-□ -001) for □□ aste □ anagement and □ollution □revention.□

14.1. All waste will be disposed of in accordance with □ederal, State and Local regulations. □ here reasonably feasible, technological changes have been implemented to minimi□e the potential for pollution of the environment.

14.2. Do not allow waste solvent to evaporate in fume hoods. All solvent waste is stored in capped containers unless transfers are being made.

14.3. The use of rotary evaporator systems to concentrate sample extracts significantly reduces the solvent released into the atmosphere.

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□ aste management practices are conducted consistent with all applicable rules and regulations. □xcess reagents, samples, and method process wastes are disposed of in an accepted manner. □ aste description rules and land disposal restrictions are followed. □ aste disposal procedures are incorporated by reference to S□□ □ S-□□S-0001. The following waste streams are produced when this method is carried out.

15.1. Assorted solvent and methylene chloride from glassware rinsing, glass fiber and sodium sulfate pre-rinsing, and extract rotary evaporator concentration. Collect the waste solvents in tripours during use. □mpty the tripours into a 1-liter to 4-liter carboy

at the fume hood. □ hen the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel solvent drum in the □3 closet. □ hen the drum is full to between two and six inches of the top, or after no more than □5 days, move the steel drum to the waste collection area for shipment.

15.2. □ iscellaneous disposable glassware, chemical resistant gloves, bench paper and similar materials that may or may not be contaminated□ha□ardous. □lace contaminated materials into a yellow landfill contaminated lab trash bucket. □ hen the bucket is full or after no more than one year, tie the plastic bag liner shut and put the dry lab trash into the appropriate steel dry lab trash collection drum in the □3 closet. □ hen the drum is full or after no more than □5 days, move it to the waste collection area for shipment.

15.3. Disposable spatulas and other items, including bench paper, that have come into contact with unextracted soil, extracted □□□ filters, sodium sulfate, silica gel, alumina, carbon □AD-2 resin, paper funnel filters, glass wool, thimbles, fish□crawfish, ash and soil contaminated with various solvent and eluents□dump the materials into an orange contaminated lab trash bucket. □ hen 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 (incineration) in the □3 closet. □ hen the drum is full or after no more than □5 days, move it to the waste collection area for shipment.

15.4. □xtracted a□ueous samples contaminated with methylene chloride and□or other organic solvents are collected at the fume hood in a 5-gallon or smaller carboy. If the samples are not at a neutral p□, add small □uantities of sodium bicarbonate to bring the waste to neutral. Stir well. □nce neutrali□ed, immediately pour the carboy contents into a blue plastic LL□ drum in the □3 closet. □ hen full to between two and six inches of the top, or after no more than □5 days, move the LL□ drum to the waste collection area for shipment.

15.5. Contaminated sulfuric acid used during □ption C extract clean-up. Collect the used sulfuric acid in empty, 2.5-liter, plastic coated □ars. □ hen full or after no more than one year, whichever comes first, transfer these □ars to the waste collection area for shipment.

15.□ Contaminated mercury used during sulfur clean-up. Collect the used mercury in empty □ounce plastic □ars. □ hen full or after no more than one year, transfer the □ar to the waste collection area for shipment.

□□. R□□□R□N□□□□RO□□R□□□R□N□□□

1□1. State of California Air □esources □oard □ ethod 42□□Determination of □olychlorinated Diben□o-p-dioxin (□CDD), □olychlorinated Diben□ofuran (□CD□), and □olychlorinated □iphenyl □missions from Stationary Sources, September 12, 1990.

1□2. □□A □ ethod 1□□□□Toxic polychlorinated □iphenyls by Isotope Dilution □igh □esolution □as Chromatography□□igh resolution □ ass Spectrometry, □ arch 199□.

1□3. □ ethod 1□□□, □evision A (□ ethod 1□□□A)□Chlorinated □iphenyl Congeners in □ ater, Soil, Sediment and Tissue by □□□C□□□□ S, August 2003.

1□4. □ ethod 1□□□C□Chlorinated □iphenyl Congeners in □ ater, Soil, Sediment, □iosolids, and Tissue by □□□C□□□□ S, April 2010.

1□5. □ S-□□S-0001, □ aste Disposal

1□□. □ S-□□-0011, Cleaning of □lassware

1□□. □ S-□□-0013, □ercent □ oisture

1□□. □ S-□A-0004, Calibration of Autopipettors, Autodispensers and □olumetric Containers

1□9. □ S-□A-000□, □ DL and IDL

1□10. □ S-□A-0041, Calibration and Calibration Check of □alances

1□11. □ S-□ I-001□, Tissue Sample □andling and □xtraction

1□12. C□ -□-□ -001, Corporate □nvironmental □ealth and Safety □ anual

1□13. □ S-□□□S-0002, Sacramento Addendum to Corporate □nvironmental □ealth and Safety □ anual.

□□. □ □□□O□ □ O□I□I□A□ION□

1□1. Deviations from reference □ ethod 1□□□A and □ ethod 1□□□C.

1□1.1. A□ueous samples with □1□ solids are extracted as received, by separatory funnel. Samples with high solids content, □1□ solids, are either shaken well to thoroughly mix the sample followed by extraction by separatory funnel, or separated into two different samples for extraction and analysis, dependent upon client re□uest.

1□1.2. A□ueous samples are extracted with 100 mL ali□uots of □ ethylene chloride rather than □0 mL ali□uots per the referred methods.

1□1.3. Solid samples are extracted as received, with □-moisture determination performed simultaneously. Dry weight correction to results is applied at the time of data reporting.

1□1.4. Tissue samples are extracted using toluene.

1□1.5. The sample extract cleanup procedures are all optional, except where noted, and based on analyst □udgment. The routine cleanup procedures include acidic silica gel and acidic alumina cleanup columns. The laboratory does not routinely use □lorisil, □□C, or anthropogenic column cleanup procedures.

1□1.□ Soxtherms are used as an option of extracting that is not listed in the source method. The soxtherm are used instead of soxhlets and microwave.

1□1.□ Alumina is used as a clean up option which is not listed.

1□1.□ □g is used as a clean up option which is not listed.

1□1.9. Carbon cleanup is not used as a cleanup option.

1□1.10. □lorisil is not used as a cleanup option.

□□. A□□A□□□ □N□□

1□1. Table 1 □Composition of the Sample □ortification Solutions

1□2. Table 2 □Instrument Calibration Solution Concentrations

1□3. Table 3 □Composition of the □atrix Spike □ortification Solution

□□. R□□I□ION □I□□OR□

19.1. □ S-ID□-0013, □evision 4.3 □ffective 0□0□201□

19.1.1. Added section 5.1.11, □□ hen dispensing methylene chloride or any other solvent from the Cycletainer, do not lock the dispenser into the open position and walk away to perform any other tasks. □ hile you may lock the dispenser open for steady flow to reduce the ergonomic impact, you must stay there with the dispenser and container being filled to prevent it from overflowing.□

19.1.2. Section □4.1.1 revised to, □Activated silica gel - Silica □el □0-□isher S□25-212, 230-400 mesh, rinsed with methylene chloride, baked at 1□5-195□C- for at least 12 hours before use. Store at 125-145□C in a covered flask or bottle.□

19.1.3. Section 11.3.3.2 changed, □□0 □C□to □□0 □C□

19.1.4. Section 11.3.5 changed, □□0 □C□to □□0 □C□

19.1.5. Section 11.3.5.1 changed, □□0 □C□to □□0 □C□

19.1.□ Section 11.□12.1 changed, □□5 □C□to □□5 □C□.

19.1.□ Section 11.9.2.3 changed, □DC□ □to □hexane□.

19.1.□ Section 15.3 changed to, □Disposable spatulas and other items, including bench paper, that have come into contact with unextracted soil, extracted □□□ filters, sodium sulfate, silica gel, alumina, carbon □AD-2 resin, paper funnel filters, glass wool, thimbles, fish□crawfish, ash and soil contaminated with various solvent and eluents□dump the materials into an orange contaminated lab trash bucket. □ hen 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 (incineration) in the □3 closet. □ hen the drum is full or after no more than □5 days, move it to the waste collection area for shipment.□

19.1.9. □emoved revision history prior to 2015, it can be found in previous versions of this S□□.

19.1.10. □ditorial changes.

19.2. □ S-ID□-0013, □evision 4.2 □ffective 02□0□201□

19.2.1. Added Section 11.□1.1, □□or leachate samples (including an ali□uot of the leachate blank), measure 200 mL of leachate into a clean 1 L sample bottle. Add approximately □00 mL of reagent water to the leachate. The sample volume in TALS for leachates is 200 mL.□

19.2.2. □ditorial changes.

19.3. □ S-ID□-0013, □evision 4.1 □ffective 0□2□201□

19.3.1. Section 11.9.2.1 □Changed 40 mL of concentrated □$_2$S□$_4$ to 20 mL. (□artition the extract in 50-125 mL of hexane against 20 mL concentrated □$_2$S□$_4$ in a separatory funnel.).

19.3.2. Section 11.9.2.3 □Added □Collect the extract into a round bottom flask.□at end of paragraph.

19.3.3. □ditorial changes.

19.4. □ S-ID□-0013, □evision 4.0, □ffective 0□31□2015

19.4.1. □pdated Copyright statement (cover).

19.4.2. Added Section □3 - All extracts must be stored capped and in the dark at □-10 °C.

19.4.3. Section 11 underwent a ma□or revision to incorporate current laboratory practices and encompass test similar with other S□□s.

19.4.4. Added Sections 1□5 □1□13 listing S□□s that are referenced in this S□□.

19.4.5. Added Sections 1□1.□□1□1.10 to list method modifications.

19.4.□ □ditorial changes.

TABLE I
Composition of the Sample Fortification Solutions

	Isotope Dilution Analyte Concentration (pg/uL in Isooctane)	Recovery Standard Solution (pg/uL in Dodecane) (Internal Standard)	Clean-up Recovery Standard Solution (Surrogate) (pg/uL in Isooctane)
$^{13}C_{12}$-2-MonoPCB (1)	20	--	--
$^{13}C_{12}$-4-MonoPCB (3)	20	--	--
$^{13}C_{12}$-2,2-DiPCB (4)	20	--	--
$^{13}C_{12}$-2,5-DiPCB (9)	--	100	--
$^{13}C_{12}$-4,4'-DiPCB (15)	20	--	--
$^{13}C_{12}$-2,2',6'-TriPCB (19)	20	--	--
$^{13}C_{12}$-2,4,4'-TriPCB (28)	--	--	100
$^{13}C_{12}$-3,4,4'-TriPCB (37)	20	--	--
$^{13}C_{12}$-2,2',5,5'-TetraPCB (52)	--	100	--
$^{13}C_{12}$-2,2',6,6'-TetraPCB (54)	20	--	--
$^{13}C_{12}$-3,3',4,4'-TetraPCB (77)	20	--	--
$^{13}C_{12}$-3,4,4',5-TetraPCB (81)	20	--	--
$^{13}C_{12}$-2,2',4,5,5'-PentaPCB (101)	--	100	--
$^{13}C_{12}$-2,2',4,6,6'-PentaPCB (104)	20	--	--
$^{13}C_{12}$-2,3,3',4,4'-PentaPCB (105)	20	--	--
$^{13}C_{12}$-2,3,3'5,5'-PentaPCB (111)	--	--	100
$^{13}C_{12}$-2,3,4,4',5-PentaPCB (114)	20	--	--
$^{13}C_{12}$-2,3',4,4'5-PentaPCB (118)	20	--	--
$^{13}C_{12}$-2',3,4,4',5-PentaPCB (123)	20	--	--
$^{13}C_{12}$-3,3',4,4',5-PentaPCB (126)	20	--	--
$^{13}C_{12}$-2,2',3',4,4',5'-HexaPCB (138)	--	100	--
$^{13}C_{12}$-2,2',4,4',6,6'-HexaPCB (155)	20	--	--
$^{13}C_{12}$-2,3,3',4,4',5-HexaPCB (156)	20	--	--
$^{13}C_{12}$-2,3,3',4,4',5'-HexaPCB (157)	20	--	--
$^{13}C_{12}$-2,3',4,4',5,5'-HexaPCB (167)	20	--	--
$^{13}C_{12}$-3,3',4,4',5,5'-HexaPCB (169)	20	--	--
$^{13}C_{12}$-2,2',3,3',5,5',6-HeptaPCB (178)	--	--	100
$^{13}C_{12}$-2,2',3,4',5,6,6'-HeptaPCB (188)	20	--	--
$^{13}C_{12}$-2,3,3',4,4',5,5'-HeptaPCB (189)	20	--	--
$^{13}C_{12}$-2,2',3,3',4,4',5,5'-OctaPCB (194)	--	100	--
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-OctaPCB (202)	20	--	--
$^{13}C_{12}$-2,3,3',4,4'5,5',6-OctaPCB (2025	20	--	--
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-NonaPCB (206)	20	--	--
$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-NonaPCB (208)	20	--	--
$^{13}C_{12}$-DecaPCB (209)	20	--	--

TABLE 2
Instrument Calibration Solution Concentrations

Compound	I□□AC □(s)	Concentration (pg□uL in Dodecane)				
		CC1	CC2	CC3	CC4	CC5
Unlabelled Analytes						
MonoPCB	1,3	1	5	50	400	2000
DiPCB	4,15	1	5	50	400	2000
TriPCB	19,37	1	5	50	400	2000
TetraPCB	54,77,81	1	5	50	400	2000
PentaPCB	104,105,114,118,123,126	1	5	50	400	2000
HexaPCB	155, 156,157,167,169	1	5	50	400	2000
HeptaPCB	188,189	1	5	50	400	2000
OctaPCB	202,205	1	5	50	400	2000
NonaPCB	206,208	1	5	50	400	2000
DecaPCB	209	1	5	50	400	2000
Internal Standards						
^{13}C-MonoPCB	1,3	100	100	100	100	100
^{13}C-DiPCB	4,15	100	100	100	100	100
^{13}C-TriPCB	19,37	100	100	100	100	100
^{13}C-TetraPCB	54,77, 81	100	100	100	100	100
^{13}C-PentaPCB	104,105,114,118,123,126	100	100	100	100	100
^{13}C-HexaPCB	155,156,157,167,169	100	100	100	100	100
^{13}C-HeptaPCB	188,189	100	100	100	100	100
^{13}C-OctaPCB	202,205	100	100	100	100	100
^{13}C-NonaPCB	206,208	100	100	100	100	100
^{13}C-DecaPCB	209	100	100	100	100	100
Recovery Standards						
^{13}C-DiPCB	9	100	100	100	100	100
^{13}C-TetraPCB	52	100	100	100	100	100
^{13}C-PentaPCB	101	100	100	100	100	100
^{13}C-HexaPCB	138	100	100	100	100	100
^{13}C-OctaPCB	194	100	100	100	100	100
Cleanup Recovery Standards						
^{13}C-TriPCB	28	100	100	100	100	100
^{13}C-PentaPCB	111	100	100	100	100	100
^{13}C-HeptaPCB	178	100	100	100	100	100

TABLE 3
Composition of the Matrix Spike Fortification Solution

Compound (Unlabelled)	IUPAC #(s)	Concentration (pg/uL in isooctane)
MonoPCB	1,3	20
DiPCB	4,15	20
TriPCB	19,37	20
TetraPCB	54,77,81	20
PentaPCB	104,105,114, 118,123,126	20
HexaPCB	155, 156,157,167,169	20
HeptaPCB	188,189	20
OctaPCB	202,205	20
NonaPCB	206,208	20
DecaPCB	209	20

Title: Preparation of Samples for Analysis of Polychlorinated Dioxins and Furans for Analysis HRGC/HRMS

[Methods 8290, 8290A & TO-9A]

Approvals (Signature/Date):

06/18/2019 Robert Hrabak Date Technical Manager	06/18/2019 Joe Schairer Date Health & Safety Manager / Coordinator
06/14/2019 Lisa Stafford Date Quality Assurance Manager	06/18/2019 Chris Williams Date Laboratory Director

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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. □ine 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 P□C 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. □ilning 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. □ote: 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 Hyflex □BR or MAPA blue latex cut-resistant gloves over chemically resistant gloves when assembling and disassembling glassware.

5.1.4. Eye protection that satisfies A□SI □87.1, 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. □itrile 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 in□uries. Laboratory associates performing these procedures are

in the best position to realize when they are at risk for these types of in□uries. 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. □ent 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 ma□or 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.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
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.
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. □itrogen 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 ± 2□C.

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 90 mm C18, or equivalent.

6.19. 30x100 mm glass fiber thimble or equivalent.

6.20. Dean-Stark trap, 5 or 10 mL, with T-oints, 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 E-8 probe or equivalent.

6.30. Glass wool, extracted with methylene chloride, dried and stored in a clean glass ar.

6.31. □acuum extraction device for solid phase extraction, 1 Liter glass funnel with 90mm filter disc holder with a vacuum source, □ontes 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 □ HCl, ACS Grade

7.1.4. 10 □ □aOH, certified (Fisher Scientific S5255 or equivalent)

7.1.5. Silica gel – 230-400 mesh (Fischer Scientific , P/□ 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. □egetable Oil (for tissue extraction only), Mazola or other suitable oil, demonstrated to be free of interferences.

7.1.8. Diatomaceaous earth – Hydromatrix (Agilent P/□ 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 - IC□ or equivalent, activated as necessary.

7.3.2. Basic Alumina - IC□ or equivalent. □o 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 □ar. 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 20 mL 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 ≤ 6□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. (□ote that $^{13}C_{12}$ -OCDF is not present in the solution.)

7.8. □ative Spike Standard

Also known as the Target Analyte Spike, Matrix Spike or □ative 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 ≤ 6□C.

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 □on-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 □ 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 □on-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 □D, unless otherwise specified by the client. □ote any actions in the narrative.

9.3. The assessment of matrix effects on method performance, as required by □ELAP, 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 □lanks

9.5.1. □ach 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. □xtract 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 S□P 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. □ust 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 □CDF) 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, □C samples, and included in all calibrations.

9.8.3. For each sample and □C 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 HR□C/HRMS instrument analysis.

9.10. Recommended Corrective Actions and Troubleshooting Steps

- □erify 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. □n a daily basis, calibrate any balance to be used in accordance with S□P WS-□A-0041.

10.2. □n a daily basis, calibrate any autopipettor to be used in accordance with S□P WS-□A-0004.

11. PROCEDURE

11.1. □ne 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 □A 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 S□P 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. □ecause 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 2-10 g of sample aliquot into a clean glass jar.

11.3.2.2. Add 1.0 mL 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 0.01 g. 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 µL of the TA spike as well.

***Note**: This spike level will give a final concentration of 200 pg/g (based on a 10 g 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 0.01 g. 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 10 g 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 (°C)
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 10 g of □a$_2$S□□and 5 g of diatomaceous earth.

11.7.□. Add 1 mL of deioni□ed water to each sample and □C in Teflon sample chamber. The number of sample chambers should match the number of field samples and □C samples.

11.7.5. Spi□e according to Section 11.□5.1, 11.□5.2, and 11.□5.3.

11.7.□. 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. □nto 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.□. Add flared extraction vessel covers to each extraction vessel containing sample and □C aliquots.

11.7.□.1. □t 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.□.2. □or 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.□. 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.□.1. □or the representative sample created in 11.7.□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.□2 and 11.7.□.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. □un the following program for sample extraction:

Step	Time (min)	Power (W)	Temperature (°C)
1	30	1200	145
2	30	1200	145
3	10	0	(ambient)

11.7.13. □ollowing extraction filter the extract through □a$_2$S□$_4$ and collect in a round bottom flas□, rinse twice more with approximately 5 mL to 10 mL of toluene for a total of about 50 mL.

11.7.1□. Add approximately 100 µL C$_{1□}$ to each round bottom flas□if □ lipid is not needed.

11.7.15. Proceed to Section 11.1□.

11.□. Extract Splitting (Wipes)

Wipe extracts prepared using any extraction technique are split prior to further wor□up, to permit an archive aliquot, or analysis by an additional method. The samples and □Cs in the batch should be spi□ed with 2□ of the nominal spi□ing amount to achieve a 1□ spi□ing amount after splitting. □nce the extract has been concentrated using the rotovap or Turbovap, proceed as follows:

11.□.1. Add approximately 1 mL of hexane or toluene to rinse the sides of the round bottom flas□. □sing a pipette, withdraw the sample from the round bottom flas□and transfer the liquid into a test-tube. □se additional amounts of solvents to rinse the flas□. Transfer all the liquid into the test-tube. Ensure that all traces of sample in the round bottom flas□have been thoroughly rinsed from all surfaces. Bring the sample volume to □0 mL or 10.0 mL (or appropriate volume) with the addition of rinse solvent.

11.□.2. □pon completion of the rinsing, cap the test tube and sha□e vigorously. Ta□e □ 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.□.2.1. □f only one analysis is required, then □ of the sample is archived and the other half is analy□ed.

11.□.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.□. Aqueous Samples (liquid/liquid extraction).

11.□.1. When setting up the glassware for a batch, for each sample label one separatory funnel with the sample ID.

11.□.2. If any samples has more than 10□ solid (□1 inch), set sample aside and contact your supervisor or PM.

11.□.3. Weigh the sample in the bottle on the top loading balance to the nearest centigram (0.01g), and record the mass.

11.□.3.1. For leachate samples (including an aliquot of the leachate blan□), measure 200 mL of leachate into a clean 1 L sample bottle. Add approximately □00 mL of reagent water to the leachate. The sample volume in TALS for leachates is 200 mL.

11.□.□. For each sample, add 1 mL of IDA standard to the sample in the original container.

11.□.5. Create a blan□ and LCS by adding 1 L of laboratory reagent water to a 1 L A□B. Spi□e 50 □L of the TA standard into the LCS container. Spi□e each one with 1 mL of IDA.

11.□.□. Pour the entire sample (approximately 1 L) into a 2 L separatory funnel that is labeled with the sample ID.

11.□.7. Add 100 mL methylene chloride to the sample bottle, seal, and sha□e for 30 seconds to rinse the inner surface. Transfer the solvent to the separatory funnel.

11.□.□. Extract the samples by sha□ing each funnel for two minutes with periodic venting.

Warning: □eparatory funnel extraction with methylene chloride is a high□risk activity. □ressure 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 □olume of the sol□ent layer□the analyst must employ mechanical techni□ues to complete the phase separation.

11.9.10. □epeat the e□traction two additional times with 100 m□ of methylene chloride each time.

11.9.11. □etermine the original sample □olume by re-weighing the sample bottle. □ecord the sample □olume to the nearest centigram □0.01 g□.

11.9.1□. □ry e□tract with sodium sulfate□ □lace glass wool in a precleaned filter funnel. □inse glass wool with methylene chloride and load funnel with □a□□□. □our e□tract through □a□□□ to remo□e water. □inse □a□□□ with fresh methylene chloride and collect in round bottom flas□.

11.9.1□. □ransfer the e□tract to a □00 m□ round-bottom pre□iously labeled with the sample I□ □then add appro□imately 100 µ□ of tetradecane.

11.9.1□. □erform macro-concentration as detailed in □ection 11.1□.

11.10. A□ueous □amples □ia solid phase e□traction.

11.10.1. □ eigh the sample in the bottle on the top loading balance to the nearest centigram □0.01g□□and record the mass.

11.10.□. □reate a blan□and □□□ by adding 1 □ of laboratory reagent water to □ additional 1 □ bottles.

11.10.□. □or each sample□add 1 m□ of I□A standard solution into the sample in the bottles. □ach ali□uot of spi□e mi□ture is added similarly.

11.10.□. □o the □□□□add □0 □□ of the □A standard.

11.10.□. □repare the □1□e□traction discs by first soa□ing them in toluene for at least □ minutes.

11.10.□. Assemble the filter holder and □acuum filtration flas□and place the e□traction disc onto the filter holder. □lace a □□-□ filter on top of the e□traction disc. If the sample has a large amount of particulates a □□-□ filter can be placed on top of the □□-□ filter. Alternati□ely□a □□ □-1□0 filter can be used in place of the two filters.

11.10.□. □lace the filtering funnel onto the disc holder and clamp it in place.

11.10.□. □inse the filter and discs with appro□imately 1□m□ of toluene and allow it to soa□ for about a minute. Apply □acuum and draw the toluene through the discs. □epeat the wash step using about 1□m□ of acetone. Apply □acuum

and draw the acetone through the discs.

11.10.9. Rinse the filter and discs with approximately 1 mL 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 – 2 mm layer of solvent on top of the discs.

11.10.10. Rinse twice with about 10 mL of reagent water, leaving a 1 – 2 mm 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 (11.6) or sonotherm (11.7) 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 (0.01 g).

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 break up the emulsions or to transfer the sample into centrifuge tubes and centrifuge at approximately 2000 RPM. The most useful method is to use a 10 % NaOH 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 5 and 9. If the pH is greater than 9, consult the supervisor and client for instructions.

11.11.2. Pour approximately 10 mL of the 10% 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 DCM waste drum.

11.11.□. □our the solution with methylene chloride bac□into the same □□ separatory funnel and drain the methylene chloride phase through □a□□□into a □00 m□ round-bottom flas□.

11.11.□. □mpty the a□ueous waste into the □□□ waste drum.

11.11.□. □erform macro-concentration as detailed in □ection 11.1□.

11.1□. □ilter□□□□ □amples

11.1□.1. □lace the glass slee□e containing the □□□and the □uart□□iber □ilter into the pre-cleaned □o□hlet e□tractor charged with toluene.

11.1□.□. Add □m□ □□000 pg□of 1□1 □□□□90 daily I□A □tandard solution to all samples and □□.

11.1□.□. Add 100 µ□ of 1□1 □□□□90 □A standard to the □□□.

11.1□.□. □□tract the samples and □□ for a minimum of 1□hours.

11.1□.□. □oncentrate the e□tract from the round bottom flas□using method described in section 11□1□.

11.1□.□. □ransfer the e□tract from the round bottom flas□with he□ane and ad□ust the □olume.

11.1□.□. □plit the e□tract □0□□0 for analysis and archi□e.

11.1□. □□tract □lean-□p

11.1□.1. □or all samples that are not air media□spi□e 1.0 m□ of the □urrogate standard prior to any cleanup□into the round bottom flas□s containing the samples and □□ □□tracts □□ee also □ection 9.□□.

11.1□.□. □roceed with further cleanups as dictated by the sample matri□and e□tract color. The “Option C” cleanup □□ection 11.1□□□and the I□□ □pper □olumn cleanup □□ection 11.1□□ a□e applied to samples with high le□els of interferences. □he I□□ column cleanup □□ection 11.1□□ s□ applied to all samples.

11.1□. Acid □artitioning (“Option C”) - **□ptional cleanup**

11.1□.1. □se this clean up as needed on samples with high le□els of interferences. □onsult with a lead chemist or department manager to determine applicability.

11.1□.□. □artition the e□tract in □0-1□□ m□ of he□ane against □0 m□ concentrated □□□□ in a separatory funnel. □ha□e for two minutes. □emo□e and discard the □□□□ layer □bottom□. □epeat the acid washing until no color is □isible in the acid layer □perform a ma□imum of fi□e acid washings□.

□ar ning□□haking 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.

11.1□.□. □artition the e□tract against □0 m□ of distilled □□. □ha□e for two minutes. □emo□e and discard the a□ueous layer □bottom□. □ry the e□tract by pouring it through a funnel containing anhydrous sodium sulfate and collect it in a round-bottom flas□. □inse the sodium sulfate with two 1□m□ portions of he□ane□add the rinsates to the flas□□and concentrate the he□ane solution to near dryness on a rotary e□aporator □□□□ water bath□□ma□ing sure all traces of toluene □when applicable□are remo□ed. □□se of blow-down with an inert gas to concentrate the e□tract is also permitted□.

11.1□. I□□ □pper □olumn □leanup – **□ptional cleanup**

11.1□.1. □se this clean up as needed on samples with high le□els of interferences. □onsult with a lead chemist or department manager to determine applicability.

11.1□.□. □et up the upper of the two chromatography columns as depicted in □igure □. □he column □□0 mm diameter□is pac□ed in this order□a glass wool plug□□g acti□ated silica gel□□g Acid silica gel□□g acti□ated silica gel□and 1 g sodium sulfate.

11.1□.□. □re-rinse the column with □0 m□ he□ane□and discard the rinsate.

11.1□.□. □osition each column with a round bottom below to collect the e□tract. Add e□tract to the column. □inse e□tract □essel □times with 1 m□ each of he□ane and add to column.

11.1□.□. □lute 90 m□ he□ane directly onto the upper I□□ column.

11.1□.□. □ollect the eluate□and concentrate □ia directions described in section 11.1□ before proceeding with the I□□ cleanup □□ection 11.1□□.

11.1□. I□□ □olumn □leanup

All samples will undergo this cleanup□either direction following concentration on the roto□ap□ ofollowing the cleanup in □ection 11.1□□□ption □□or □ection 11.1□□1□□ □pper □olumn□.

11.1□.1. □et up two chromatography columns as depicted in □igure □. □he upper

column □0 mm diameter□is pac□ed in this order□a glass wool plug□□g acti□ated silica gel□□g Acid silica gel□□g acti□ated silica gel□and 1 g sodium sulfate. □he lower column □1 □mm diameter□is pac□ed in this order□ a glass wool plug□□g acid alumina□and 1 g sodium sulfate.

11.1□.□. □re-rinse each column with □0 m□ he□ane□ nd discard the rinsate.

11.1□.□. □ut one column abo□e the other.

11.1□.□. Add e□tract to the top column □silica column□. □inse e□tract □essel □times with 1 m□ each of he□ane and add to column.

11.1□.□. □lute □0 m□ he□ane directly onto acid silica column □upper column□.

11.1□.□. □iscard upper column.

11.1□.□. □lute lower column with 10 m□ of □0□ methylene chloride□he□ane. □iscard in proper waste stream.

11.1□.□. □lute lower column with □0 m□ of □□□ methylene chloride□he□ane. □a□e and collect in culture tube.

11.1□.9. □roceed with additional cleanups as necessary.

11.1□. □arbon □olumn □lean-up □□□□olumn□– □ptional cleanup

□repare an acti□ated □arbon □ □ilica □el column as described in below. □efer to the diagram in □igure □as well.

11.1□.1. □ush a glass wool plug down to the □.□inch mar□in a pre-cut □□column.

11.1□.□. Add 1 g of □□ acti□ated carbon□silica. □op with a glass wool plug.

11.1□.□. With the column oriented with “A” on the top □and the carbon on the lower end of the column□□ pre-elute with □m□1□1 methylene chloride□cyclohe□ane.

11.1□.□. □iscard pre-eluates.

11.1□.□. Invert the column so that the column is oriented with the “B” on the top and pre-elute with □m□of 1□1 methylene chloride.

11.1□.□. □ilute the e□tract to 1 m□ with he□ane and transfer to the column □still oriented in the “B” direction□.

11.1□.□. □inse sample □ial onto the column with □□□m□1□1 methylene chloride□cyclohe□ane.

11.1□.□. □lute with □m□ 1□1 methylene chloride□cyclohe□ane. Additional amount can be used if samples has high matri□interference.

11.1□.9. □lute with □m□ □□□0□□methylene chloride□methanol□ben□ene.

11.1□.10. □iscard eluates.

11.1□.11. Turn the column over (so that the "A" end is on top), and elute with 30 mL of toluene. □ollect this eluate.

11.1□.1□. □oncentrate to □□A□ dryness using the □oto□ap □□ection 11.1□□ or □urbo□ap □□ection 11.19□ □hen proceed to the reco□ery standard step □□ection 11.□0.

11.1□. □acro-concentration □□tary □□aporator□

□oncentrate the e□tracts in separate round bottom flas□s on rotary e□aporator.

11.1□.1. Assemble the rotary evaporator according to manufacture's instructions, and warm the water bath. □n a daily basis□preclean the rotary e□aporator by sol□ent rinsing. □etween samples□□-□m□ rinses of toluene followed by a □-□ m□ rinse of he□ane should be rinsed down the feed tube into a waste bea□er.

Rotovap Conditions		
Solvent	**Bath Temperature (C)**	**Vacuum Setting (PSI)**
Toluene	80	25
Hexane	76	15
Methylene Chloride	76	No vacuum applied

11.1□.□. Attach the round bottom flas□containing the sample e□tract to the rotary e□aporator. □lowly apply □acuum to the system□and begin rotating the sample flas□.

11.1□.□. □ower the flas□into the water bath and ad□ust the speed of rotation and the temperature as re□uired. At the proper rate of concentration□the flow of sol□ent into the recei□ing flas□will be steady□but no bumping or □isible boiling of the e□tract 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:

$$\%\,\text{Lipids} = \frac{\text{Final Vessel Mass} - \text{Initial Vessel Mass}}{\text{Sample Size}} \times 100\%$$

11.18.5. Proceed to extract cleanup or transfer to a micro concentration vial for the recovery standard step (Section 11.2).

11.13. Micro-concentration (Turbovap)

Concentrate the extracts in 15 mL culture tubes in a turbo-evaporator. The turbo-evaporator model that the laboratory uses can hold up to 54 15 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 (35°C for toluene and 45°C for hexane or hexane-methylene chloride mixtures).

11.13.1. 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.

11.13.2. When evaporating 1 mL toluene it will normally take approximately 15-20 minutes with the temperature setting described above.

11.13.3. When evaporating 1 mL hexane-methylene chloride (1:1) will normally take approximately 2-3 minutes with the temperature setting described above.

11.13.4. For samples requiring % Lipids analysis refer to Section 11.18.4.

11.13.5. Proceed to extract cleanup or transfer to a micro concentration vial for the recovery standard step (Section 11.2).

11.2. Recovery Standard

11.2.1. Add 25 µL of the internal standard solution (Table 2) to each extract.

11.2.2. 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.

11.2.3. With a stream of dry purified nitrogen reduce the extract volume to 250 µL.

11.2.4. Transfer the extract to an autoinjection vial and store in the dark at room temperature. If the samples are for method 8270A the extracts must be

stored in the freezer.

11.2□.5. A smaller final volume can be used to decrease the detection limit upon client approval.

11.2□.□. A larger final volume can be use to decrease potential matrix interferences in the column and acid cleanups were unsuccessful.

11.21. Sample Dilution Procedure

11.21.1. Simple dilutions: Dilutions from 2□ to 5□□ can be achieved without respiking the final extract. The calculation to determine the final extract concentration is as follows:

$$\text{Final Conc. of Extract} = \frac{\text{(Conc. of original extract x Amount of aliquot taken)}}{\text{Volume of diluted extract}}$$

Ex:
$$\frac{\text{(1 g x 2 µL)}}{\text{(2□µL x 1□□µL)}} = \frac{1\text{ g}}{1\text{□□µL}}\ \text{F}$$

Record the final sample concentration on the extract label.

11.21.2. Complex dilution requiring respiking of □□A and □□ standard:

Dilutions greater than 5x must be done by diluting and respiking the extract with □□A and □□ 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. 1□□□ 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 □□, reduced to 20 µL FV.

Record the final sample concentration on 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. .

1□.1. □he □roup□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 re□uired e□pertise.

1□.2. □ ethod □etection □imit

□he laboratory must □enerate a □alid method detection limit for each analyte of interest. □he □ □□ must be below the reportin□limit for each analyte. □he procedure for determination of the method detection limit is □i□en in □□ □□□ □art 1□□□□ppendi□ □□ and further defined in □□□□ □□□□□□□□□□.□□ □□s are a□ailable in the □uality □ssurance □epartment.

1□.□. Initial □emonstration

□he laboratory must ma□e an initial demonstration of capability for each indi□idual method. □emonstration of capability for both soil and water matrices is re□uired. □his re□uires analysis of □□ chec□samples containin□all of the standard analytes for the method. □or some tests it may be necessary to use more than one □□ chec□mi□to co□er all analytes of interest.

1□.□.1. □our ali□uots of the □□ chec□sample are analy□ed usin□the same procedures used to analy□e samples□includin□sample preparation. □he concentration of the □□ chec□sample should be less than or e□ui□alent to the □□□ samples.

1□.□.2. □alculate the a□era□e reco□ery and standard de□iation of the reco□ery for each analyte of interest. □ompare these to the laboratory □enerated □□ □imits.

1□.□. If any analyte does not meet the acceptance criteria the test must be repeated. □nly those analytes that did not meet criteria in the first test need to be e□aluated. □epeated failure for any analyte indicates the need for the laboratory to e□aluate the analytical procedure and ta□e correcti□e action.

1□. POLLUTION CONTROL

It is □urofins TestAmerica's policy to evaluate each method and look for opportunities to minimi□e waste □enerated □i.e. e□amine recyclin□options□orderin□chemicals based on □uantity needed□preparation of rea□ents based on anticipated usa□e and rea□ent stability□. □mployees must abide by the policies in □ection 1□of the □orporate □n□ironmental □ealth and □afety □ anual □□□ □□□□ □□ □1for "Waste Management and Pollution Prevention."

1□.1. □he use of □oto□□aps and □urbo□□aps rather than □uderna□□anish reduction allows e□traction sol□ents to be collected and disposed of rather than released to the atmosphere.

1□.2. □oluene□which is a less ha□ardous sol□ent□has been substituted for ben□ene as an e□traction sol□ent.

1□.□. □he use of □o□□herm e□traction rather than so□hlet e□traction□when appropriate□ reduces the □olume of sol□ent used.

1□.□. □tandards should be prepared in □olumes consistent with laboratory use to minimi□e the □olume of e□pired standards that must be discarded.

1□.□. □ll waste will be disposed of in accordance with □ederal□□tate□and □ocal re□ulations. □ here reasonably feasible□technolo□ical chan□es ha□e been implemented to minimi□e the potential for pollution of the en□ironment.

1□.□. □o not allow waste sol□ent to □ent into the hoods. □ll sol□ent waste is stored in capped containers unless they are bein□filled.

1□.□. □ransfer waste sol□ent from collection cups □tri□pour and similar containers□to □u□s and□or carboys as □uic□ly as possible to minimi□e e□aporation.

1□. □ ASTE MANA□EMENT

□ aste mana□ement practices are conducted consistent with all applicable rules and re□ulations. □□cess rea□ents□samples and method process wastes are disposed of in an accepted manner. □ aste description rules and land disposal restrictions are followed. □ aste disposal procedures are incorporated by reference to □□□□ □□□□□□□ □□1□he followin□ waste streams are produced when this method is carried out.

1□.1. □□tracted a□ueous□leachate samples contaminated with methylene chloride are collected at the fume hood in a □□□allon or smaller carboy. If the samples are not at a neutral p□□add small □uantities of sodium bicarbonate to brin□the waste to neutral. □tir well. □nce neutrali□ed□immediately pour the carboy contents into a blue plastic □□□ drum in the □□closet. □ hen full to between two and si□inches of the top□or after no more than □□days□mo□e the □□□ drum to the waste collection area for shipment.

1□.2. □□tracted soil samples and thimbles□e□tracted □□□filters□□□□□2 resin□paper funnel filters□□lass wool□sodium sulfate□assorted disposable □lassware□fish□crawfish or similar materials□ □ilica □el□alumina□and carbon from column clean□ups□contaminated with □arious sol□ents and eluates. □ump the materials into an oran□e contaminated lab trash buc□et. □ hen the buc□et is full or at the end of the day□tie the plastic ba□liner shut and put the lab trash into the appropriate steel collection drum □hi□h □olatiles waste□in the □□closet. □ hen the drum is full or after no more than □□days□mo□e it to the waste collection area for shipment.

1□.□. □ iscellaneous disposable □lassware□test tubes□syrin□es□filter dis□s□chemical resistant □lo□es□bench paper and similar materials that may or may not be contaminated□ ha□ardous. □lace contaminated materials into a yellow contaminated lab trash buc□et. □ hen the buc□et is full or after no more than one year□tie the plastic ba□liner shut and

put the lab trash into the appropriate steel collection drum □haardous waste landfill□in the □□closet. □hen the drum is full or after no more than □□days□mo□e it to the waste collection area for shipment.

1□.□. □lammable sol□ent waste □enerated durin□□lassware and sodium sulfate cleanin□. □ol□ent waste collected durin□roto□□ap□turbo□□ap reduction of e□tracted samples. □ssorted flammable sol□ent waste □enerated durin□□uart□fiber filter preparation□□□□ adsorbent preparation□□□□□2 resin preparation□□□□□□□□2 cartrid□e preparation□ □lassware rinsin□and sodium sulfate pre□rinsin□. □ollect the flammable waste sol□ents in tripours durin□use. □mpty the tripours into a 1□liter to □□liter carboy at the fume hood. □hen the carboy is full□or at the end of your shift□whiche□er comes first□ empty the carboy into the steel flammable sol□ent drum in the □□closet. □hen full to between two and si□inches of the top□or after no more than □□days□mo□e the steel drum to the waste collection area for shipment.

1□.□. □aste methylene chloride □enerated durin□□lassware and sodium sulfate cleanin□and □arious rinses□roto□□ap□turbo□□ap reduction□□uart□fiber filter preparation□□□□ adsorbent preparation□□□□□2 resin preparation□and □□□□□□□2 cartrid□e preparation. □eep waste methylene chloride separated from waste flammable sol□ents. □ollect the waste methylene chloride in tripours durin□use. □mpty the tripours into a 1□liter to □□liter carboy at the fume hood. □hen the carboy is full□or at the end of your shift□whiche□er comes first□empty the carboy into the steel methylene chloride waste drum in the □□closet. □hen the drum is full to si□inches of the top□or after no more than □□days□mo□e the steel drum to the waste collection area for shipment.

1□.□. □ontaminated distilled water used durin□e□tract cleanup. □ollect the contaminated water in a 1□liter to □□liter carboy at the fume hood. □hen the carboy is full□or at the end of your shift□whiche□er comes first□empty the carboy into the plastic □□□ drum in the □□closet. □hen full to between two and si□inches of the top□ on□after no more than □□days□mo□e the plastic drum to the waste collection area for shipment.

1□. REFERENCES/CROSS REFERENCES

1□.1. □□ □□□□□est □ethods for □□aluatin□□olid □aste□□hird edition□□pdate I□. □ethod □2□□l□□olychlorinated □iben□odio□ins □□□□□s□and □olychlorinated □iben□ofurans □□□□□s□by hi□h□□esolution □ass □pectrometry□□ebruary 2□□□.

1□.2. □□ □□□□□est □ethods for □□aluatin□□olid □aste□□hird edition□□pdate I□. □ethod □□□□□icrowa□e □□traction. □ebruary 2□□□.

1□.□. □□ □□□□□est □ethods for □□aluatin□□olid □aste□□hird edition□□pdate III. □ethod □2□1□□olychlorinated □iben□odio□ins □□□□□s□and □olychlorinated □iben□ofurans □□□□□s□by hi□h□□esolution □ass □pectrometryl □eptember 1□□□.

1□.□. □□ □□□□□est □ ethods for □□aluatin□ □olid □ aste□□hird edition□□pdate III. □ ethod □□2□□□ amplin□□ ethod for □olychlorinated □iben□o□p□dio□ins and □olychlorinated □iben□ofurans □missions from □tationary □ources. □ecember 1□□□.

1□.□. □ompendium □ ethod □□□9A "Determination of Polychlorinated, Polybrominated, and □rominated□□hlorinated □iben□o□p□dioxins and Dibenzofurans in Ambient Air", EPA compendium of □ ethods for the □etermination of □o□ic □r□anic □ompounds in □mbient □ir□second edition□□anuary 1□□□.

1□.□. □rotocol for the □nalysis of 2□□□□□□□□□□ by □□□□□□□□ □□. □. □. □tanley and □. □. □ac□□□□□ □□□□□□□□□□□ □.□

1□.□. "Safety in Academic Chemistry Laboratories", American Chemical Society □ublication□□ommittee on □hemical □afety □□rd □dition□1 □□□.□

1□.□. "Carcinogens □□ or□in□with □arcino□ens□ □epartment of □ealth□□ducation□and □ elfare□□ublic □ealth □er□ice□□enter for □isease □ontrol. National Institute for □ccupational □afety and □ealth. □ublication No. □□□2□□□□u□ust 1□□□.

1□.□. □□□□□ □afety and □ealth □tandards□□eneral Industry□□2□□□□ 1□1 □□□ccupational □afety and □ealth □dministration□□ □□□ 22□□ re□ised □anuary 1□□□.

1□. METHOD MODIFICATIONS

1□.1. □e□iations from □□□ □2□□ and □2□□□.

1□.1.1. □etradecane instead of nonane is used as the final sol□ent to increase the stability of e□tracts and standards. □etradecane is less □olatile than nonane. □oss of analyte as a result of sol□ent incompatibility is monitored throu□h reco□ery chec□s and calibration acceptance criteria.

1□.1.2. □□tract clean□ups are performed at the discretion of the analyst when interferences are obser□ed. □hen□the analyst should select the clean□up procedure appropriate to the interferent.

1□.1.□. □ection □.□.□.□of □ ethod □2□□ indicates that e□tracts should be transferred with he□ane□then toluene. □oluene is used to transfer e□tracts to maintain compound solubility and minimi□e analyte loss.

1□.1.□. □ection □.□1.2 of □ ethod □2□□ specifies that a Na□l solution should be used for partitionin□. Instead□the laboratory uses laboratory water only. Na□l is used to brea□up emulsions that may form. □n analyst may use Na□l□Na□□□or any mechanical means to brea□up an emulsion.

1□.1.□. □ection □.□.□of □ ethod □2□□ specifies that he□ane 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.

10.1.3. Carbon columns are packed with silica gel in place of celite. Elution solvents are changed accordingly. Section 11.3 Method 23 Section 9.3.3.2 2378 Section 9.3.4.

10.2. Modifications from 1613 method

10.2.1. Quartz fiber filters are cleaned by Soxhlet extraction with methylene chloride not baked at 300 for hours.

10.2.2. The XAD material may be precleaned with methylene chloride or other appropriate solvent. The XADs are not reused.

10.2.3. The 13C12-2378-TCDD surrogate is present at varying levels in the calibration curve (0.200 pg/ µL).

10.2.4. Samples are extracted with toluene not benzene.

10.2.5. Concentration is performed by rotary evaporation not Kuderna-Danish.

10.2.6. All cleanup procedures are optional and applied based on the analyst's discretion.

10.2.7. The laboratory uses 2 labeled recovery standard for the quantitation of labeled internal standards.

10.2.8. The final volume is adjusted to 20 µL in tetradecane.

10.2.9. Calibration and quantitation are performed in accordance to this SOP.

10. ATTACHMENTS

10.1. Table 1 – Types of Matrices

10.2. Table 2 – Composition of Sample Fortification and Recovery Standard Solutions.

10.3. Table 3– The Seventeen 2378 substituted PCDD and PCDF congeners

10.3. Figure 1 – Analysis Flowchart

10.3. Figure 2 – ION column cleanup

10.3. Figure 3– 22 Column cleanup

10.6. Appendix – Periodic Pipet Performance

10. REVISION HISTORY

10.1. Previous revision 2.0 Effective 11/21/19

10.1.1. Section 11.12.3 revised “50 µL” to “100 µL”.

10.1.2. Section 11.11.1 updated bath temps in table for hexane and methylene chloride.

10.1.3. Editorial changes.

10.2. Previous revision 2.1 Effective 11/21/19

10.2.1. Section 7.1.5, “70-230 mesh” revised to “230-400 mesh”.

10.2.2. Added Section 15.5, “Waste methylene chloride generated during glassware and sodium sulfate cleaning and various rinses (roto-vap, turbo-vap reduction, quart fiber filter preparation, adsorbent preparation, 2 resin preparation and 2 cartridge preparation. Keep waste methylene chloride separated from waste flammable solvents. Collect the waste methylene chloride in tripours during use. Empty the tripours into a 10 liter to 20 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 flammable closet. When the drum is full to six inches of the top or after no more than 90 days, move the steel drum to the waste collection area for shipment.”

10.2.3. Removed revision history prior to 11/21/19, it can be found in previous versions of this SOP.

10.2.4. Editorial changes.

10.3. Previous revision 2.2 Effective 12/20/19

10.3.1. Section 2.2, added “leachate” as an applicable matrix.

10.3.2. Inserted Section 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.”

10.4. Previous revision 2.3 Effective 12/21/20

10.4.1. Section 11.10 – Changed boiling chip solvent rinse from methylene chloride

to he□ane.

1□.□.2. □ection □.2 – Inserted □th sentence to para□raph – "An LCSD is required if a MS/MSD is not extracted with the batch."

1□.□.□. □ection 11.1□.2 and 11.1□.□– □han□ed □□m□ of concentrated □2□□□ □□ection 11.1□.2□nd □□m□ of distilled □2□ □□ection 11.1□.□□o 2□ m□.

1□.□.□. □ditorial chan□es.

1□.□. □ □□1□□□□ □□□e□ision 2.2□□ffecti□e □□2□2□1□

1□.□.1. □dded □ection □1.□– "Microwave ovens used for extraction or digestion of samples can create ele□ated pressure in the e□traction□di□estion containers. □ icrowa□es used for these processes must be e□uipped with an automated □apor sensor and shutoff system that automatically shuts down the di□estion□e□traction process when □apor pressure reaches pre□specific le□els. Users must follow procedures in the microwave operator's manual to ensure that the □apor sensors are functional and wor□in□properly prior to startin□ each extraction/digestion batch."

1□.□.2. □ection □□remo□ed reference to □iesel□el□and updated rea□ent information□ added reference to diatomaceous earth, and moved the "reagents" section to □.1. □lso remo□ed information re□ardin□1□□1 Na□□□□ ater.

1□.□.□. □ection 11.□.□– □eplaced □ttawa sand with Na2□□□.

1□.□.□. □dded □ections 11.□.□– 11.□.11 specifyin□microwa□e e□traction procedure.

1□.□.□. □e□ision history prior to 2□12 has been remo□ed. It is a□ailable for re□iew in pre□ious □ersions of this □□□.

1□.□.□. □ditorial chan□es.

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T□□□□ □□M□□□□□□□S□□ □□□S□□□□ □□□□ □3□□□□TCDD□□ □□□□ M□□□□□ C□□□□□□□□□ L□□ □□□P□□□□
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	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
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

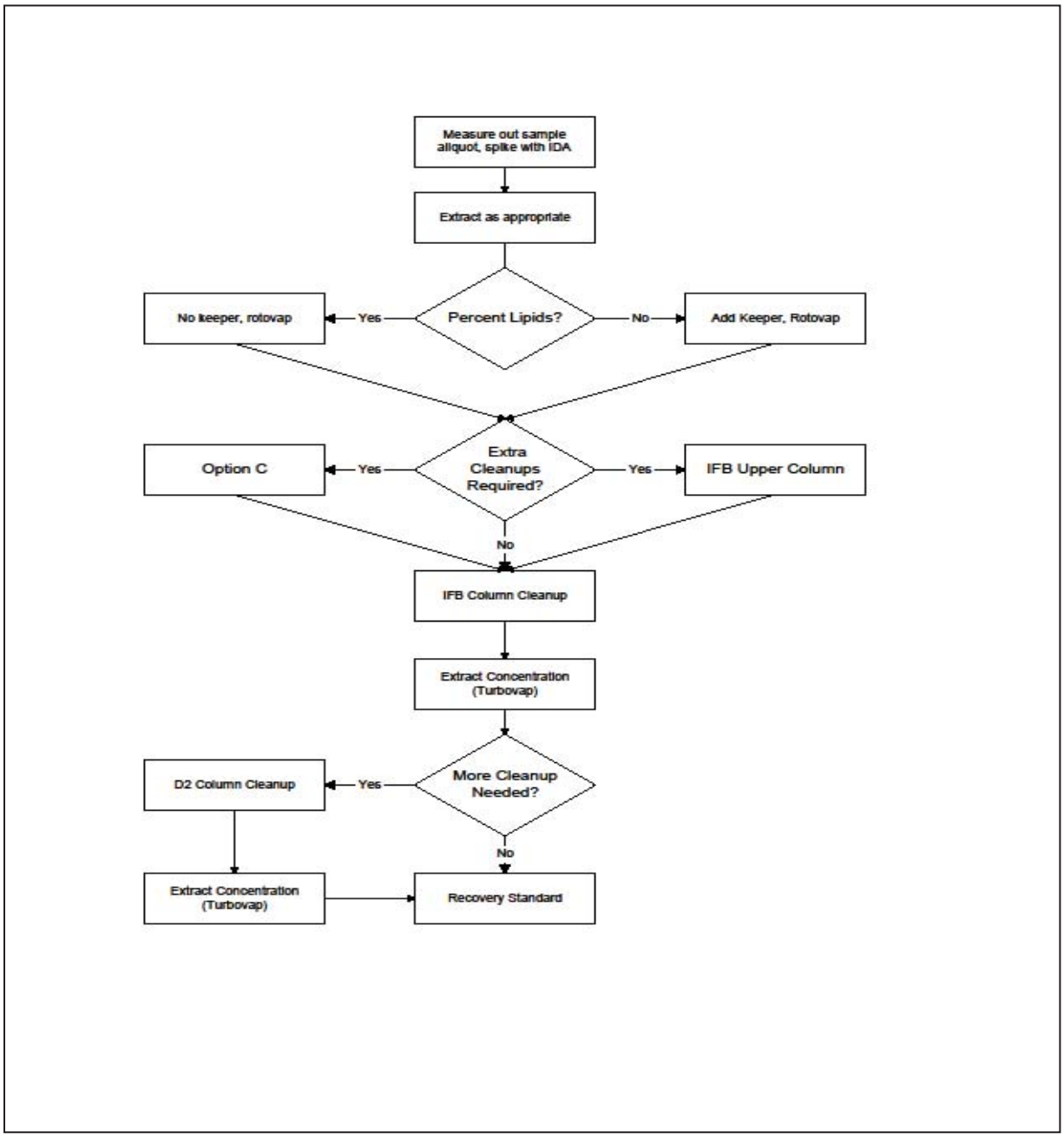
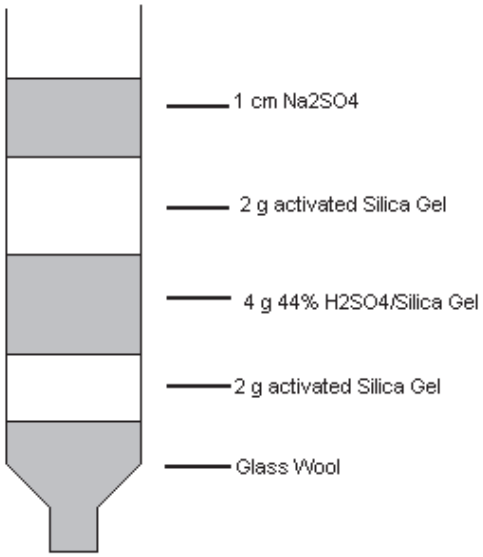
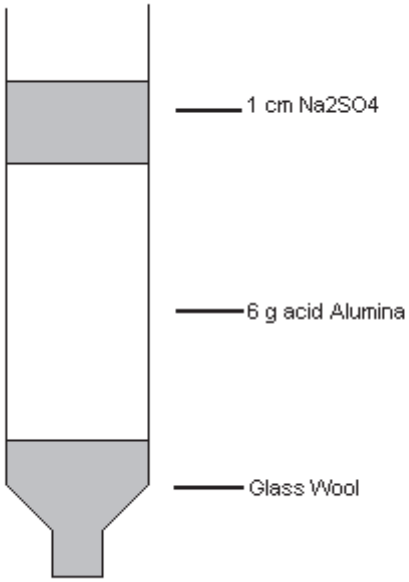


Figure □
□iagra□ of □F□ □olu□ □ □lea□u□

□se □□ □ □ colu□n □or to□colu□n (□□□ Colu□n)
□se 1□ □ □ colu□n □or botto□ colu□n□(□cid □lu□ina)
***Note**: Upper and lower columns are piggy backed for IFB cleanup, upper column only can be used for additional cleaning.*

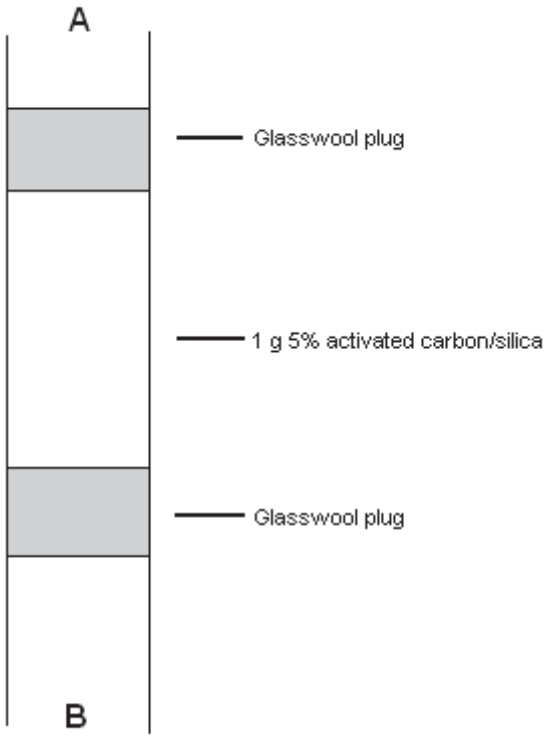


Instead of 1 cm Na2SO4, it's 1 g.



Instead of 1 cm Na2SO4, it's 1 g.

Figure 3
D2 Carbon Column



□□□en□i□□
□□reening □□e □abora□or□ □□or 2□3□□□□□ongener□

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 (□EPA) 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 E□□S.

Title: PCB Analysis by HRGC/HRMS [Methods 1668A & 1668C]

Approvals (Signature/Date):

09/03/2020 Robert Hrabak Date Technical Manager	08/05/2020 Joe Schairer Date Health & Safety Manager / Coordinator
09/15/2020 Lisa Stafford Date Quality Assurance Manager	08/05/2020 Chris Williams Date Laboratory Director

Facility Distribution No. Uncontrolled **Distributed To:** Sacramento Bids Folder

1. SCOPE AND APPLICATION

[redacted]

2. SUMMARY OF METHOD

[redacted]

[redacted]

[redacted] 2,2',5,5' [redacted] 2,2',4,5,5' [redacted] 2,2',3,4,4',5' [redacted] 2,2',3,3',5,5',6,6' [redacted]

[redacted]

[redacted]

[redacted]

[redacted] 3,3',4,4',5 [redacted]

3. DEFINITIONS

[illegible]

[illegible]

[illegible]

[illegible]

4. INTERFERENCES

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

5. SAFETY

[illegible]

Materials used in this SOP, and their Hazards.			
Material	**Hazards**	**Exposure Limit (1)**	**Signs and symptoms of exposure**
Dodecane (3-2-0)	Flammable	None listed	May cause respiratory tract, skin or eye irritation. Harmful or fatal if swallowed or aspirated.

Materials used in this SOP, and their Hazards.			
Material	**Hazards**	**Exposure Limit (1)**	**Signs and symptoms of exposure**
Iso-octane (2,2,4-trimethylpentane, 2,4,4-trimethylpentane) (3-3-0)	Flammable	300 PPM TWA	Causes eye or respiratory tract irritation. Repeated/prolonged exposure can cause defatting of skin. High concentrations can produce drowsiness. May be fatal if swallowed or aspirated.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

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.

7. REAGENTS AND STANDARDS

[redacted]

[redacted]

[redacted]

[redacted]

may be used until the manufacturer's expiration date is exceeded.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

9. QUALITY CONTROL

NOTE: When laboratory contamination is suspected sample results can be assumed to be maximum possible concentrations. It may be useful to consult project action limits to see if these concentrations may be acceptable to the client. This does NOT preclude the immediate need to

initiate contamination clean-up procedures in the sample preparation area.

[illegible]

[illegible]

[illegible] **Method 1668A** [illegible]

[illegible] **Method 1668C** [illegible]

[illegible]

[illegible]

[illegible]

[illegible] are recovery corrected per the isotope dilution technique. For an analyte reported as 'Not Detected' the associated reporting limit represents its maximum possible concentration.

10. CALIBRATION

[illegible] 003, "Calibration Curves and Selection of Calibration Points".

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

NOTE: Commercially available PFK can contain varying levels of contamination. A minor PFK mass (223.9872) is known to interfere with the dichloro- PCB secondary quantitation ion (M+2). If this interferent is present it may not be possible to meet the S/N and ion ratio criteria for CS-2 In these cases the calibration/verification is considered acceptable if the CS-3 through CS-6 levels meet all criteria and the CS-2 meets RT criteria and the primary quantitation ion meets 10:1 signal to noise.

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

Equation 1

$$RRF(n) = \frac{A_{TA} \times Q_{IDA}}{Q_{TA} \times A_{IDA}}$$

Equation 2

$$RRF(m) = \frac{A_{is} \times Q_{rs}}{Q_{is} \times A_{rs}}$$

[illegible]

[illegible]

The RRF for the total PCBs for each homologous series is calculated as the average of RRFs of the first and last eluting isomers of the corresponding homologous series. The sole exception is $^{13}C_{12}$-3,3',4,4',5-penta PCB (EPA #126) which is not used in the average response for the penta-PCB homologous series.

10.4.4. The initial calibration is accepted if the relative standard deviation (% RSD) for the Toxic and LOC native compounds listed in Method 1668A does not exceed ± 20 percent. The first eluting congeners for each chlorination level are not Toxic congeners, and are only used for retention time reference and in the estimated calculation of homolog concentrations. The calibration for the labeled isotope dilution analytes (IDAs) is acceptable if the %RSD does not exceed ± 40 percent.

Note- The incorporation of alternate calibration analytes and acceptance criteria may be implemented on a project specific basis.

10.4.5. The signal to noise ratio (s/n) for the GC signals present in every selected ion current profile must be > 3.0:1.

10.4.6. An injection of the 209 PCB congener mix will be performed annually at concentration at or near the laboratory reporting limit for the congeners not included in the multi-point calibration curve. Solution must be 1 – 2 times the reporting limit. For DoD/DOE QSM compliant work a quarterly LOQ check at the CS-2 level and an LOD check at 2-4X the DL level must be performed.

10.5. Initial calibration verification standard (ICV): A second source calibration standard is analyzed following the initial calibration curve, prior to samples. Each of the Toxic and LOC congeners in the ICV must be within ± 30% of its expected value for method 1668A, and ± 25% of its expected value for method 1668C. The remaining congeners must be within ± 50% of their expected values. IDA must meet the same criteria as for the daily calibration check (below).

10.6. Daily Calibration Check (CS4)

10.6.1. 1668A Calibration Criteria

10.6.1.1. Daily calibration check is required every 12 hours. The daily calibration check is acceptable if the % Difference in RRF for the Toxic and LOC native compounds listed in Method 1668A are not greater than ± 30% from the initial calibration. The first eluting congeners for each chlorination level are not Toxic congeners, and are only used for retention time reference and in the estimated calculation of homolog

concentrations. The daily calibration check of IDAs is acceptable if the RRFs are not greater than ± 50 % of the mean RRF calculated from the initial calibration curve, unless otherwise directed in the client's SOW. The daily calibration check of the cleanup recovery surrogates (S□) is acceptable if the RRFs are not greater than -40 % or □30% of the mean RRF calculated from the initial calibration curve, unless otherwise directed in the client's SOW. The ratio of the ions for the target analytes (TA) and isotope dilution analytes (IDAs) must be within the limits specified in Table II. If the daily calibration check fails to meet the above criteria, a new initial calibration curve is required.

10.6.1.2. For 1668C QC acceptance criteria see Table VIII.

10.6.2. The valley height between the shorter of the two peaks for the congener pairs 34/23 and 18□182 must be less than 40%. Congeners 156 and 15□must co-elute within 2 seconds of the peak maximum.

10.6.3. If the criteria in Section 10.5.2 are not met, maintenance must be performed. If a second injection fails the column should be replaced.

10.6.3.1. The retention times and response factors should be updated after the criteria for Section 10.5 is met.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of a supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Non-Conformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Non-Conformance 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. Analysis

11.2.1. 1-2 uL of the final 20 uL extract of the sample is injected into the GC.

11.2.2. Acquire SIM data under the same acquisition and mass spectrometer conditions that the initial calibration curve was acquired.

12. CALCULATIONS/DATA REDUCTION

12.1. For a gas chromatographic peak to be identified as a PCB peak, the following criteria must be met:

12.1.1. For the PCB congeners (a total of 2 congeners) which have an isotopically labeled IS As in the sample extract, the retention time must be within -1 to + seconds of the related IS A.

12.1.2. For the Toxic/LOC congeners (2 congeners) that are present in the five initial calibration solutions, the retention time must be within 0.000 retention time units of the relative limits measured in the routine calibration (CS standard).

12.1.. For the remaining 1 2 PCB congeners (mono-nona) which do not have an isotopically labeled internal standard and are not in the five point initial calibration solutions, the retention time should be within 0.00 retention time units of the relative times measured in the 20 PCB single point calibrations solution. If mono-nona totals are being reported for all or just the remaining isomers the retention time for these compounds should be within the corresponding homologous retention time windows which are established by analyzing either a calibration solution or the 20 single point calibration solution.

12.1.. For the two ions monitored for each analyte, the apex of the peaks must occur within - 2 seconds of each other.

12.1.. The ratio of the relative intensity of the selected isotopic ions is required to be within the limits (Table II).

12.1.. A GC/MS peak must be .0times higher than the noise level for positive identification of a PCB compound, and 10 times higher than the noise level for all labeled compounds.

12.1.. For total isomers to be positively identified they must be within the retention time window of their respective homologous series as specified by the 20 PCB calibration standard.

12.1.. The loss of one or more chlorines from high chlorinated congeners may contribute to the less-chlorinated congeners peaks that elute at the same retention time which can have an adverse effect on the quantitation of the less-chlorinated congeners. Also in the analysis of total PCBs the extra erroneous peaks in the chromatogram of less-chlorinated congeners produced by the fragment of the high–chlorinated congeners may lead to a high bias in the concentration of the less-chlorinated congeners. If identification is ambiguous, an experienced analyst will determine the presence or absence of the congeners to be reported and the data flagged or narrated appropriately. (The flags will include but are not limited to an elevated detection limit or an estimated positive concentration).

12.2. For gas chromatographic peaks that have met the criteria in Section 12.1, the concentration of the PCBs is calculated by using the following formula:

Equation 3

$$C_{TA} = \frac{A_{TA} \times Q_{IDA}}{A_{IDA} \times W \times RRF_{TA}}$$

Where:

C_{TA} = Concentration of unlabeled TA PCB congener,
A_{TA} = Sum of the integrated ion areas of quantitation ions for unlabeled PCBs,
A_{IDA} = Sum of the integrated ion areas of the quantitation ions for the labeled IDAs,
Q_{IDA} = Quantity, in pg, of the IDAs added to the sample before extraction,
W = Sample size in g (if solid) or L (if liquid), and
$RR_{(TA)}$ = Calculated mean relative response factor for the TA.

12.3. The percent recovery of the IDAs is calculated by using the following formula:

Equation 4

$$\text{IDA Percent Recovery} = \frac{A_{IDA} \times Q_{IS}}{Q_{IDA} \times A_{IS} \times RRF_{(IDA)}} \times 100$$

Where:

A_{IDA} = Sum of the integrated ion areas of the quantitation ions for the labeled isotope dilution analyte (IDA).
A_{IS} = Sum of the integrated ion areas of the quantitation ions for the labeled internal standard (IS).
Q_{IDA} = Quantity, in pg, of the isotope dilution analyte (IDA) added to the sample before extraction,
Q_{IS} = Quantity, in pg, of the internal standard (IS) added to the cleaned up sample extract before HRGC/HRMS analysis, and
$RRF_{(IDA)}$ = Mean relative response factor for the labeled IDA relative to the appropriate internal standard

12.4. The total concentration for each homologous series of PCBs calculated by summing up the concentration of all positively identified isomers of each homologous series.

12.5. Target compounds that exceed the upper calibration range of the calibration solutions, will be qualified as estimated unless otherwise directed by specific project request. A dilution factor appropriate to bringing the toxic congeners within the calibration range should be used. For other congeners a dilution that, at a minimum, brings the response to a concentration within the detector's response range is acceptable. If possible, the dilution should bring the concentration within the calibration range of the initial calibration solutions. Results for PCB congeners in a sample that has been diluted are reported at the least dilute level at which the area at the quantitation m/z is in the linear response range and the corresponding labeled compound recovery is within the acceptance range.

12.6. Reporting Results

12.6.1. Unless otherwise directed TestAmerica Sacramento will report results in the

following units: aqueous samples (pg/L), solids and sediments (pg/g), air samples (pg/sample). Tissues are reported in pg/g wet weight.

12.6.2. Unless otherwise requested the Toxic/L□C congeners will be reported to an RL consistent with the CS□2 calibration level. □ther congeners will be reported to an RL of 10□ the CS□2 level. Reporting limits for coeluting groups will be multiplied by the number of congeners present. □PA CLP wor□will be reported per □xhibit C Table 1 of the statement of Wor□(CBC1□x). Results below the reporting limit will be reported only upon specific request and the reporting process must be agreed upon with the client before samples are processed.

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 WS-QA-0006. MDLs are available in the Quality Assurance department.

13.2. Initial Demonstration

Each analyst must make a one time initial demonstration of capability for each individual method. Demonstration of capability for both soils 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.

13.2.1. 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 equivalent to a mid level calibration standard.

13.2.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these results with the historical acceptance criteria.

13.2.3. 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.

13.2.4. A passing PT sample can be substituted for the 4 aliquots in Section 13.2.1.

13.3. Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

□□□PO□□□T□ON CONTRO□

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 □ealth and Safety Manual □CW-E-M-001) for "Waste Management and Pollution Prevention."

14.1. 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.

14.2. Do not allow waste solvent to evaporate in fume hoods. All solvent waste is stored in capped containers unless transfers are being made.

□□□□ A□TE MANA□EMENT

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-E□S-0001. The following waste streams are produced when this method is carried out.

1□.1. Autovials contaminated with dodecane. 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 ninety days.

1□.2. 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 steel landfill lab trash drum in the □3 closet. When the drum is full, or after no more than □□days, move it to the waste collection area for shipment.

□□□REFERENCE□□CRO□□REFERENCE□

16.1. State of California Air Resources Board Method 42□□Determination of Polychlorinated Dibenzo-p-dioxin □PCDD□, Polychlorinated Dibenzofuran □PCDF□, and Polychlorinated Biphenyl Emissions from Stationary Sources, September 12, 1□□0.

16.2. EPA Method 166□□Toxic polychlorinated Biphenyls by Isotope Dilution □igh Resolution □as Chromatography/□igh resolution Mass Spectrometry, March 1□□□.

16.3. Method 166□, Revision A □Method 166□A□□Chlorinated Biphenyl Congeners in Water, Soil, Sediment and Tissue by □R□C/□RMS, August 2003.

16.4. □Method 166□C□Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by □R□C/□RMS, April 2010.

16.□. Method □0□2A□Polychlorinated Biphenyls □PCBs□By □as Chromatography, February 200□.

16.6. EPA SOW CBC01.2□Analysis of Chlorinated Biphenyl Congeners □PCBs□, December 200□.

□□□METHOD MOD□F□CAT□ON□

1□.1. Deviations from reference Method 166□A

1□.1.1. The acceptance criteria of not greater than □/- 20□ and □/- 30□ for the Initial Calibration and Continuing Calibration Check respectively, includes the Toxic and LOC native compounds listed in Method 166□A and 166□C,

1□.1.2. The retention time for PCB-20□ does not have to be greater than □□minutes if all criteria listed in Section 10.□are achieved. The laboratory uses □C conditions different than those recommended in Method 166□A.

1□.1.3. The retention time windows used to identify the chlorination levels is established from the analysis of the native window defining mix included in the calibration curve and native spiking mix.

1□.1.4. The laboratory routinely uses a SPB-Octyl column for primary analysis.

□□□ATTACHMENT□

1□.1. Table I- Composition of the Sample Fortification Solutions

1□.2. Table II- Ion-Abundance Ratio Acceptable Ranges

1□.3. Table III- □C Temperature Program

1□.4. Table I□- Ions Monitored for □R□C/□RMS Analysis of PCBs

1□.□. Table □- □igh-Resolution Concentration Calibration Solution

1□.6. Table □I- Composition of the Matrix Spike Fortification Solution

1□.□. Table □II- Analyte List

1□.□. Table □III – Acceptance Criteria for □ER, IPR, OPR, and Labeled Compounds in Samples

□□□RE□□□□ON H□□TOR□

1□.1. WS-ID-0013, Revision 4.□, Effective 0□/1□/2020

1□.1.1. Section 1.2 revised to, "The CBs determined in this method are the 12 polychlorinated biphenyls □PCBs□designated as toxic by the World □ealth Organization □W□O□. These are congeners □□, □1,10□, 114, 11□, 123, 126, 1□6, 1□□, 16□, 16□,nd 1□□. This method also determines the remaining 1□□PCBs, of which 130 are resolved on the SPB-octyl column to be determined as individual congeners. The remaining 6□congeners are determined as mixtures of isomers □co-elutions□. This method can also be used to determine the 20 PCBs on the □ational Oceanic and Atmospheric Administration □□OAA□list."

1□.1.2. Section 2.3 revised to, "The preparation of the final extract for the instrumental analysis is accomplished by adding □isotopically □13 C12□labeled internal standards □IS -Table I□. The internal standard 13C-2,□-DiCB □EPA□□□is used to quantitate the mono and di chlorinated biphenyl Isotope Dilution Analytes □IDAs□ and 13C-TrCB-1□. The internal standard 13C-2,2',5,5'-TCB □EPA □□2□is used to determine the percent recoveries of tri- and tetra-chlorinated biphenyl IDAs. The 13C-2,2',4,5,5'-PeCB □EPA □101□is used to determine the percent recoveries of penta-chlorinated biphenyls. The 13C-2,2',3,4,4',5'-□xCB □EPA □13□□is used to determine the percent recoveries of the hexa chlorinated biphenyl IDAs. The 13C-2,2',3,3',5,5',6,6'-OCB □EPA □1□4□internal standard is used to determine the percent recoveries of hepta- through deca- chlorinated biphenyl IDAs."

1□.1.3. Section 5.1.1 revised to include, "closed toed non-absorbent shoes".

1□.1.4. Section 5.2 table added, "Iso-octane □2,2,4-trimethylpentane, 2,4,4-trimethylpentane)" and associated information. Added NFPA hazard numbers for Dodecane and revised associated information.

1□.1.□. Section 6.4 revised to, "Data System- Capable of collecting, recording and storing MS data. The system utilizes MassLynx version 4.1 software and Chrom Peak Review, version 2.3.7 or equivalent."

1□.1.6. Section 10.4.4 removed sentence, "The second source criterion for the natives □TA□is □30□ deviation from the curve."

1□.1.□. Section 10.4.5 revised, "2.5:1" to "3.0:1"

1□.1.□. Section 12.1.6 revised, "2.5" to "3.0"

1□.1.□. Section 15.2 revised, "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 steel landfill lab trash drum in the □3 closet. When the drum is full, or after no more than □□ days, move it to the waste collection area for shipment."

1□.1.10. Editorial changes.

1□.2. WS-ID-0013, Revision 4.6, Effective 0□/02/2016

1□.2.1. Section 1.1 – Added 166□C.

1□.2.2. Section □2 – Added "A second LCS, laboratory control sample duplicate (LCSD), must be performed in the absence of a matrix spike /matrix spike duplicate □MS/MSD or MS/SD□ pair. " to end of paragraph.

1□.2.3. Editorial changes.

1□.3. WS-ID-0013, Revision 4.□, Effective 12/31/201□

1□.3.1. □pdated Copyright Statement on cover page.

1□.3.2. Changed all references to DOD to be DOE inclusive – Sections 1.□, □1.1, and 10.4.6.

1□.3.3. Section 5.2, changed all references to "MSDS" to "SDS".

1□.3.4. Added Section 6.□, Recommended Preventative Maintenance.

1□.3.□. Inserted Section 10.5, "Initial calibration verification standard □IC□□□A second source calibration standard is analyzed following the initial calibration curve, prior to samples. Each of the Toxic and LOC congeners in the IC□ must be within ± 30□ of its expected value for method 166□A, and ± 2□□ of its expected value for method 166□C. The remaining congeners must be within ± □0□ of their expected values. IDA must meet the same criteria as for the daily calibration check □below□."

1□.4. WS-ID-0013, Revision 4.4, Effective 0□/0□/2014

1□.4.1. Revised Section □3 to read - "All extracts must be stored capped at □-10 □C and completely analyzed within one year of extraction. This holding time is consistent with EPA Method □0□2 □ref 16.□□and EPA SOW CBC01.2. □Ref 16.6□."

1□.4.2. Inserted Section 16.□– "Method □0□2A□Polychlorinated Biphenyls □PCBs□By

□as Chromatography, February 200□"

1□.4.3. Inserted Section 16.6 – "EPA SOW CBC01.2□Analysis of Chlorinated Biphenyl Congeners □PCBs□, December 200□"

1□.4.4. Editorial changes.

1□.□. WS-ID-0013, Revision 4.3, Effective 0□/10/2013

1□.□.1. Incorporated SOP □WS-ID-001□– Method 166□C□into this SOP.

1□.□.2. Added Table □III with Method 166□C acceptance criteria.

1□.□.3. Editorial changes.

1□.6. WS-ID-0013, Revision 4.2, Effective 03/1□/2013

1□.6.1. □pdated selected ion masses in Table I□

1□.6.2. Editorial changes.

1□.□. WS-ID-0013, Revision 4.1, Effective 11/11/2011

1□.□.1. □pdated Table□s□2, 3, and 4.

1□.□.2. Editorial changes.

1□.□. WS-ID-0013, Revision 4, Effective □/31/2010

1□.□.1. Deleted the last sentence in Section 2.6.

1□.□.2. Deleted all references to the DB-□column type in Sections 4.□, 6.2.1 and 10.2.1.

1□.□.3. Deleted all references to extraction equipment or requirements.

1□.□.4. Deleted Section 6.2.3.

1□.□.□.Deleted Reference 16.1.

1□.□.6. Editorial changes.

1□.□.□.Tables I, II, III, □ and □I were updated.

TA□□E □

C□□ □□□□□□□□ □□□□□ □□□ □□F□□□□□□□□□□□□ □□□□□□□□

	Isotope Dilution Analytes □IDA□ Concentration □pg/uL in Isooctane□	Internal Standards □IS□ Solution □pg/uL in Dodecane□	Clean-up Recovery Surrogates □S□□ □pg/uL in Dodecane□
$^{13}C_{12}$-2-MonoPCB □1□	20	--	--
$^{13}C_{12}$-4-MonoPCB □3□	20	--	--
$^{13}C_{12}$-2,2-DiPCB □4□	20	--	--
$^{13}C_{12}$-2,□-DiPCB □□□	--	100	--
$^{13}C_{12}$-4,4'-DiPCB □1□□	20	--	--
$^{13}C_{12}$-2,2',6'-TriPCB □1□□	20	--	--
$^{13}C_{12}$-2,4,4'-TriPCB □2□□	--	--	100
$^{13}C_{12}$-3,4,4'-TriPCB □3□□	20	--	--
$^{13}C_{12}$-2,2',5,5'-TetraPCB □□2□	--	100	--
$^{13}C_{12}$-2,2',6,6'-TetraPCB □□4□	20	--	--
$^{13}C_{12}$-3,3',4,4'-TetraPCB □□□□	20	--	--
$^{13}C_{12}$-3,4,4',5-TetraPCB □□1□	20	--	--
$^{13}C_{12}$-2,2',4,5,5'-PentaPCB □101□	--	100	--
$^{13}C_{12}$-2,2',4,6,6'-PentaPCB □104□	20	--	--
$^{13}C_{12}$-2,3,3',4,4'-PentaPCB □10□□	20	--	--
$^{13}C_{12}$-2,3,3'5,5'-PentaPCB □111□	--	--	100
$^{13}C_{12}$-2,3,4,4',5-PentaPCB □114□	20	--	--
$^{13}C_{12}$-2,3',4,4'5-PentaPCB □11□□	20	--	--
$^{13}C_{12}$-2',3,4,4',5-PentaPCB □123□	20	--	--
$^{13}C_{12}$-3,3',4,4',5-PentaPCB □126□	20	--	--
$^{13}C_{12}$-2,2',3',4,4',5'-□exaPCB □13□□	--	100	--
$^{13}C_{12}$-2,2',4,4',6,6'-□exaPCB □1□□□	20	--	--
$^{13}C_{12}$-2,3,3',4,4',5-□exaPCB □1□6□	20	--	--
$^{13}C_{12}$-2,3,3',4,4',5'-□exaPCB □1□□□	20	--	--
$^{13}C_{12}$-2,3',4,4',5,5'-□exaPCB □16□□	20	--	--
$^{13}C_{12}$-3,3',4,4',5,5'-□exaPCB □16□□	20	--	--
$^{13}C_{12}$-2,2',3,3',5,5',6-□eptaPCB □1□□□	--	--	100
$^{13}C_{12}$-2,2',3,4',5,6,6'-□eptaPCB □1□□□	20	--	--
$^{13}C_{12}$-2,3,3',4,4',5,5'-□eptaPCB □1□□□	20	--	--
$^{13}C_{12}$-2,2',3,3',4,4',5,5'-OctaPCB □1□4□	--	100	--
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-OctaPCB □202□	20	--	--
$^{13}C_{12}$-2,3,3',4,4'5,5',6-OctaPCB □20□□	20	--	--
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-□onaPCB □206□	20	--	--
$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-□onaPCB □20□□	20	--	--
$^{13}C_{12}$-DecaPCB □20□□	20	--	--

TA□□E □□
□□□□A □□□□□□□□R□□□□A □□□□□□□□R□□□□□

□umber of □alogen Atoms	Theoretical Ion Type	Ratio	Control Limits	Lower □pper
1 Cl	M/M□2	3.13	2.66	3.60
2 Cl	M/M□2	1.□6	1.33	1.□□
3 Cl	M/M□2	1.04	0.□□	1.20
4 Cl	M/M□2	0.□□	0.6□	0.□□
□ Cl	M/M□2	1.□□	1.32	1.□□
6 Cl	M□2/M□4	1.24	1.0□	1.43
□ Cl	M□2/M□4	1.0□	0.□□	1.21
□ Cl	M□2/M□4	0.□□	0.□6	1.02
□ Cl	M□2/M□4	0.□□	0.6□	0.□□
10 Cl	M□4/M□6	1.16	0.□□	1.33

TA□□E □□□
□C T□□ □□□□□□□□P□□□□□□

Parameter	SPB-Octyl
Run time □m□	□1.6
Initial Temp □□C□	1□0
Initial Time □m□	2.0
Rate □1 □□C/m□	2.0
Temp □1 □□C□	2□6
Time □1 □m□	0.0
Rate □2 □□C/m□	1□
Temp □2 □□C□	2□□
Time □2 □m□	12
Rate □3 □□C□	120
Final Temp □□C□	120
Final Time □m□	2

TA□□E □□
□□□□M□□□□□□□ □□□HR□C□HRM□A□□□□□□□□PC□□

Descriptor	Accurate Mass	Ion I.D.	Analyte
1	1□□.9□3	M	MonoCB
	1□0.0363	M□2	MonoCB
	200.0□□□	M	MonoCB IDA
	202.0□66	M□2	MonoCB IDA
	21□.□□□6	Lock	PF□
	222.0003	M	DiCB
	223.□□□4	M□2	DiCB
	22□.□□44	M □4	DiCB
	234.0406	M	DiCB IDA
	236.03□6	M□2	DiCB IDA
2	222.0003	M	DiCB
	223.□□□4	M□2	DiCB
	22□.□□44	M □4	DiCB
	234.0406	M	DiCB IDA
	236.03□6	M□2	DiCB IDA
	242.□□□6	Lock	PF□
	2□□.□613	M	TriCB
	2□□.□□□4	M□2	TriCB
	2□□.□□□4	M □4	TriCB
	26□.0016	M	TriCB IDA
	26□.□□□6	M□2	TriCB IDA
	2□□.□224	M	TetraCB
	2□1.□1□4	M□2	TetraCB
	2□3.□16□	M □4	TetraCB
	301.□626	M	TetraCB IDA, IS
	303.□□□□	M□2	TetraCB IDA, IS
3	2□□.□613	M	TriCB
	2□□.□□□4	M□2	TriCB
	2□□.□□□4	M □4	TriCB
	26□.0016	M	TriCB IDA
	26□.□□□6	M□2	TriCB IDA
	2□0.□□2□	Lock	PF□
	2□□.□224	M	TetraCB
	2□1.□1□4	M□2	TetraCB
	2□3.□16□	M □4	TetraCB
	301.□626	M	TetraCB IDA, IS
	303.□□□□	M□2	TetraCB IDA
	323.□□34	M	PentaCB
	32□.□□04	M□2	PentaCB
	32□.□□□□	M □4	PentaCB
	33□.□20□	M□2	PentaCB IDA, IS
	33□.□1□□	M □4	PentaCB IDA, IS

Descriptor	Accurate Mass	Ion I.D.	Analyte
4	2□□.□224	M	TetraCB
	2□1.□1□4	M□2	TetraCB
	2□3.□16□	M □4	TetraCB
	301.□626	M	TetraCB IDA, IS
	303.□□□□	M□2	TetraCB IDA
	323.□□34	M	PentaCB
	32□.□□04	M□2	PentaCB
	32□.□□□□	M □4	PentaCB
	330.□□□2	Lock	PF□
	33□.□20□	M□2	PentaCB IDA, IS
	33□.□1□□	M □4	PentaCB IDA, IS
	3□□.□41□	M□2	□exaCB
	361.□3□□	M□4	□exaCB
	363.□3□6	M □6	□exaCB
	3□1.□□1□	M□2	□exaCB IDA
	3□3.□□□□	M□4	□exaCB IDA
	3□3.□02□	M□2	□eptaCB
	3□□.□□□□	M□4	□eptaCB
	3□□.□□66	M □6	□eptaCB
	40□.□42□	M□2	□eptaCB IDA
	40□.□3□□	M□4	□eptaCB IDA
□	3□4.□□□2	Lock	PF□
	3□□.□41□	M□2	□exaCB
	361.□3□□	M□4	□exaCB
	363.□3□6	M □6	□exaCB
	3□1.□□1□	M□2	□exaCB IDA
	3□3.□□□□	M□4	□exaCB IDA
	3□3.□02□	M□2	□eptaCB
	3□□.□□□□	M□4	□eptaCB
	3□□.□□66	M □6	□eptaCB
	40□.□42□	M□2	□eptaCB IDA
	40□.□3□□	M□4	□eptaCB IDA
	42□.□63□	M□2	Octa CB
	42□.□606	M□4	Octa CB
	431.□□□6	M □6	Octa CB
	43□.□03□	M□2	OctaCB IDA
	441.□00□	M□4	OctaCB IDA
	4□4.□□□2	Lock	PF□
6	3□3.□02□	M□2	□eptaCB
	3□□.□□□□	M□4	□eptaCB
	3□□.□□66	M □6	□eptaCB
	40□.□42□	M□2	□eptaCB IDA
	40□.□3□□	M□4	□eptaCB IDA
	42□.□63□	M□2	Octa CB

Descriptor	Accurate Mass	Ion I.D.	Analyte
	42□.□606	M□4	Octa CB
	431.□□□6	M □6	Octa CB
	43□.□03□	M□2	OctaCB IDA
	441.□00□	M□4	OctaCB IDA
	442.□□2□	QC	PF□
	4□4.□□□2	Lock	PF□
	461.□246	M □2	□onaCB
	463.□216	M □4	□onaCB
	46□.□1□□	M □6	□onaCB
	4□3.□64□	M □2	□onaCB IDA
	4□□.□61□	M □4	□onaCB IDA
	4□□.6□□6	M □2	DecaCB
	4□□.6□26	M □4	DecaCB
	4□□.6□□□	M □6	DecaCB
	□0□.□2□□	M □2	DecaCB IDA
	□0□.□22□	M □4	DecaCB IDA
	□11.□1□□	M □6	DecaCB IDA

TABLE 1
HRGC/HRMS Calibration Concentrations

Compound	IUPAC #s	Concentration (pg/µL in dodecane)					
		Optional CS1	CS2	CS3	CS4	CS5	CS6
Target Analytes (TA's)							
MonoPCB	1,3	0.2	1	5	20	400	1000
DiPCB	4,15	0.2	1	5	20	400	1000
TriPCB	19,37	0.2	1	5	20	400	1000
TetraPCB	54,70,81	0.2	1	5	20	400	1000
PentaPCB	104,105,114,118,123,126	0.2	1	5	20	400	1000
HexaPCB	155, 156,157,167,169	0.2	1	5	20	400	1000
HeptaPCB	189,194	0.2	1	5	20	400	1000
OctaPCB	202,205	0.2	1	5	20	400	1000
NonaPCB	206,208	0.2	1	5	20	400	1000
DecaPCB	209	0.2	1	5	20	400	1000
Isotope Dilution Analytes (IDA)							
^{13}C-MonoPCB	1,3	100	100	100	100	100	100
^{13}C-DiPCB	4,15	100	100	100	100	100	100
^{13}C-TriPCB	19,37	100	100	100	100	100	100
^{13}C-TetraPCB	54, 70, 81	100	100	100	100	100	100
^{13}C-PentaPCB	104,105,114,118,123,126	100	100	100	100	100	100
^{13}C-HexaPCB	155,156,157,167,169	100	100	100	100	100	100
^{13}C-HeptaPCB	189,194	100	100	100	100	100	100
^{13}C-OctaPCB	202,205	100	100	100	100	100	100
^{13}C-NonaPCB	206,208	100	100	100	100	100	100
^{13}C-DecaPCB	209	100	100	100	100	100	100
Internal Standards (IS)							
^{13}C-DiPCB	15	100	100	100	100	100	100
^{13}C-TetraPCB	52	100	100	100	100	100	100
^{13}C-PentaPCB	101	100	100	100	100	100	100
^{13}C-HexaPCB	138	100	100	100	100	100	100
^{13}C-OctaPCB	194	100	100	100	100	100	100
Cleanup Recovery Surrogates (S)							
^{13}C-TriPCB	28	100	100	100	100	100	100
^{13}C-PentaPCB	111	100	100	100	100	100	100
^{13}C-HeptaPCB	188	100	100	100	100	100	100

T□□□□□□

C□□ □□□□□□□□□ □□□□□□M□□□□□□ □□□□□□F□□□□□□□□□□□□ □□□□□□□□□

Compound □□nlabelled□	I□PAC □s□	Concentration □pg/uL in Dodecane□
MonoPCB	1,3	20
DiPCB	4,1□	20
TriPCB	1□,3□	20
TetraPCB	□4,□□,□1	20
PentaPCB	104,10□,114, 11□,123,126	20
□exaPCB	1□□, 1□6,1□,16□16□	20
□eptaPCB	1□□,1□□	20
OctaPCB	202,20□	20
□onaPCB	206,20□	20
DecaPCB	20□	20

T
A

S□□AME	Compound	CAS □o	S□□AME	Compound	CAS □o
PCB 1 □B□□	2-Chlorobiphenyl	20□1-60-□	PCB 106 □B□□	2,3,3□,4,□-Pentachlorobiphenyl	□0424-6□-0
			PCB 10□ □B□□		
PCB 2 □B□□	3-Chlorobiphenyl	20□1-61-□	10□ □1□PAC□	2,3,3□,4□,□-Pentachlorobiphenyl	□0424-6□-□
			PCB 10□ □B□□		
PCB 3 □B□□	Biphenyl, 4-chloro	20□1-62-□	10□ □1□PAC□	2,3,3□,4,□-Pentachlorobiphenyl	□0362-41-3
			PCB 10□ □B□□		
PCB 4 □B□□	2,2□-Dichlorobiphenyl	1302□-0□-□	10□ □1□PAC□	2,3,3□,4,6-Pentachlorobiphenyl	□44□2-3□-□
PCB □ □B□□	2,3-Dichlorobiphenyl	1660□-□1-□	PCB 110 □B□□	2,3,3□,4□,6-Pentachlorobiphenyl	3□3□0-03-□
PCB 6 □B□□	2,3□-Dichlorobiphenyl	2□□6□-□0-6	PCB 111 □B□□	2,3,3□,□,□-Pentachlorobiphenyl	3□63□-32-0
PCB □ □B□□	2,4-Dichlorobiphenyl	332□4-□0-3	PCB 112 □B□□	2,3,3□,□,6-Pentachlorobiphenyl	□44□2-36-□
PCB □ □B□□	2,4□-Dichlorbiphenyl	34□□3-43-□	PCB 113 □B□□	2,3,3□,□,6-Pentachlorobiphenyl	6□1□4-10-□
PCB □ □B□□	2,□-Dichlorobiphenyl	34□□3-3□-1	PCB 114 □B□□	2,3,4,4□,□-Pentachlorobiphenyl	□44□2-3□-0
PCB 10 □B□□	2,6-Dichlorobiphenyl	33146-4□-1	PCB 11□ □B□□	2,3,4,4□,6-Pentachlorobiphenyl	□44□2-3□-1
PCB 11 □B□□	3,3□-Dichlorobiphenyl	20□0-6□-1	PCB 116 □B□□	2,3,4,□,6-Pentachlorobiphenyl	1□2□□-0□-□
PCB 12 □B□□	3,4-Dichlorobiphenyl	2□□4-□2-□	PCB 11□ □B□□	2,3,4□,□,6-Pentachlorobiphenyl	6□1□4-11-6
PCB 13 □B□□	3,4□-Dichlorobiphenyl	2□□4-□0-□	PCB 11□ □B□□	2,3□,4,4□,□-Pentachlorobiphenyl	31□0□-00-6
PCB 14 □B□□	3,□-Dichlorobiphenyl	34□□3-41-□	PCB 11□ □B□□	2,3□,4,4□,6-Pentachlorobiphenyl	□6□□□-1□-□
PCB 1□ □B□□	4,4□-Dichlorobiphenyl	20□0-6□-2	PCB 120 □B□□	2,3□,4,□,□-Pentachlorobiphenyl	6□1□4-12-□
PCB 16 □B□□	2,2□,3-Trichlorobiphenyl	3□444-□□-□	PCB 121 □B□□	2,3□,4,□,6-Pentachlorobiphenyl	□6□□□-1□-0
PCB 1□ □B□□	2,2□,4-Trichlorobiphenyl	3□6□0-66-3	PCB 122 □B□□	2□,3,3□,4,□-Pentachlorobiphenyl	□6□42-0□-4
PCB 1□ □B□□	2,2□,□-Trichlorobiphenyl	3□6□0-6□-2	PCB 123 □B□□	2□,3,4,4□,□-Pentachlorobiphenyl	6□□10-44-3
PCB 1□ □B□□	2,2□,6-Trichlorobiphenyl	3□444-□3-4	PCB 124 □B□□	2□,3,4,□,□-Pentachlorobiphenyl	□0424-□0-3
PCB 20 □B□□	2,3,3□-Trichlorobiphenyl	3□444-□4-□	PCB 12□ □B□□	2□,3,4,□,6-Pentachlorobiphenyl	□44□2-3□-2
PCB 21 □B□□	2,3,4-Trichlorobiphenyl	□□□02-46-0	PCB 126 □B□□	3,3□,4,4□,□-Pentachlorobiphenyl	□□46□-2□-□
PCB 22 □B□□	2,3,4□-Trichlorobiphenyl	3□444-□□-□	PCB 12□ □B□□	3,3□,4,□,□-Pentachlorobiphenyl	3□63□-33-1
PCB 23 □B□□	2,3,□-Trichlorobiphenyl	□□□20-44-0	PCB 12□ □B□□	2,2□,3,3□,4,4□-□exachlorobiphenyl	3□3□0-0□-3
PCB 24 □B□□	2,3,6-Trichlorobiphenyl	□□□02-4□-□	PCB 12□ □B□□	2,2□,3,3□,4,□-□exachlorobiphenyl	□□21□-1□-4
PCB 2□ □B□□	2,3□,4-Trichlorobiphenyl	□□□12-3□-3	PCB 130 □B□□	2,2□,3,3□,4,□-□exachlorobiphenyl	□2663-66-□
PCB 26 □B□□	2,3□,□-Trichlorobiphenyl	3□444-□1-4	PCB 131 □B□□	2,2□,3,3□,4,6-□exachlorobiphenyl	61□□□-□0-□
PCB 2□ □B□□	2,3□,6-Trichlorobiphenyl	3□444-□6-□	PCB 132 □B□□	2,2□,3,3□,4,6□-□exachlorobiphenyl	3□3□0-0□-1
PCB 2□ □B□□	2,4,4□-Trichlorobiphenyl	□012-3□-□	PCB 133 □B□□	2,2□,3,3□,□,□-□exachlorobiphenyl	3□6□4-04-3
PCB 2□ □B□□	2,4,□-Trichlorobiphenyl	1□□62-0□-4	PCB 134 □B□□	2,2□,3,3□,□,6-□exachlorobiphenyl	□2□04-□0-□
PCB 30 □B□□	2,4,6-Trichlorobiphenyl	3□6□3-□2-6	PCB 13□ □B□□	2,2□,3,3□,□,6□-□exachlorobiphenyl	□2□44-13-□
PCB 31 □B□□	2,4□,□-Trichlorobiphenyl	16606-02-3	PCB 136 □B□□	2,2□,3,3□,6,6□-□exachlorobiphenyl	3□411-22-2
PCB 32 □B□□	2,4□,6-Trichlorobiphenyl	3□444-□□-□	PCB 13□ □B□□	2,2□,3,4,4□,□-□exachlorobiphenyl	3□6□4-06-□
PCB 33 □B□□	2□,3,4-Trichlorobiphenyl	3□444-□6-□	PCB 13□ □B□□	2,2□,3,4,4□,□□-□exachlorobiphenyl	3□06□-2□-2
PCB 34 □B□□	2□,3,□-Trichlorobiphenyl	3□6□0-6□-□	PCB 13□ □B□□	2,2□,3,4,4□,6-□exachlorobiphenyl	□6030-□6-□
PCB 3□ □B□□	3,3□,4-Trichlorobiphenyl	3□6□0-6□-6	PCB 140 □B□□	2,2□,3,4,4□,6□-□exachlorobiphenyl	□□2□1-64-4
PCB 36 □B□□	3,3□,□-Trichlorobiphenyl	3□444-□□-0	PCB 141 □B□□	2,2□,3,4,□,□□-□exachlorobiphenyl	□2□12-04-6
PCB 3□ □B□□	3,4,4□-Trichlorobiphenyl	3□444-□0-□	PCB 142 □B□□	2,2□,3,4,□,6-□exachlorobiphenyl	41411-61-4
PCB 3□ □B□□	3,4,□-Trichlorobiphenyl	□3□□□-66-1	PCB 143 □B□□	2,2□,3,4,□,6□-□exachlorobiphenyl	6□1□4-1□-0
PCB 3□ □B□□	3,4□,□-Trichlorobiphenyl	3□444-□□-1	PCB 144 □B□□	2,2□,3,4,□,6-□exachlorobiphenyl	6□1□4-14-□
PCB 40 □B□□	2,2□,3,3□-Tetrachlorobiphenyl	3□444-□3-□	PCB 14□ □B□□	2,2□,3,4,6,6□-□exachlorobiphenyl	□44□2-40-□
PCB 41 □B□□	2,2□,3,4-Tetrachlorobiphenyl	□2663-□□-□	PCB 146 □B□□	2,2□,3,4□,□,□-□exachlorobiphenyl	□1□0□-16-□
PCB 42 □B□□	2,2□,3,4□-Tetrachlorobiphenyl	36□□□-22-□	PCB 14□ □B□□	2,2□,3,4□,□,6-□exachlorobiphenyl	6□1□4-13-□
PCB 43 □B□□	2,2□,3,□-Tetrachlorobiphenyl	□0362-46-□	PCB 14□ □B□□	2,2□,3,4□,□,6□-□exachlorobiphenyl	□44□2-41-6
PCB 44 □B□□	2,2□,3,□□-Tetrachlorobiphenyl	41464-3□-□	PCB 14□ □B□□	2,2□,3,4□,□□,6-□exachlorobiphenyl	3□3□0-04-0
PCB 4□ □B□□	2,2□,3,6-Tetrachlorobiphenyl	□0362-4□-□	PCB 1□0 □B□□	2,2□,3,4□,6,6□-□exachlorobiphenyl	6□1□4-0□-1
PCB 46 □B□□	2,2□,3,6□-Tetrachlorobiphenyl	41464-4□-□	PCB 1□1 □B□□	2,2□,3,□,□,6-□exachlorobiphenyl	□2663-63-□

S□□AME	Compound	CAS □o	S□□AME	Compound	CAS □o
PCB 4□ □B□□	2,2□,4,4□-Tetrachlorobiphenyl	243□-□□-□	PCB 1□2 □B□□	2,2□,3,□,6,6□-□exachlorobiphenyl	6□1□4-0□-2
PCB 4□ □B□□	2,2□,4,□-Tetrachlorobiphenyl	□0362-4□-□	PCB 1□3 □B□□	2,2□,4,4□,□,□-□exachlorobiphenyl	3□06-□2-□1
PCB 4□ □B□□	2,2□,4,□-Tetrachlorobiphenyl	41464-40-□	PCB 1□4 □B□□	2,2□,4,4□,□,6-□exachlorobiphenyl	6014-□22-4
PCB □0 □B□□	2,2□,4,6-Tetrachlorobiphenyl	62□□6-6□-0	PCB 1□□ □B□□	2,2□,4,4□,6,6□-□exachlorobiphenyl	33□□□-03-2
PCB □1 □B□□	2,2□,4,6□-Tetrachlorobiphenyl	6□1□4-04-□	PCB 1□6 □B□□	2,3,3□,4,4□,□-□exachlorobiphenyl	3□3□0-0□-4
PCB □2 □B□□	2,2□,□,□-Tetrachlorobiphenyl	3□6□3-□□-3	PCB 1□□ □B□□	2,3,3□,4,4□,□□-□exachlorobiphenyl	6□□□2-□0-□
PCB □3 □B□□	2,2□,□,6-Tetrachlorobiphenyl	41464-41-□	PCB 1□□ □B□□	2,3,3□,4,4□,6-□exachlorobiphenyl	□44□2-42-□
PCB □4 □B□□	2,2□,6,6□-Tetrachlorobiphenyl	1□□6□-0□-□	PCB 1□□ □B□□	2,3,3□,4,□,□-□exachlorobiphenyl	3□63□-3□-3
PCB □□ □B□□	2,3,3□,4-Tetrachlorobiphenyl	□433□-24-2	PCB 160 □B□□	2,3,3□,4,□,6-□exachlorobiphenyl	41411-62-□
PCB □6 □B□□	2,3,3□,4□-Tetrachlorobiphenyl	41464-43-1	PCB 161 □B□□	2,3,3□,4,□,6-□exachlorobiphenyl	□44□2-43-□
PCB □□ □B□□	2,3,3□,□-Tetrachlorobiphenyl	□0424-6□-□	PCB 162 □B□□	2,3,3□,4□,□,□-□exachlorobiphenyl	3□63□-34-2
PCB □□ □B□□	2,3,3□,□□-Tetrachlorobiphenyl	41464-4□-□	PCB 163 □B□□	2,3,3□,4□,□,6-□exachlorobiphenyl	□44□2-44-□
PCB □□ □B□□	2,3,3□,6-Tetrachlorobiphenyl	□44□2-33-6	PCB 164 □B□□	2,3,3□,4□,□□,6-□exachlorobiphenyl	□44□2-4□-0
PCB 60 □B□□	2,3,4,4□-Tetrachlorobiphenyl	3302□-41-1	PCB 16□ □B□□	2,3,3□,□,□,6-□exachlorobiphenyl	□44□2-46-1
PCB 61 □B□□	2,3,4,□-Tetrachlorobiphenyl	332□4-□3-6	PCB 166 □B□□	2,3,4,4□,□,6-□exachlorobiphenyl	41411-63-6
PCB 62 □B□□	2,3,4,6-Tetrachlorobiphenyl	□4230-22-□	PCB 16□ □B□□	2,3□,4,4□,□,□-□exachlorobiphenyl	□2663-□2-6
PCB 63 □B□□	2,3,4□,□-Tetrachlorobiphenyl	□44□2-34-□	PCB 16□ □B□□	2,3□,4,4□,□,6-□exachlorobiphenyl	□□2□1-6□-□
PCB 64 □B□□	2,3,4□,6-Tetrachlorobiphenyl	□2663-□□-□	PCB 16□ □B□□	3,3□,4,4□,□,□-□exachlorobiphenyl	32□□4-16-6
PCB 6□ □B□□	2,3,□,6-Tetrachlorobiphenyl	332□4-□4-□	PCB 1□0 □B□□	2,2□,3,3□,4,4□,□- □eptachlorobiphenyl	3□06□-30-6
PCB 66 □B□□	2,3□,4,4□-Tetrachlorobiphenyl	32□□□-10-0	PCB 1□1 □B□□	2,2□,3,3□,4,4□,6- □eptachlorobiphenyl	□2663-□1-□
PCB 6□ □B□□	2,3□,4,□-Tetrachlorobiphenyl	□3□□□-□3-□	PCB 1□2 □B□□	2,2□,3,3□,4,□,□- □eptachlorobiphenyl	□2663-□4-□
PCB 6□ □B□□	2,3□,4,□□-Tetrachlorobiphenyl	□3□□□-□2-□	PCB 1□3 □B□□	2,2□,3,3□,4,□,6-□eptachlorobiphenyl	6□1□4-16-1
PCB 6□ □B□□	2,3□,4,6-Tetrachlorobiphenyl	60233-24-1	PCB 1□4 □B□□	2,2□,3,3□,4,□,6□- □eptachlorobiphenyl	3□411-2□-□
PCB □0 □B□□	2,3□,4,□,□-Tetrachlorobiphenyl	32□□□-11-1	PCB 1□□ □B□□	2,2□,3,3□,4,□,□,6- □eptachlorobiphenyl	401□6-□0-□
PCB □1 □B□□	2,3□,4□,6-Tetrachlorobiphenyl	41464-46-4	PCB 1□6 □B□□	2,2□,3,3□,4,4,6,6□- □eptachlorobiphenyl	□2663-6□-□
PCB □2 □B□□	2,3□,□,□□-Tetrachlorobiphenyl	41464-42-0	PCB 1□□ □B□□	2,2□,3,3□,4,□,□,6- □eptachlorobiphenyl	□2663-□0-4
PCB □3 □B□□	2,3□,□□,6-Tetrachlorobiphenyl	□433□-23-1	PCB 1□□ □B□□	2,2□,3,3□,□,□,6,6□- □eptachlorobiphenyl	□2663-64-6
PCB □4 □B□□	2,4,4□,□-Tetrachlorobiphenyl	326□0-□3-0	PCB 1□□ □B□□	2,2□,3,4,4,□,□,□- □eptachlorobiphenyl	3□06□-2□-3
-PCB □□ □B□□	2,4,4□,6-Tetrachlorobiphenyl	32□□□-12-2	PCB 1□0 □B□□	2,2□,3,4,4,□,□,6-□eptachlorobiphenyl	□44□2-4□-2
PCB □6 □B□□	2□,3,4,□-Tetrachlorobiphenyl	□0362-4□-0	PCB 1□1 □B□□	2,2□,3,4,4,□,□,6□- □eptachlorobiphenyl	6014□-23-□
PCB □□ □B□□	3,3□,4,4□-Tetrachlorobiphenyl	32□□□-13-3	PCB 1□2 □B□□	2,2□,3,4,4□,□□,6- □eptachlorobiphenyl	□2663-6□-1
PCB □□ □B□□	3,3□,4,□-Tetrachlorobiphenyl	□0362-4□-1	PCB 1□3 □B□□	2,2□,3,4,4□,6,6□- □eptachlorobiphenyl	□44□2-4□-3
PCB □□ □B□□	3,3□,4,□□-Tetrachlorobiphenyl	41464-4□-6	PCB 1□4 □B□□	2,2□,3,4,□,□,6-□eptachlorobiphenyl	□2□12-0□-□
PCB □0 □B□□	3,3□,□,□□-Tetrachlorobiphenyl	332□4-□2-□	PCB 1□□ □B□□	2,2□,3,4,□,6,6□-□eptachlorobiphenyl	□44□2-4□-4
PCB □1 □B□□	3,4,4□,□-Tetrachlorobiphenyl	□0362-□0-4	PCB 1□6 □B□□	2,2□,3,4□,□,□,6- □eptachlorobiphenyl	□2663-6□-0
PCB □2 □B□□	2,2□,3,3□,4- Pentachlorobiphenyl	□2663-62-4			

S□□AME	Compound	CAS □o	S□□AME	Compound	CAS □o
	2,2□,3,3□,□-			2,2□,3,4□,□,6,6□-	
PCB □3 □B□□	Pentachlorobiphenyl	6014□-20-2	PCB 1□□ □B□□	□eptachlorobiphenyl	□44□□-□□-□
	2,2□,3,3□,6-			2,3,3□,4,4□,□,□-	
PCB □4 □B□□	Pentachlorobiphenyl	□2663-60-2	PCB 1□□ □B□□	□eptachlorobiphenyl	3□63□-31-□
	2,2□,3,4,4□-				
PCB □□ □B□□	Pentachlorobiphenyl	6□□10-4□-4	PCB 1□0 □B□□	2,3,3□,4,4□,□,6- □eptachlorobiphenyl	41411-64-□
				2,3,3□,4,4□,□□,6-	
PCB □6 □B□□	2,2□,3,4,□-Pentachlorobiphenyl	□□312-6□-1	PCB 1□1 □B□□	□eptachlorobiphenyl	□44□2-□0-□
	2,2□,3,4,□□-				
PCB □□ □B□□	Pentachlorobiphenyl	3□3□0-02-□	PCB 1□2 □B□□	2,3,3□,4,□,□,6- □eptachlorobiphenyl	□44□2-□1-□
				2,3,3□,4□,□,□,6-	
PCB □□ □B□□	2,2□,3,4,6-Pentachlorobiphenyl	□□21□-1□-3	PCB 1□3 □B□□	□eptachlorobiphenyl	6□□□2-□1-□
	2,2□,3,4,6□-			2,2□,3,3□,4,4□,□,□-	
PCB □□ □B□□	Pentachlorobiphenyl	□3□□□-□□-2	PCB 1□4 □B□□	Octachlorobiphenyl	3□6□4-0□-□
	2,2□,3,4□,□-			2,2□,3,3□,4,4□,□,6-	
PCB □0 □B□□	Pentachlorobiphenyl	6□1□4-0□-0	PCB 1□□ □B□□	Octachlorobiphenyl	□2663-□□-2
				2,2□,3,3□,4,6,6□-	
PCB □1 □B□□	2,3□,4□,6-Tetrachlorobiphenyl	41464-46-4	PCB 1□6 □B□□	□eptachlorobiphenyl	□2663-6□-□
				2,2□,3,3□,4,□□,6-	
PCB □2 □B□□	2,3□,□,□-Tetrachlorobiphenyl	41464-42-0	PCB 1□□ □B□□	□eptachlorobiphenyl	□2663-□0-4
				2,2□,3,3□,□,□□,6-	
PCB □3 □B□□	2,3□,□□,6-Tetrachlorobiphenyl	□433□-23-1	PCB 1□□ □B□□	□eptachlorobiphenyl	□2663-6□-□
				2,2□,3,3□,□,6,6□-	
PCB □4 □B□□	2,4,4□,□-Tetrachlorobiphenyl	326□0-□3-0	PCB 1□□ □B□□	□eptachlorobiphenyl	□2663-64-6
				2,2□,3,4,4□,□,□-	
PCB □□ □B□□	2,4,4□,6-Tetrachlorobiphenyl	32□□□-12-2	PCB 1□0 □B□□	□eptachlorobiphenyl	3□06□-2□-3
PCB □6 □B□□	2□,3,4,□-Tetrachlorobiphenyl	□0362-4□-0	PCB 1□1 □B□□	2,2□,3,4,4□,□,6- □eptachlorobiphenyl	□44□2-4□-2
				2,2□,3,4,4□,□,6□-	
PCB □□ □B□□	3,3□,4,4□-Tetrachlorobiphenyl	32□□□-13-3	PCB 1□2 □B□□	□eptachlorobiphenyl	6014□-23-□
				2,2□,3,4,4□,□□,6-	
PCB □□ □B□□	3,3□,4,□-Tetrachlorobiphenyl	□0362-4□-1	PCB 1□3 □B□□	□eptachlorobiphenyl	□2663-6□-1
				2,2□,3,4,4□,6,6□-	
PCB □□ □B□□	3,3□,4,□□-Tetrachlorobiphenyl	41464-4□-6	PCB 1□4 □B□□	□eptachlorobiphenyl	□44□2-4□-3
PCB □0 □B□□	3,3□,□,□□-Tetrachlorobiphenyl	332□4-□2-□	PCB 1□□ □B□□	2,2□,3,4,□,□,6- □eptachlorobiphenyl	□2□12-0□-□
PCB □1 □B□□	3,4,4□,□-Tetrachlorobiphenyl	□0362-□0-4	PCB 1□6 □B□□	2,2□,3,4,□,6,6□- □eptachlorobiphenyl	□44□2-4□-4
	2,2□,3,3□,4-			2,2□,3,4□,□,□,6-	
PCB □2 □B□□	Pentachlorobiphenyl	□2663-62-4	PCB 1□□ □B□□	□eptachlorobiphenyl	□2663-6□-0
	2,2□,3,3□,□-			2,2□,3,4□,□,6,6□-	
PCB □3 □B□□	Pentachlorobiphenyl	6014□-20-2	PCB 1□□ □B□□	□eptachlorobiphenyl	□44□□-□□-□
	2,2□,3,3□,6-			2,3,3□,4,4□,□,□-	
PCB □4 □B□□	Pentachlorobiphenyl	□2663-60-2	PCB 1□□ □B□□	□eptachlorobiphenyl	3□63□-31-□
	2,2□,3,4,4□-				
PCB □□ □B□□	Pentachlorobiphenyl	6□□10-4□-4	PCB 1□0 □B□□	2,3,3□,4,4□,□,6- □eptachlorobiphenyl	41411-64-□
				2,3,3□,4,4□,□□,6-	
PCB □6 □B□□	2,2□,3,4,□-Pentachlorobiphenyl	□□312-6□-1	PCB 1□1 □B□□	□eptachlorobiphenyl	□44□2-□0-□
	2,2□,3,4,□□-				
PCB □□ □B□□	Pentachlorobiphenyl	3□3□0-02-□	PCB 1□2 □B□□	2,3,3□,4,□,□,6- □eptachlorobiphenyl	□44□2-□1-□
				2,3,3□,4□,□,□,6-	
PCB □□ □B□□	2,2□,3,4,6-Pentachlorobiphenyl	□□21□-1□-3	PCB 1□3 □B□□	□eptachlorobiphenyl	6□□□2-□1-□
	2,2□,3,4,6□-			2,2□,3,3□,4,4□,□,□-	
PCB □□ □B□□	Pentachlorobiphenyl	□3□□□-□□-2	PCB 1□4 □B□□	Octachlorobiphenyl	3□6□4-0□-□

S□□AME	Compound	CAS □o
PCB □0 □B□□	2,2□,3,4,□□-Pentachlorobiphenyl	6□1□4-0□-0
PCB □1 □B□□	2,2□,3,4,6-Pentachlorobiphenyl	6□1□4-0□-□
PCB □2 □B□□	2,2□,3,□,□□-Pentachlorobiphenyl	□2663-61-3
PCB □3 □B□□	2,2□,3,□,6-Pentachlorobiphenyl	□3□□□-□6-1
PCB □4 □B□□	2,2□,3,□,6□-Pentachlorobiphenyl	□3□□□-□□-0
PCB □□ □B□□	2,2□,3,□□,6-Pentachlorobiphenyl	3□3□□-□□-6
PCB □6 □B□□	2,2□,3,6,6□-Pentachlorobiphenyl	□3□□□-□4-□
PCB □□ □B□□	2,2□,3□,4,□□-Pentachlorobiphenyl	41464-□1-1
PCB □□ □B□□	2,2□,3□,4,6-Pentachlorobiphenyl	60233-2□-2
PCB □□ □B□□	2,2□,4,4□,□□-Pentachlorobiphenyl	3□3□0-01-□
PCB 100 □B□□	2,2□,4,4□,6-Pentachlorobiphenyl	3□4□□-□3-1
PCB 101 □B□□	2,2□,4,□,□□-Pentachlorobiphenyl	3□6□0-□3-2
PCB 102 □B□□	2,2□,4,□,6□-Pentachlorobiphenyl	6□1□4-06-□
PCB 103 □B□□	2,2□,4,□□,6-Pentachlorobiphenyl	6014□-21-3
PCB 104 □B□□	2,2□,4,6,6□-Pentachlorobiphenyl	□6□□□-16-□
PCB 10□ □B□□	2,3,3□,4,4□-Pentachlorobiphenyl	32□□□-14-4
PCB 1□□ □B□□	2,2□,3,3□,4,4□,□□,6-Octachlorobiphenyl	□2663-□□-2
PCB 1□6 □B□□	2,2□,3,3□,4,4□,□□,6□-Octachlorobiphenyl	42□40-□0-1
PCB 1□□ □B□□	2,2□,3,3□,4,4□,6,6□-Octachlorobiphenyl	330□1-1□-□
PCB 1□□ □B□□	2,2□,3,3□,4,□,□□,6-Octachlorobiphenyl	6□1□4-1□-2
PCB 1□□ □B□□	2,2□,3,3□,4,□,6,6□-Octachlorobiphenyl	□2663-□3-□
200 □1□PAC□ PCB 200 □B□□	2,2□,3,3□,4,□□,6,6□-Octachlorobiphenyl	401□6-□1-□
201 □1□PAC□ PCB 201 □B□□	2,2□,3,3□,4,□,□□,6□-Octachlorobiphenyl	□2663-□□-□
1□□ □1□PAC□	2,2□,3,3□,□□,□□,6,6□-Octachlorobiphenyl	2136-□□-4
PCB 202 □B□□	octachlorobiphenyl	□2663-□6-0
PCB 203 □B□□	2,2□,3,4,4□,□□,6-Octachlorobiphenyl	□44□2-□2-□
PCB 204 □B□□	2,2□,3,4,4□,□□,6,6□-Octachlorobiphenyl	□44□2-□3-0
PCB 20□ □B□□	2,3,3□,4,4□,□□,□□,6-Octachlorobiphenyl	401□6-□2-□
PCB 206 □B□□	2,2□,3,3□,4,4□,□□,6,6□-□onachlorobiphenyl	□2663-□□-3
PCB 20□ □B□□	2,2□,3,3□,4,□,□□,6,6□-□onachlorobiphenyl	□2663-□□-1
PCB 20□ □B□□	□onachlorobiphenyl	20□1-24-3
	Decachlorobiphenyl	

T□□□□ □ □□□

M□□□□□ □□□□C □C A□□□□□□□□C□□□□□□ □□□ □ER□PR□OPR □□□ □ □□□□□□ □□□ □□□□□□□□ □□□ □□□□

Congener □ame	Test Concentration □ng/ml□	□ER □□ □	IPR RSD □□ □	IPR Mean Recovery □□ □	OPR Recovery □□ □	Labeled compound Recovery in Samples □□ □
PCB-1	100	□□-12□	2□	□0-130	60-13□	NA
PCB-3	100	□□-12□	2□	□0-130	60-13□	
PCB-4	100	□□-12□	2□	□0-130	60-13□	
PCB-1□	100	□□-12□	2□	□0-130	60-13□	
PCB-1□	100	□□-12□	2□	□0-130	60-13□	
PCB-3□	100	□□-12□	2□	□0-130	60-13□	
PCB-□4	100	□□-12□	2□	□0-130	60-13□	
PCB-□□	100	□□-12□	2□	□0-130	60-13□	
PCB-□1	100	□□-12□	2□	□0-130	60-13□	
PCB-104	100	□□-12□	2□	□0-130	60-13□	
PCB-10□	100	□□-12□	2□	□0-130	60-13□	
PCB-114	100	□□-12□	2□	□0-130	60-13□	
PCB-11□	100	□□-12□	2□	□0-130	60-13□	
PCB-123	100	□□-12□	2□	□0-130	60-13□	
PCB-126	100	□□-12□	2□	□0-130	60-13□	
PCB-1□□	100	□□-12□	2□	□0-130	60-13□	
PCB-1□6	100	□□-12□	2□	□0-130	60-13□	
PCB-1□□	100	□□-12□	2□	□0-130	60-13□	
PCB-16□	100	□□-12□	2□	□0-130	60-13□	
PCB-16□	100	□□-12□	2□	□0-130	60-13□	
PCB-1□□	100	□□-12□	2□	□0-130	60-13□	
PCB-1□□	100	□□-12□	2□	□0-130	60-13□	
PCB-202	100	□□-12□	2□	□0-130	60-13□	
PCB-20□	100	□□-12□	2□	□0-130	60-13□	
PCB-206	100	□□-12□	2□	□0-130	60-13□	
PCB-20□	100	□□-12□	2□	□0-130	60-13□	
PCB-20□	100	□□-12□	2□	□0-130	60-13□	
Internal Standards						
$^{13}C_{12}$-PCB-1	100	□0-14□	□0	20-13□	1□-14□	□-14□
$^{13}C_{12}$-PCB-3	100	□0-14□	□0	20-13□	1□-14□	□-14□
$^{13}C_{12}$-PCB-4	100	□0-14□	□0	20-13□	1□-14□	□-14□
$^{13}C_{12}$-PCB-1□	100	□0-14□	□0	20-13□	1□-14□	□-14□
$^{13}C_{12}$-PCB-1□	100	□0-14□	□0	20-13□	1□-14□	□-14□
$^{13}C_{12}$-PCB-3□	100	□0-14□	□0	20-13□	1□-14□	□-14□
$^{13}C_{12}$-PCB-□4	100	□0-14□	□0	20-13□	1□-14□	□-14□
$^{13}C_{12}$-PCB-□□	100	□0-14□	□0	4□-13□	40-14□	10-14□
$^{13}C_{12}$-PCB-□1	100	□0-14□	□0	4□-13□	40-14□	10-14□
$^{13}C_{12}$-PCB-104	100	□0-14□	□0	4□-13□	40-14□	10-14□
$^{13}C_{12}$-PCB-10□	100	□0-14□	□0	4□-13□	40-14□	10-14□
$^{13}C_{12}$-PCB-114	100	□0-14□	□0	4□-13□	40-14□	10-14□
$^{13}C_{12}$-PCB-11□	100	□0-14□	□0	4□-13□	40-14□	10-14□
$^{13}C_{12}$-PCB-123	100	□0-14□	□0	4□-13□	40-14□	10-14□
$^{13}C_{12}$-PCB-126	100	□0-14□	□0	4□-13□	40-14□	10-14□

$^{13}C_{12}$-PCB-1□□	100	□0-14□	□0	4□-13□	40-14□	10-14□
$^{13}C_{12}$-PCB-1□6	100	□0-14□	□0	4□-13□	40-14□	10-14□
$^{13}C_{12}$-PCB-1□□	100	□0-14□	□0	4□-13□	40-14□	10-14□
$^{13}C_{12}$-PCB-16□	100	□0-14□	□0	4□-13□	40-14□	10-14□
$^{13}C_{12}$-PCB-16□	100	□0-14□	□0	4□-13□	40-14□	10-14□
$^{13}C_{12}$-PCB-1□□	100	□0-14□	□0	4□-13□	40-14□	10-14□
$^{13}C_{12}$-PCB-1□□	100	□0-14□	□0	4□-13□	40-14□	10-14□
$^{13}C_{12}$-PCB-202	100	□0-14□	□0	4□-13□	40-14□	10-14□
$^{13}C_{12}$-PCB-20□	100	□0-14□	□0	4□-13□	40-14□	10-14□
$^{13}C_{12}$-PCB-206	100	□0-14□	□0	4□-13□	40-14□	10-14□
$^{13}C_{12}$-PCB-20□	100	□0-14□	□0	4□-13□	40-14□	10-14□
$^{13}C_{12}$-PCB-20□	100	□0-14□	□0	4□-13□	40-14□	10-14□
Cleanup Standards						
$^{13}C_{12}$-PCB-2□	100	6□-13□	□0	20-13□	1□-14□	□-14□
$^{13}C_{12}$-PCB-111	100	□□-12□	□0	4□-13□	40-14□	10-14□
$^{13}C_{12}$-PCB-1□□	100	□□-12□	□0	4□-13□	40-14□	10-14□

Title: Analysis of Samples for Polychlorinated Dioxins and Furans by HRGC/HRMS

[Methods 8290, 8290A & TO-9A]

Approvals (Signature/Date):

10/29/2019	10/22/2019
Robert Hrabak Date	Joe Schairer Date
Technical Manager	Health & Safety Manager / Coordinator
10/23/2019	10/23/2019
Lisa Stafford Date	Chris Williams Date
Quality Assurance Manager	Laboratory Director

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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 or higher and the Autospec Premiere system utilizes MassLynx version 4.1 or higher software. The Thermo DFS system utilizes the Thermo Fisher XCalibur verison 2.2.0 or higher 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 ≤ 6°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 0 – 6°C, 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 Method TO-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 0 – 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 blank 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.

9.9. If a single client sample has a detection for 2,3,7,8-TCDD by itself and all other site related samples are non-detect or low level for 2,3,7,8-TCDD the data will warrant further investigation. The client will be contacted to discuss site history and expected levels. The sample may warrant re-extraction and re-analysis in conjunction with directions of the client.

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:

$$RRF(n) = \frac{A_x \times Q_{IDA}}{Q_x \times A_{IDA}} \quad RRF(m) = \frac{A_{IDA} \times Q_{IS}}{Q_{IDA} \times A_{IS}}$$

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:

$$RRF(m) = \frac{A^m_{IDA} \times Q_{IS}}{Q^m_{IDA} \times A_{IS}}$$

$$\overline{RRF}(m) = (1/5)\sum_{j=1}^{5} RRF_j(m)$$

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 (unlabeled) 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 ≤ 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.

13.4.1. 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 8.1, Effective 11/07/2019

19.1.1. Section 6.1.1added, “Capable of collecting, recording and storing MS data. The VG70 and Autospec Ultima systems utilize Opus version 3.6 software or higher and the Autospec Premiere system utilizes MassLynx version 4.1 or higher software. The Thermo DFS system utilizes the Thermo Fisher XCalibur verison 2.2.0 or higher software.”

19.1.2. Throughout SOP revised temperature criteria from “4 ± 2°C” to “0 - 6°C”.

19.1.3. Editorial changes.

19.2. WS-ID-0005, Revision 8.0, Effective 02/27/2019

19.2.1. Added Section 9.9, "If a single client sample has a detection for 2,3,7,8-TCDD by itself and all other site related samples are non-detect or low level for 2,3,7,8-TCDD the data will warrant further investigation. The client will be contacted to discuss site history and expected levels. The sample may warrant re-extraction and re-analysis in conjunction with directions of the client."

19.2.2. Removed revision history prior to 2016. It can be found in previous versions of this SOP.

19.2.3. Editorial changes.

19.3. WS-ID-0005, Revision 7.9, Effective 10/07/2016

19.3.1. 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. "

19.3.2. 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 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-HpCDF	100	100	100	100	100
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

	Compound	Concentration (ng/mL)				
RRF (n)(m)		CS2	CS3	CS4 (ICV(6))	CS5	CS6
	$^{13}C_{12}$-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

Descriptor	Exact m/z [1]	m/z Type	Elemental Composition	Substance [2]
	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
	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

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[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

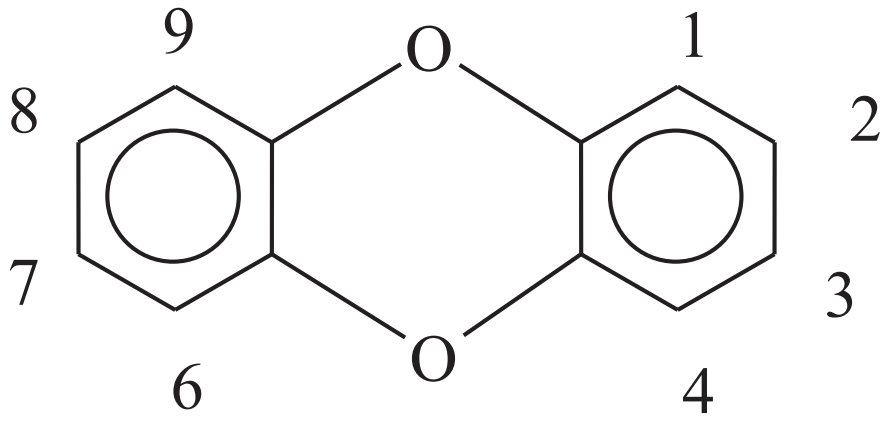
Number	Compound(s)	TEF
1	2,3,7,8-TCDD	1.00
2	1,2,3,7,8-PeCDD	0.50
3	1,2,3,6,7,8-HxCDD	0.10
4	1,2,3,7,8,9-HxCDD	0.10
5	1,2,3,4,7,8-HxCDD	0.10
6	1,2,3,4,6,7,8-HpCDD	0.01
7	OCDD	0.001
8	2,3,6,7-TCDF	0.1
9	1,2,3,7,8-PeCDF	0.05
10	2,3,4,7,8PeCDF	0.5
11	1,2,3,6,7,8-HxCDF	0.1
12	1,2,3,7,8,9-HxCDF	0.1
13	1,2,3,4,7,8-HxCDF	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

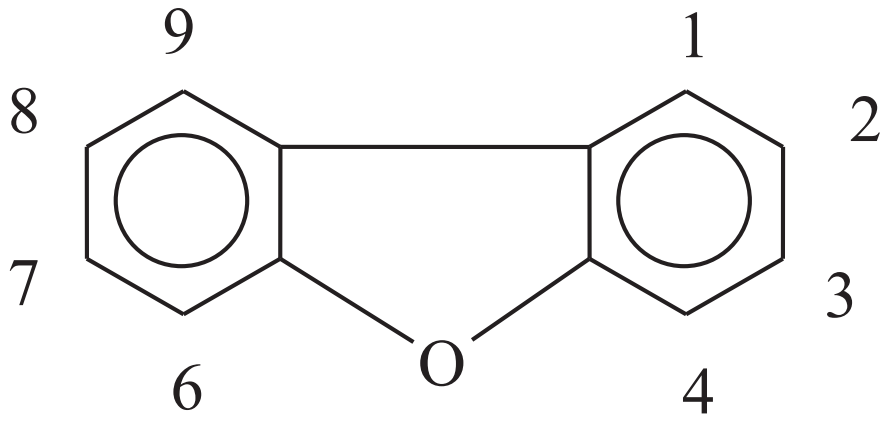
Analyte	Analyte RRT Reference[(a)]
1,2,3,4,7,8-HxCDD	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD
1,2,3,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF
1,2,3,7,8,9-HxCDF	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF
2,3,4,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF

(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

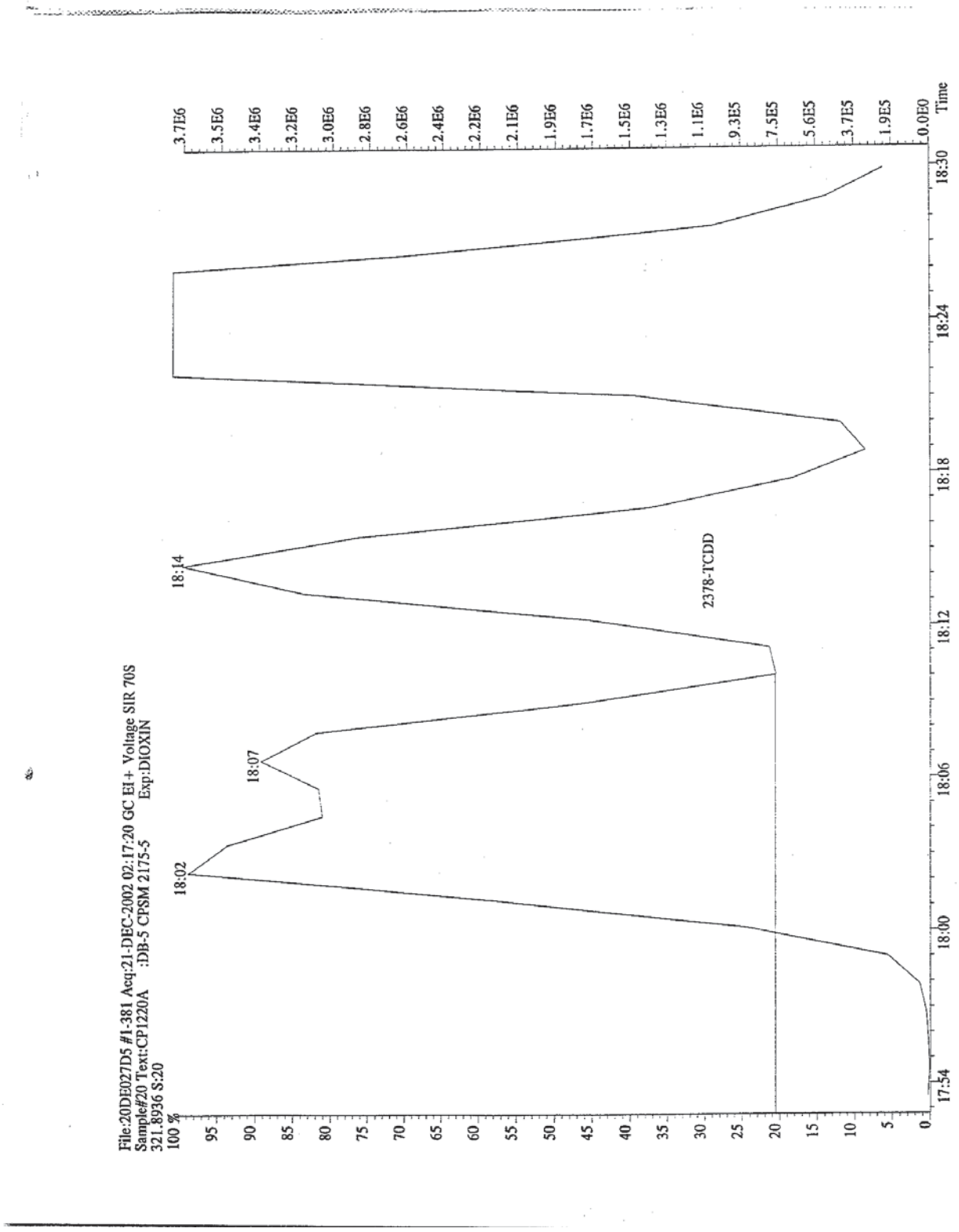

File:20DE027D5 #1-381 Acq:21-DEC-2002 02:17:20 GC EI+ Voltage SIR 70S
Sample#20 Text:CP1220A :DB-5 CPSM 2175-5 Exp:DIOXIN
321.8936 S:20
100 %
18:02
18:07
18:14
2378-TCDD
Time
17:54
18:00
18:06
18:12
18:18
18:24
18:30
3.7E6
3.5E6
3.4E6
3.2E6
3.0E6
2.8E6
2.6E6
2.4E6
2.2E6
2.1E6
1.9E6
1.7E6
1.5E6
1.3E6
1.1E6
9.3E5
7.5E5
5.6E5
3.7E5
1.9E5
0.0E0

FIGURE 3

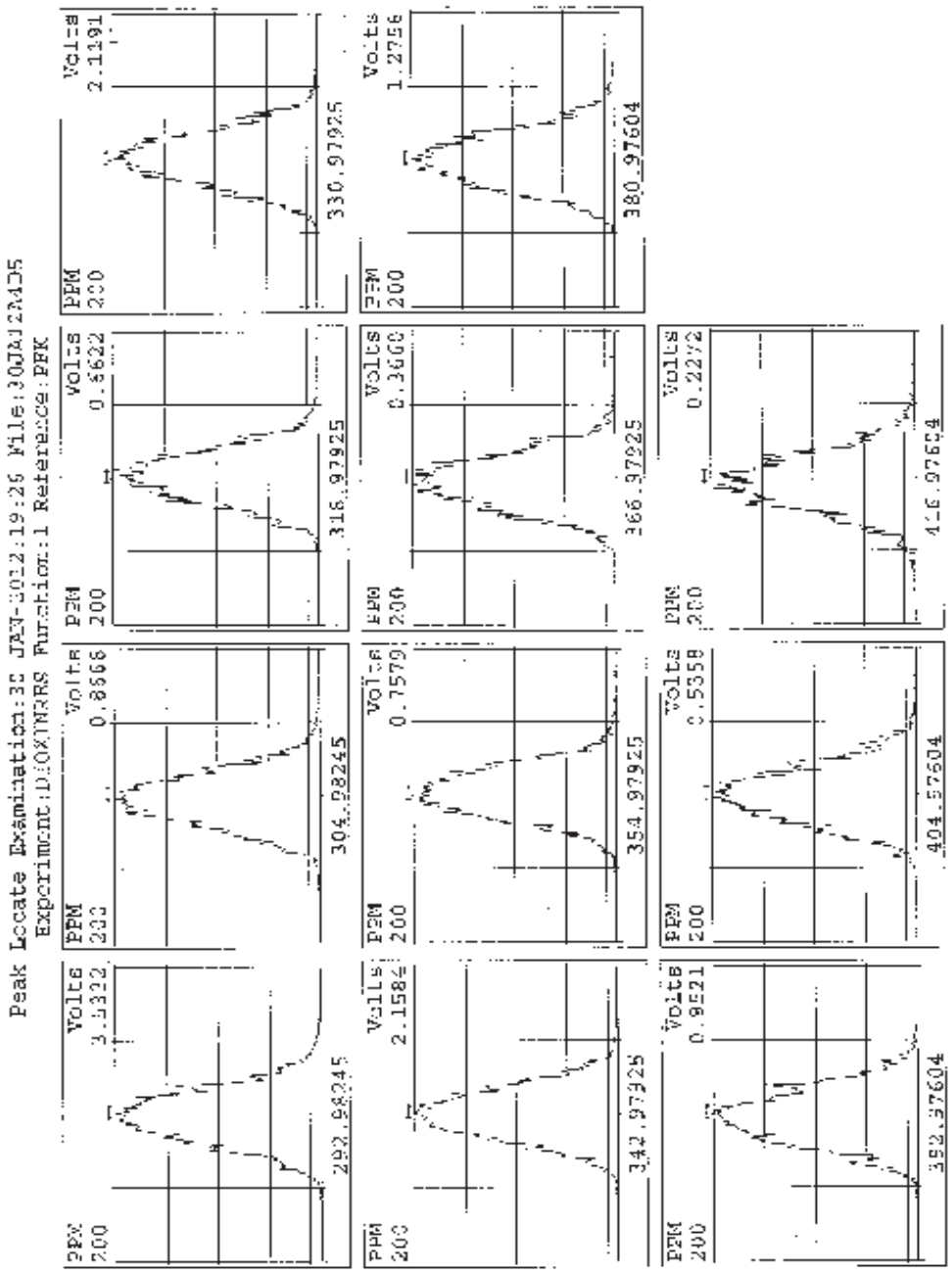
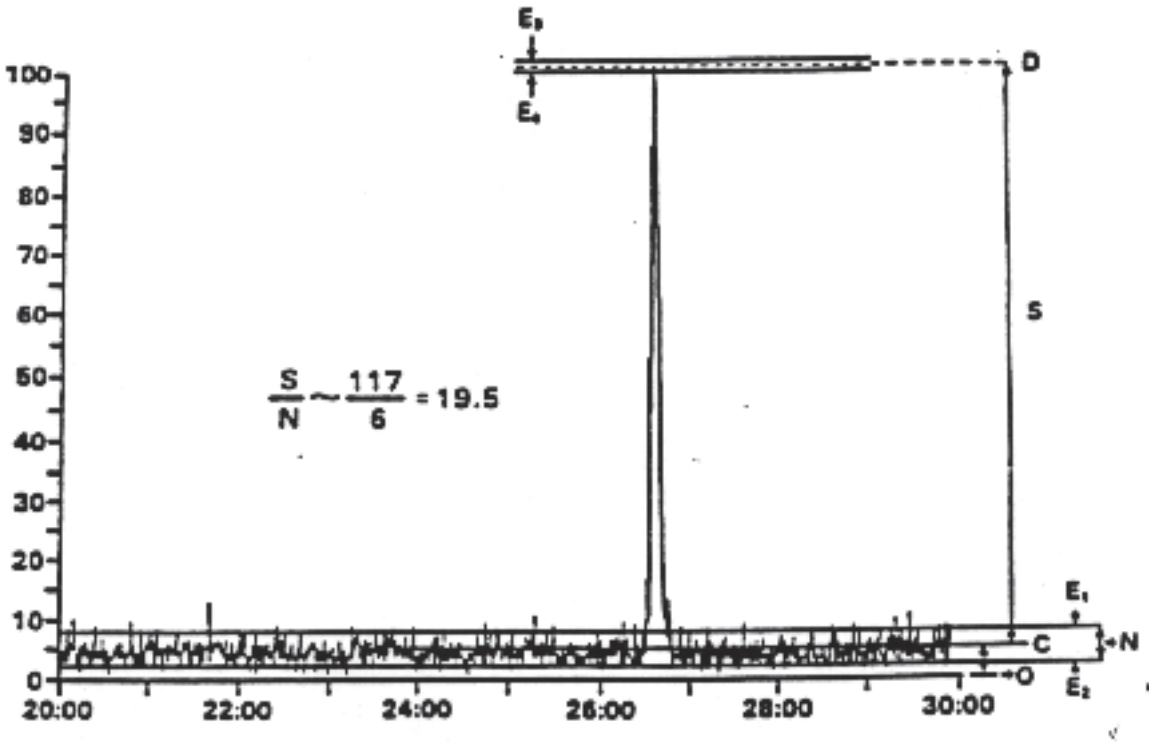

Peak Locate Examination:3C JAN-2012:19:25 File:30JAN2A05
Experiment:D:\OXINRS Purchase:1 Reference:PPK
PPM
200
Volts
3.5332
292.98245
Volts
0.8668
304.98245
Volts
0.1622
316.97925
Volts
2.1191
330.97925
Volts
2.1582
342.97925
Volts
0.7579
354.97925
Volts
0.3663
366.97925
Volts
1.2756
380.97604
Volts
0.9521
392.97604
Volts
0.5358
404.97604
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 (□EPA) 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 E□□S.

Title: Per- and Polyfluorinated Alkyl Substances (PFAS) in Water, Soils, Sediments and Tissue

[Method 537 (Modified), Method PFAS by LCMSMS Compliant with QSM Table B-15, Revision 5.1 and higher]

Approvals (Signature/Date):

09/19/2019	09/20/2019
Robert Hrabak Date	Joe Schairer Date
Technical Manager	Health & Safety Manager / Coordinator
09/20/2019	09/20/2019
Lisa Stafford Date	Chris Williams Date
Quality Assurance Manager	Laboratory Manager

Facility Distribution No. Uncontrolled **Distributed To:** Sacramento QA

1. SCOPE AND APPLICATION

1.1. This procedure describes the analysis of water, soil, sediment, and tissue samples for the following compounds using liquid chromatography / tandem mass spectrometry (LC/MS/MS).

Compound Name	Abbreviation	CAS #
Perfluoroalkylcarboxylic acids (PFCAs)		
Perfluoro-n-butanoic acid	PF□A	375-22-4
Perfluoro-n-pentanoic acid	PFPeA	270□-□0-3
Perfluoro-n-hexanoic acid	PF□xA	307-24-4
Perfluoro-n-heptanoic acid	PF□pA	375-85-□
Perfluoro-n-octanoic acid	PFOA	335-□7-1
Perfluoro-n-nonanoic acid	PFNA	375-□5-1
Perfluoro-n-decanoic acid	PFDA	335-7□-2
Perfluoro-n-undecanoic acid	PFUdA (PFUnA)	2058-□4-8
Perfluoro-n-dodecanoic acid	PFDoA	307-55-1
Perfluoro-n-tridecanoic acid	PFTrDA	72□2-□4-8
Perfluoro-n-tetradecanoic acid	PFTeDA (PFTA)	37□-0□-7
Perfluoro-n-hexadecanoic acid (non-routine analyte)	PF□xDA	□7□05-1□-5
Perfluoro-n-octadecanoic acid (non-routine analyte)	PFODA	1□517-11-□
Perfluorinated sulfonic acids (PFSAs)		
Perfluoro-1-butanesulfonic acid	PF□S	375-73-5
Perfluoro-1-pentanesulfonic acid	PFPeS	270□-□1-4
Perfluoro-1-hexanesulfonic acid	PF□xS	355-4□-4
Perfluoro-1-heptanesulfonic acid	PF□pS	375-□2-8
Perfluoro-1-octanesulfonic acid	PFOS	17□3-23-1
Perfluoro-nonanesulfonic acid	PFNS	□825-□12-1
Perfluoro-1-decanesulfonic acid	PFDS	335-77-3
Perfluoro-1-dodecansulfonic acid	PFDoS	7□780-3□-5
Perfluorinated sulfonamides (FOSA)		
Perfluoro-1-octanesulfonamide	FOSA	754-□1-□
N-ethylperfluoro-1-octanesulfonamide	Et-FOSA	4151-50-2
N-methylperfluoro-1-octanesulfonamide	Me-FOSA	3150□-32-8
Perfluorinated sulfonamide ethanols (FOSE)		
2-(N-ethylperfluoro-1-octanesulfonamido) ethanol	Et-FOSE	1□□-□□-2
2-(N-methylperfluoro-1-octanesulfonamido) ethanol	Me-FOSE	24448-0□-7
Perfluorinated sulfonamidoacetic acids (FOSAA)		

Compound Name	Abbreviation	CAS #
N-ethylperfluoro-1-octanesulfonamidoacetic acid	EtFOSAA	2□□-50-□
N-methylperfluoro-1-octanesulfonamidoacetic acid	MeFOSAA	2355-31-□
Fluorotelomer sulfonates (FTS)		
1□,1□,2□,2□-perfluorohexane sulfonate *(4:2)*	4□2 FTS	757124-72-4
1□,1□,2□,2□-perfluorooctane sulfonate *(6:2)*	□2 FTS	27□1□-□7-2
1□,1□,2□,2□-perfluorodecane sulfonate *(8:2)*	8□2 FTS	3□10834-4
1□,1□,2□,2□-perfluorododecane sulfonate *(10:2)*	10□2 FTS	12022□-□0-0

***Note**: Abbreviations in parenthesis are the abbreviations listed in Method 537/537.1, where they differ from the abbreviation used by the laboratory's LIMS.*

1.2. Additional analytes supported by this method□The following analytes can be supported by this method under special request.

Compound Name	Abbreviation	CAS #
Fluorinated Replacement Chemicals		
4,8-dioxa-3□-perfluorononanoic	Dona (ADONA[(1)])	□1□005□4-4
Perfluoro(2-propoxypropanoic) acid or □exafluoropropylene oxide dimer acid	□FPO-DA or GenX	13252-13-□
F53□ (reported as the summation of the following)	F53□	NA
□-Chlorohexadecafluoro-3-oxanonane-1-sulfonate	F53□ major (□Cl-PF3ONS)	75□42□-58-1
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonate	F5□ minor (11Cl-PF3OUdS)	8332□-8□-□

(1) In some literature, the acronym ADONA refers to the ammonium salt, CAS 958445-44-8, and DONA refers to the parent acid. In Method 537.1, ADONA refers to the parent acid. DONA is the acronym present on the laboratory raw data.

1.3. The working range of the method is listed below. The linear range can be extended by diluting the extracts.

Matrix	Nominal Sample Size	Reporting Limit	Working Range
Water	250 mL	2.0 ng/L – 20 ng/L	2.0 ng/L - 400 ng/L
Soil/Sediment	5 g	0.2 □g/kg – 2.0 □g/kg	0.2 □g/kg - 40 □g/kg
Tissue	1 g	1.0 □g/kg – 10 □g/kg	1.0 □g/kg – 200 □g/kg

1.4. The procedure for the analysis of water samples via in line solid phase extraction (SPE) for a subset of the list in Section 1.1 using liquid chromatography / tandem mass spectrometry (LC/MS/MS) on a [redacted] is described in Attachment 1 of this SOP.

1.5. This procedure also includes direction for preparing and analyzing samples to determine "Total Oxidizable Precursors", which may assist in improving understanding of potential PFAS environmental risk.

1.6. When undertaking projects for the Department of Defense (DoD) and/or the 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. Water samples are extracted using a solid phase extraction (SPE) cartridge. PFAS are eluted from the cartridge with an [redacted] solution.

2.2. Soil/sediment/tissue samples are extracted with a KOH/methanol solution using an orbital shaker for 3 hours followed by sonication for 12 hours. The mixture is centrifuged and the solvent filtered.

2.3. The final 80:20 methanol:water extracts are analyzed by LC/MS/MS. PFAS are separated from other components on a C18 column with a solvent gradient program using [redacted]. The mass spectrometer detector is operated in the electrospray (ESI) negative ion mode for the analysis of PFAS.

2.4. An isotope dilution technique is employed with this method for the compounds of interest. The isotope dilution analytes (IDA) consist of carbon-13 labeled analogs, oxygen-18 labeled analogs, or deuterated analogs of the compounds of interest, and they are spiked into the samples at the time of extraction. This technique allows for the correction for analytical bias encountered when analyzing more chemically complex environmental samples. The isotopically labeled compounds are chemically similar to the compounds of concern and are therefore affected by sample-related interferences to the same extent as the compounds of concern. Compounds that do not have an identically labeled analog are quantitated by the IDA method using a closely related labeled analog.

2.5. Quantitation by the internal standard method is employed for the IDA analytes/recoveries. Peak response is measured as the area of the peak.

2.6. Samples for the "Total Oxidizable Precursor" assay (TOP) are analyzed in two phases – an aliquot is prepared and analyzed as a normal sample, and a second aliquot is subjected to [redacted] prior to solid phase extraction and analysis. The total perfluorocarboxylic acid value is determined for each aliquot, and the difference calculated.

3. DEFINITIONS

3.1. PFCAs: Perfluorocarboxylic acids

3.2. PFSAs: Perfluorinated sulfonic acids

3.3. FOSA: Perfluorinated sulfonamide

3.4. PFOA: Perfluorooctanoic acid

3.5. PFOS: Perfluorooctane sulfonic acid

3.6. PTFE: Polytetrafluoroethylene (e.g. Teflon□)

3.□. SPE: Solid phase extraction

3.8. PP: Polypropylene

3.□. PE: Polyethylene

3.10. HDPE: High density polyethylene

3.11. AFFF: Aqueous Film Forming Foam

3.12. IDA: Isotope dilution analyte

3.13. Further definitions of terms used in this SOP may be found in the glossary of the Laboratory Quality Assurance Manual (QAM).

□. INTERFEREN□ES

4.1. PFAS have been used in a wide variety of manufacturing processes, and laboratory supplies should be considered potentially contaminated until they have been tested and shown to be otherwise. The materials and supplies used during the method validation process have been tested and shown to be clean. These items are listed below in Section 6.

4.2. To avoid contamination of samples, standards are prepared in a ventilation hood in an area separate from where samples are extracted.

4.3. PTFE products can be a source of PFOA contamination. The use of PTFE in the procedure should be avoided or at least thoroughly tested before use. Polypropylene (PP) or polyethylene (PE, HDPE) products may be used in place of PTFE products to minimize PFOA contamination.

4.3.1. Standards and samples are injected from polypropylene autosampler vials with polypropylene screw caps once. Multiple injections may be performed on Primers when conditioning the instrument for analysis.

4.3.2. □andom evaporation losses have been observed with the polypropylene caps causing high IDA recovery after the vial was punctured and sample re-injected. For this reason, it is best to inject standards and samples once in the analytical sequence.

4.3.3. Teflon-lined screw caps have detected PFAS at low concentrations. □epeated injection from the same teflon-lined screw cap have detected PF□A at increasing concentration as each repeated injection was performed, therefore, it is best to use polypropylene screw caps.

4.4. □olumetric glassware and syringes are difficult to clean after being used for solutions containing high levels of PFOA. These items should be labeled for use only with similarly concentrated solutions or verified clean prior to re-use. To the extent possible, disposable labware is used.

4.5. □oth branched and linear PFAS isomers can potentially be found in the environment. Linear and branched isomers are known to exist for PFOS, PFOA, PFHxS, PF□S, EtFOSAA, and MeFOSAA based upon the scientific literature. If multiple isomers are present for one of these PFAS they might be adjacent peaks that completely resolve or not, but usually with a deflection point resolved during peak integration. The later of these peaks matches the retention time of its labeled linear analog. In general, earlier peaks are the branched isomers and are not the result of peak splitting.

As of this writing, only PFOS, PFOA, and PFHxS are commercially available as technical mixtures. These reference standards of the technical mixtures for these specific PFAS are used to ensure that all appropriate peaks are included during peak integration.

4.6. In an attempt to reduce PFOS bias, it is required that m/z 4□□□80 transition be used as the quantitation transition.

4.□. Per the Certificate of Analysis for labeled perfluorohexadecanoic acid ($13C_2$-PFHxDA) produced by Wellington Laboratories, the stock standard contains roughly 0.3□ of native perfluorohexadecanoic acid. This equates to roughly 0.30 ng/L or 0.015 ug/kg of perfluorohexadecanoic acid expected in all samples and blanks.

4.8. Aluminum foil should not be used for this analysis due to the potential for PFAA.

□. SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Sacramento Supplement to the CSM, and this document. 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 □□ □ □□□□□□□□to a supervisor, the EH□S Staff, or a senior manager.

5.1. Specific Safety Concerns

5.1.1. Preliminary toxicity studies indicate that PFAS could have significant toxic effects. In the interest of keeping exposure levels as low as reasonably achievable, PFAS and PFAS samples must be handled in the laboratory as hazardous and toxic chemicals.

5.1.2. 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.

5.1.3. 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. Eye protection that satisfies A□SI □8□1 (as per the TestAmerica Corporate Safety Manual), laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated will be removed and discarded□other gloves will be cleaned immediately.

5.1.5. Perfluorocarboxylic acids are acids and are not compatible with strong bases.

5.1.6. The use of vacuum systems presents the risk of imploding glassware. All glassware used during vacuum operations must be thoroughly inspected prior to each use. □lass 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.

5.1.□. Glass containers are not to be used for “tumbling” soil samples.

5.2. Primary Materials □sed

The following is a list of the materials used in this method, which have a serious or significant hazard rating. □OTE: 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.

M	H	E	S
Acetic Acid (3-2-1)	Corrosive Poison Flammable	10 ppm-TWA 15 ppm-STEL	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. reathing difficulties may occur.
[redacted] (3-0-0)	Corrosive Poison	50 ppm-TWA	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage to the upper respiratory tract. 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 damage, including blindness. rief exposure to 5000 PPM can be fatal.
Hexane (2-3-0)	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. apors may cause irritation to the skin and eyes.
Hydrochloric Acid (3-0-1)	Corrosive Poison	5 ppm (Ceiling)	Can cause pain and severe burns upon inhalation, ingestion, eye or skin contact. Exposure to concentrated solutions may cause deep ulcerations to skin, permanent eye damage, circulatory failure and swallowing may be fatal.
Methanol (2-3-0)	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.
Potassium Hydroxide (3-0-1)	Corrosive Poison		Severe irritant. Can cause severe burns upon inhalation, ingestion, eye or skin contact. Exposure to concentrated solutions may cause severe scarring of tissue, blindness, and may be fatal if swallowed.
[redacted] (2-0-1-O)	Oxidizer	one	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.

M□□□□□□□□[1]	H□□□□□□	E□□□□□□□ □□□ □□[2]	S□□□□□□S□□ □□□□ □□□E□□□□□□
Sodium Hydroxide (3-0-1)	Corrosive Poison	2 mg/cm^3 (Ceiling)	Severe irritant. Can cause severe burns upon inhalation, ingestion, eye or skin contact. Exposure to concentrated solutions may cause severe scarring of tissue, blindness, and may be fatal if swallowed.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

□. E□UI□MENT AND SU□□□IES

6.1. 15 mL polypropylene test tubes with polypropylene screw caps.

6.2. 50 mL graduated plastic centrifuge tubes.

6.3. 125 mL HDPE bottles with HDPE screw caps.

6.4. 250 mL HDPE bottles with HDPE screw caps. The average weight of the HDPE bottles with HDPE screw caps are calibrated once per year. The calibration is performed by weighing 10 bottles with caps and dividing by 10 to get the average weight. The average weight is used in section (11.3.5.1.d).

6.5. Analytical balance capable of accurately weighing to the nearest 0.0001g, and checked for accuracy each day it is used in accordance with WS-QA-0041.

6.6. Extract concentrator or nitrogen manifold with water bath heating to 50-55□C.

6.□. Syringe filter, Millipore Millex-H□ 0.45 um, or equivalent. Do not use PTFE type filters.

6.8. 300 □L autosampler vials, polypropylene, with polypropylene screw caps, Waters P□ 1860004112, or equivalent.

6.□. SPE columns

6.□.1. Waters Oasis WA□ 150 mg/6 cc (P□ 1860024□3) for the cleanup of solids.

6.□.2. Waters Oasis WA□ 500 mg/6 cc (P□ 18600464□) for extraction of PFAS from aqueous sample.

6.10. □raphitized carbon (Envi-Carb™ or equivalent).

6.11. □acuum manifold for Solid Phase Extraction (SPE).

6.12. Miscellaneous laboratory apparatus (beakers, test tubes, volumetric flasks, pipettes, etc.). These should be disposable where possible, or marked and segregated for high-level versus low-level use.

6.13. Water bath: Heated with concentric ring cover capable of temperature control (±5 ℃) up to ±5℃. The bath must be used in a fume hood.

6.14. Plastic tub for an ice bath, AK±O-±.S.T. part ±o. 35-180 or equivalent.

6.15. pH indicator paper, wide range.

6.16. ±ottle rotating apparatus for soil extractions.

6.1±. ±lass fiber filter, Whatman ±F/F, catalog number 1825 0±0 or equivalent.

6.18. Liquid Chromatography/Tandem Mass Spectrometer (LC/MS/MS) – Either of the instruments described below, or equivalent, may be used for this method. ±oth HPLC are equipped with a refrigerated autosampler, an injection valve, and a pump capable of variable flow rate. The use of a column heater is required to maintain a stable temperature throughout the analytical run. Data is processed using Chrom Peak ±eview, version 2.1 or equivalent. The MS/MSD is capable of running in the ±I-ESI mode at the recommended flow rate with a minimum of 10 scans per peak.

6.18.1. [redacted] LC/MS/MS

This system consists of a Shimadzu HPLC interfaced with a [redacted] Triple Quad MS. The instrument control and data acquisition software is SCIE± Analyst, version 1.6.3 or equivalent.

6.18.1.1. [redacted] HPLC equipped with [redacted] pumps and one D±±-20 degassing unit or equivalent.

6.18.1.2. [redacted].

6.18.1.3. PFAS Isolator column, [redacted]. This is plumbed between the ±PLC pumps and autosampler valve to minimize PFAS background from the ±PLC solvent lines and filters.

6.1±. Preventive and routine maintenance is described in the table below

H□□□ □MS□MS □□□□□□□□□□□□M□□□□□□□□□	
A□N□□□□□□	**D□□□□ □□ □□□ □□ □□□□**
Change pump seals. Change in-line filters in autosampler (HPLC). Check/replace in-line frit if excessive pressure or poor performance. □eplace column if no change following in-line frit change. Clean corona needle. □eplace sample inlet tube in APCI (10.1 cm). □eplace fused silica tube in ESI interface. Clean lenses. Clean skimmer. □allast rough pump 30 minutes. Create all eluents in □eagent module, label eluent containers with TALS label and place 2nd label into maintenance log when put into use.	Check solvent reservoirs for sufficient level of solvent. □erify that pump is primed, operating pulse free. Check needle wash reservoir for sufficient solvent. □erify capillary heater temperature functioning. □erify vaporizer heater temperature. □erify rough pump oil levels. □erify turbo-pump functioning. □erify nitrogen pressure for auxiliary and sheath gasses. □erify that corona and multiplier are functioning.
S□□ □□Annually	**Annually**
Replace rough-pump oil (4-6 months). Replace oil mist and odor elements. Replace activated alumina filter if applicable	Vacuum system components including fans and fan covers. Clean/replace fan filters, if applicable.

7. REAGENTS AND STANDARDS

7.1. Reagent grade chemicals shall be used in all tests whenever available. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on the Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1.1. Acetic acid, glacial

7.1.2. [redacted]. The resultant solution is filtered through a 0.22 µm filter before use. This solution has volatile components, thus it should be replaced every 7 days or sooner.

7.1.3. Ammonium hydroxide (NH4OH), 0.3% in methanol: Prepared by diluting

12 mL of ammonium hydroxide into 4L of methanol.

7.1.4. Hexane

7.1.5. Hydrochloric acid (HCl), 2.0 M solution in water

7.1.6. Hydrochloric acid (HCl), concentrated, reagent grade

7.1.7. Methanol

[redacted]

7.1.9. [redacted], reagent grade

7.1.10. Ottawa Sand

7.1.11. Sodium hydroxide (NaOH), 0.1 N, in water: Prepared by diluting 400 mL of 1N NaOH into 3.6L of water for a total volume of 4 L.

7.1.12. Sodium hydroxide (NaOH), 10 N, reagent grade

7.1.13. □ ater, Nanopure or Millipore, must be free of interference and target analytes.

7.2. Standards

7.2.1. P□AS are purchased as high purity solids (96% or greater) or as certified solutions. Standard materials are verified compared to a second source material at the time of initial calibration. The solid stoc□material is stored at room temperature or as specified by the manufacturer or vendor.

7.2.1.1. Per the Certificate of Analysis for labeled perfluorohexadecanoic acid ($13C_2$-P□Hx□A) produced by □ ellington Laboratories, the stoc□standard contains roughly 0.3% of native perfluorohexadecanoic acid. This e□uates to roughly 0.30 ng/L or 0.015 µg/□g of perfluorohexadecanoic acid expected in all samples and blan□s.

7.2.2. As of this writing, only P□OS, P□OA, P□HxS, □t-□OSAA and Me-□OSAA are commercially available as technical mixtures. These reference standards of the technical mixtures for these specific P□AS are used to ensure that all appropriate pea□s are included during pea□integration.

7.2.3. □f solid material is used for preparing a standard, stoc□standard solutions are prepared from the solids and are stored at 0 - 6□C. Stoc□standard solutions

should be brought to room temperature before using. Standards are monitored for signs of degradation or evaporation. Standard solutions must be replaced at least annually from the date of preparation.

7.2.4. P□□S, P□HxS, P□HpS, P□OS, P□□S, and many other P□AS are not available in the acid form, but rather as their corresponding salts, such as sodium or potassium. The standards are prepared and corrected for their salt content according to the e□uation below.

$Mass_{acid}$ □ Measured $Mass_{salt}$ □ M□ $_{acid}$ / M□ $_{salt}$

□ here: M□ $_{acid}$ is the molecular weight of P□AA

M□ $_{salt}$ is the molecular weight of the purchased salt.

7.2.5. □or example, the molecular weight of P□OS is 500.1295 and the molecular weight of NaP□OS is 523.1193. Therefore, the amount of NaP□OS used must be ad□usted by a factor of 0.956.

7.2.6. □ hile P□AS standards commercially purchased are supplied in glass ampoules, all subse□uent transfers or dilutions performed by the analyst must be prepared and stored in polypropylene or H□P□ containers.

7.3. Calibration Standards

The calibration stoc□solution is prepared by diluting the appropriate amounts of stoc□ solutions in □0% methanol/water. The calibration stoc□solution is diluted with methanol to produce initial calibration standards. These are the normal calibration levels used. A different range can be used if needed to achieve lower reporting limits or a higher linear range.

7.4. □nitial Calibration (□CAL) Levels (ng/mL)

□□□ □□un□	□S□□	□S□□	□S□□	□S□□	□S□□	□S□□	□S□7
□□□lu□□al□yl□a□□□yl□□a□□□□□□□A□□							
P□□A	0.025	0.05	0.25	1	2.5	10	20
P□PeA	0.025	0.05	0.25	1	2.5	10	20
P□HxA	0.025	0.05	0.25	1	2.5	10	20
P□HpA	0.025	0.05	0.25	1	2.5	10	20
P□OA	0.025	0.05	0.25	1	2.5	10	20
P□NA	0.025	0.05	0.25	1	2.5	10	20
P□□A	0.025	0.05	0.25	1	2.5	10	20
P□UdA	0.025	0.05	0.25	1	2.5	10	20
P□□oA	0.025	0.05	0.25	1	2.5	10	20
P□Tr□A	0.025	0.05	0.25	1	2.5	10	20
P□Te□A	0.025	0.05	0.25	1	2.5	10	20
P□Hx□A	0.025	0.05	0.25	1	2.5	10	20

□□□ □□un□	□S□□	□S□□	□S□□	□S□□	□S□□	□S□□	□S□7
P□O□A	0.025	0.05	0.25	1	2.5	10	20
□□□lu□□na□□□ □ul□□n□□a□□□□□□SA□□							
P□□S	0.025	0.05	0.25	1	2.5	10	20
P□PeS	0.025	0.05	0.25	1	2.5	10	20
P□HxS□	0.025	0.05	0.25	1	2.5	10	20
P□HpS	0.025	0.05	0.25	1	2.5	10	20
P□OS□	0.025	0.05	0.25	1	2.5	10	20
P□NS	0.025	0.05	0.25	1	2.5	10	20
P□□S	0.025	0.05	0.25	1	2.5	10	20
P□□oS	0.025	0.05	0.25	1	2.5	10	20
□□□lu□□na□□□ □ul□□na□ □□□□□□□SA□							
FOSA	0.025	0.05	0.25	1	2.5	10	20
Et-FOSA	0.025	0.05	0.25	1	2.5	10	20
Me-FOSA	0.025	0.05	0.25	1	2.5	10	20
Perfluorinated sulfonamide ethanols (FOSE)							
Et-FOSE	0.025	0.05	0.25	1	2.5	10	20
Me-FOSE	0.025	0.05	0.25	1	2.5	10	20
Perfluorinated sulfonamidoacetic acids (FOSAA)							
EtFOSAA*	0.025	0.05	0.25	1	2.5	10	20
MeFOSAA*	0.025	0.05	0.25	1	2.5	10	20
Fluorotelomer sulfonates (FTS)							
4:2 FTS	0.025	0.05	0.25	1	2.5	10	20
6:2 FTS	0.025	0.05	0.25	1	2.5	10	20
8:2 FTS	0.025	0.05	0.25	1	2.5	10	20
10:2 FTS	0.025	0.05	0.25	1	2.5	10	20
Labeled Isotope Dilution Analytes (IDA)							
13C4-PFBA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C5-PFPeA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C2-PFHxA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C4-PFHpA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C4-PFOA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C5-PFNA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C2-PFDA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C2-PFUdA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C2-PFDoA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
18O2-PFHxS	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C4-PFOS	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C3-PFBS	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C2-PFTeDA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C2-PFHxDA	2.5	2.5	2.5	2.5	2.5	2.5	2.5

□ompound	□S□	□S□	□S□	□S□	□S□	CS-6	CS-7
13C8-FOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
d5-EtFOSAA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
d3-MeFOSAA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
M2-4:2FTS ǂ	2.5	2.5	2.5	2.5	2.5	2.5	2.5
M2-6:2FTS	2.5	2.5	2.5	2.5	2.5	2.5	2.5
M2-8:2FTS	2.5	2.5	2.5	2.5	2.5	2.5	2.5
d5-EtFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
d3-MeFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
d9-Et-FOSE	2.5	2.5	2.5	2.5	2.5	2.5	2.5
d7-Me-FOSE	2.5	2.5	2.5	2.5	2.5	2.5	2.5
Internal Standard (IS)							
13C2-PFOA	2.5	2.5	2.5	2.5	2.5	2.5	2.5

** Both branched and linear isomers are used.*

ǂ *- This compound is used as a reverse surrogate for the TOP analysis.*

***Note**: Sample extracts are in 80% MeOH/H $_2$O.*

Compound	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6	CS-7
Fluorinated Replacement Chemicals							
HFPO-DA	0.025	0.05	0.25	1.0	2.5	10	20
9CI-PF3ONS (F53B major)	0.025	0.05	0.25	1.0	2.5	10	20
11CI-PF3OUdS (F53B minor)	0.025	0.05	0.25	1.0	2.5	10	20
Dona	0.025	0.05	0.25	1.0	2.5	10	20
Labeled Isotope Dilution Analytes							
13C3-HFPO-DA	0.025	0.05	0.25	1.0	2.5	10	20

***Note**: Sample extracts are in 80% MeOH/H $_2$O.*

***Note**: The above calibration limits are provided only as an example. The actual ICAL level used for each analytical batch will depend upon the LOQ requirements of the program.*

7.4.1. A technical (qualitative) grade PFOA standard which contains both linear and branched isomers is used as a retention time (RT) marker. This is used to integrate the total response for both linear and branched isomers of PFOA in environmental samples while relying on the initial calibration with the linear isomer quantitative standard This technical (qualitative) grade PFOA standard is analyzed initially, after every initial calibration or when significant changes are made to the HPLC parameters.

7.4.1.1. Attach this document to the ICV from the associated ICAL by scanning the document and associating it to the file as a

document type of High Res □ □Tune in TAL□. □se the following naming convention□"_TFOA_Instrument_Date." □□ample□□TFOA□A1□□1□□ ar□□1□.

7.□. Initial Calibration Verification □tandard (ICV)

A second source solution for PFA□ is purchased from the same vendor□the PFC□□ □□ contains most of the target analytes in this mi□ture and is used as an ICV. A few compounds are not available in this mi□ture, may not be available as another lot, and are not available from another vendor. For these analytes only, a second analyst may prepare a second source standard from the same source as the ICAL to produce an ICV. The recommended concentration of the ICV standard should be in the mid□range of the calibration curve. The concentration may be ad□usted if the initial calibration levels are changed or altered. The I□A and I□ are added at a fi□ed concentration of □□ng□mL.

7.□. LC□□□ atri□PFC □pike □olution, □□ng□mL

The PFC spike solution is prepared by diluting all PFA□ to produce a solution containing each PFA□ at a concentration of □□ng□mL in methanol.

7.7. PFC Isotope □ilution Analyte □olution, □□ng□mL

The PFC□I□A solution is prepared by diluting all labeled PFA□ to produce a solution containing each compound at a concentration of □□ ng□mL in methanol. This is added to all samples prior to e□traction.

7.□. Reverse □urrogate □olution, 1□□□ng□mL

The reverse surrogate solution is prepared by diluting □ □4□□FT□ to produce a solution containing this compound at a concentration of 1□□□ng□mL in methanol. This is added to all samples for the TOP assay to monitor the efficiency of the o□idation process.

7.□. Internal □tandard □olution, □□ng□mL

The internal standard solution is prepared by diluting 1□C□□PFOA to produce a solution containing this compound at a concentration of □□ng□mL in methanol. This is added to all e□tracts prior to analysis.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

□.1. □ ater samples are collected in pre□cleaned □□□mL H□P□ containers. □oil samples are collected in pre□cleaned □ oz H□P□ containers. Other containers may also be suitable. □amples are chilled to □□□C for shipment to the laboratory.

□.1.1. □ ater samples collected from a known chlorinated source should be preserved with Trizma.

□.□. □amples are logged in following normal laboratory procedures and are stored under refrigeration at □ □□C. □ ater samples must be e□tracted within 1□days of collection. □oil samples must also be e□tracted within 14 days of collection. Tissue samples must be e□tracted within 1 year of collection if stored at ≤□1 □C. □□tracts must be refrigerated at □ □□C, and analyzed within 4□days from e□traction.

***Note**: As of this writing, Method 537 provides for a 14 day holding time for water samples preserved with Trizma buffer. The scientific literature indicates that perfluorinated substances are highly persistent in the environment. TestAmerica Sacramento has conducted time stability studies that support a 14 day holding time for aqueous samples with and without Trizma preservation. TestAmerica Denver has conducted stability studies indicating that medium- and low-level solutions of PFOA are stable for at least three months in polystyrene and polypropylene plastics at 0-6°C. The 14/40 day holding times given above are based on the stability study and general EPA convention for the holding time of extractable organic compounds in water and soil.*

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability (IDOC)

The initial demonstration and method detection limit (MDL) studies described in Section 13 must be acceptable before analysis of samples may begin.

9.2. 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-003) for further details of the batch definition.

9.2.1. 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 matrix spike/matrix 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, an LCSD may be substituted if batch precision is required by the program or client. 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.

9.3. One method blank (MB, laboratory reagent 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 solid samples, the method blank is an aliquot of Ottawa sand. 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, and then implemented when target analytes are detected in the method blank above the reporting limit or when 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-003 for specific acceptance criteria.

9.3.1. If the MB produces a peak within the retention time window of any of the analytes, determine the source of the contamination and eliminate the interference before processing samples.

9.3.2. 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.

9.3.3. 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.

9.3.4. Re-extraction and reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

9.3.5. Refer to WS-PQA-003 for further details of the corrective actions.

9.3.6. Projects performed under the auspices of the DOD/DOE must meet QSM specific criteria for method blanks. Results are acceptable if the blank contamination is less than ½ of the reporting limit/LOQ 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. Reprepare and reanalyze all field and QC samples associated with the contaminated method blank.

9.4. 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 for aqueous samples and Ottawa sand for solids) 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 of the control limits. 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. The control limits for the LCS are stored in TALS.

9.5. A matrix spike/matrix spike duplicate (MS/MSD or MS/SD) 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 nonconformance memo, and then implemented when recoveries of any spiked analyte are outside of the control limits provided by TALS or by the client.

9.6. A duplicate control sample (LCSD or DCS) may be added when insufficient sample volume is provided to process an MS/MSD pair, or is requested by the client. The LCSD is evaluated in the same manner as the LCS. See WS-PQA-003 for specific acceptance criteria.

9.□. Initial calibration verification (IC□) –A second source standard is analyzed with the initial calibration curve. The concentration should be at the mid range of the curve.

Corrective actions for the IC□ include□

- Rerun the IC□.
- Remake or acquire a new IC□.
- Evaluate the instrument conditions.
- Evaluate the initial calibration standards.
- Rerun the initial calibration.

9.□. Isotope Dilution Analytes

9.□.1. The IDA solution is added to each field and QC sample at the time of extraction, as described in Section 11. As described in Section □, this solution consists of isotopically labeled analogs of the analytes of interest.

9.□.2. IDA recoveries are flagged if they are outside of the acceptance limits (25–150%). Quantitation by isotope dilution generally precludes any adverse effect on data quality due to IDA recoveries being outside of the acceptance limits as long as the signal-to-nose ratio is greater than 10□1.

9.□.2.1. 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.□.2.2. Re-extraction of samples should be performed if the signal-to-noise for any IDA is less than 10□1 or if the IDA recoveries fall below 10%.

9.□.2.2.1. Re-extraction may be necessary under other circumstances when data quality has been determined to be adversely affected.

9.□.2.3. □or samples analyzed in accordance with version 5.1 of the DoD/DOE QSM, the IDA recovery criteria is 50-150%. If QC or field samples do not meet these criteria then re-extraction is required.

9.□.2.4. □or samples analyzed in accordance with version 5.3 of the DOD/DOE QSM, IDA recovery are not calculated. The areas of the IDA must be within 50-150% of the areas in the ICAL, or initial CC□ if an ICAL is not analyzed on the same day

9.□.2.4.1. If out, re-analyze.

9.□.2.4.2. If still out, re-extract the samples (as a greater dilution or smaller sample size may be needed).

9.9. Internal Standard

9.9.1. The Internal Standard (IS) is added to each field and QC samples prior to analysis. The CC□ IS response (peak area) must not deviate by more than 50% from the average response (peak area) of the initial calibration.

9.9.2. Sample IS response (peak area) must be within □50% of the response (peak area) in the most recent CC□.

9.9.3. If the IS does not meet criteria, re-analyze the extract. If the IS meets criteria in the second analysis, report that analysis. If the IS does not meet criteria in the second analysis, report the first analysis with narration.

9.10. TOP Oxidation Efficiency

9.10.1. If the data indicates incomplete oxidation (i.e. the Post-TOP M2-4□2 □TS recovery is greater than 10% or the Post-TOP precursor concentration is greater than 10% of the Pre-TOP concentration) then a second aliquot (10 mL or a 0.2g equivalent) should be processed.

9.10.2. A reduced sample size may be used initially if sample history or other information indicates the sample is grossly contaminated.

9.11. Ion Ratio

9.11.1. Compare the quantifier/qualifier SRM transition ratio in the sample to the SRM transition ratio in the standard.

9.11.2. The quantifier/qualifier SRM ion ratio should be within □50% of the average of the quantifier/qualifier SRM ion ratios calculated from the midlevel ICAL point or from the CC□, if an ICAL is not run.

9.11.3. At this time the ion ratio evaluation is a quantitative identification tool. Analyst judgement should be used if the ratio does not meet criteria. Data should be qualified "I" if the ratio is not met.

9.11.4. □or samples analyzed in accordance with the DoD/DOE QSM version 5.3□if the quantitation ion peak does not meet the maximization criteria the peak shall be included in the summed integration. The result should be flagged "estimated, high bias". As there not a default qualifier for this in the TALS formatter for , use the "see case narrative" flag and NCM the issue.

10. CALIBRATION

10.1. □or 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-P-003 "Calibration Curves and Selection of Calibration Points".

10.2. Routine instrument operating conditions are listed in the table in Section 11.16.

10.3. Instrument Tuning □ Mass Calibration

10.3.1. Mass Calibration is performed by instrument manufacturer service representatives in accordance with the manufacturer's procedures during installation, and annually thereafter.

10.3.2. Instrument tuning is done initially when the method is first developed and thereafter as needed during troubleshooting. Tuning is done by infusing each individual compound (native and IDA) into the mobile phase using a tee fitting at a point just before the entrance to the electrospray probe. The responses for the parent and daughter ions for each compound are observed and optimized for sensitivity and resolution. Mass assignments are reviewed and updated as needed. The mass assignments must be within ± 0.5 amu of the values shown in the table in Section 11.16.

10.3.3. Once the optimal mass assignments (within □0.5 amu of true) are made immediately following the initial tune, the lowest level standard from the initial calibration curve is assessed to ensure that a signal to noise ratio

greater than 10 to 1 (S/N □10□1) is achieved for each P□AS analyte. The first level standard from the initial calibration curve is used to evaluate the tune stability on an ongoing basis. The instrument mass windows are set initially at □0.5 amu of the true value□therefore, continued detection of the analyte transition with S/N □10□1 serves as verification that the assigned mass remains within □0.5 amu of the true value, which meets the DoD/DOE QSM 5.1 tune criterion. □or QSM 5.1 work, the instrument sensitivity check (section 10.12.4) is also evaluated to ensure that the signal to noise criteria is met.

10.3.3.1. □or samples run in accordance with the DoD/DOE QSM version 5.3, the instrument must have a valid mass calibration prior to sample analysis. This is verified through the acquisition of a full scan continuum mass spectrum of a P□AS stock standard. All masses must be verified to be within □0.5 amu of true value.

10.4. 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 pump seals. A new calibration is not required after minor maintenance.

10.5. 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. Average Response □actor and linear fit calibrations require five points, whereas Quadratic (second order) calibrations require six points.

10.6. A fixed injection volume is used for quantitation purposes and is to be the same for both the sample and standards.

10.□. All units used in the calculations must be consistently uniform, such as concentration in ng/mL.

10.□. Initial Calibration

10.□.1. A number of analytical standards of different analyte concentrations are used to generate the curve. Each standard is injected once to obtain the peak response for each analyte at each concentration. These standards define the working range of the analysis.

10.□.1.1. A minimum of five analytical standards is used when using average response factor and/or linear calibration fits.

10.□.1.2. A minimum of six analytical standards is used when a quadratic fit is used to generate the curve.

10.□.2. Calibration is by average response factor, linear fit, or by quadratic fit. Quadratic fit is used for the analyte if the response is non-linear.

10.□.2.1. □or average response factor (R□a), the relative standard deviation (RSD) for all compounds quantitated against an identically labeled analog must be □35% for the curve to be valid.

10.□.2.2. □or average response factor (R□a), the relative standard deviation (RSD) for all compounds quantitated against a closely related labeled analog IDA must be □50% for the curve to be valid.

10.□.2.3. □or linear fit, the intercept of the line must be less than ½ the reporting limit, and the coefficient of determination (r2) must be greater than or equal to 0.990 for the curve to be considered valid (or the correlation coefficient (r) □0.995).

10.□.2.4. The Internal Standard (IS) response (peak area) must not deviate by more than 50% from the average response (peak area) of the initial calibration.

10.□.2.5. Criteria for samples analyzed in accordance with QSM 5.1 or higher□

- The %RSD of the R□S for all analytes must be □20%.
- Linear or non-linear calibrations must have r^2 □0.99 for each analyte.
- Analytes must be within □0-130% of their true value for each calibration standard.

10.9. Calibration Curve □its

10.9.1. Linear regression or quadratic curves may be used to fit the data to a calibration function. Detailed descriptions and formulas for each fitting type can be found in SOP CA-Q-P-003, "Calibration Curves and Selection of Calibration Points".

10.9.2. The linear curve uses the following function□

Equation 1

$$y = bx + c$$

Where:

$$y = \frac{\text{Area (analyte)}}{\text{Area (IS)}} \times \text{Concentration (IS)}$$

$$x = \text{concentration}$$

□ = □lo□e
c = interce□t

□□□□□□he □a□ratic c□r□e □e□the □ollo□in□□nction:

Equation 2

$$y = ax^{\square} + bx + c$$

Where y□x□□an□c are the □a□e a□a□o□e an□a = c□r□at□re□

□□□□□□□al□ation o□Cali□ration C□r□e□

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- □he a□□ol□te □al□e o□the interce□t o□a re□re□□ion line (linear or non□linear) at □ero re□□on□e □□□t□e le□□than the re□ortin□li□it□
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□□□□Initial Cali□ration □lan□(IC□)

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- The instrument plan must be the same

Initial Calibration Verification (ICV)

Following the ICAL and the ICV an ICV standard obtained from a different source or vendor than the ICAL standard is analyzed. This ICV standard is a mid-range standard.

The recovery for the ICV must meet the appropriate following criteria:

The native analyte must be within or equal to for all native analytes quantitated against an identically labeled analog IDA.

The native analyte must be within or equal to for all native analytes quantitated against a closely related labeled analog IDA.

The IDA must be within or equal to

Criteria for labeled analytes in accordance with SOP or higher: Analyte concentrations must be within of their true values for all analytes IDA and targets.

See Section for corrective action in the event that the ICV does not meet the criteria above.

Continuing Calibration Verification (CCV)

Analyze a CCV at the beginning of a run, the end of a run and after every labeled to determine if the calibration is still valid. The exception is after an acceptable CCV and ICV are run, labeled can be analyzed before a CCV is required. The CCV must alternately at the middle level and the lowest calibration standard throughout the run (low level (LOQ)) to middle level. The CCV and ICV do not need to be run every day. To start an analytical run a CCV can be analyzed and if it meet acceptance criteria a run can be started. In addition the low standard in the CCV must be analyzed and must be within of the expected value.

The recovery for the CCV standard must be equal to or within for all native quantitated against an identically labeled analog and equal to or within to for all native quantitated against a closely related labeled analog. The recovery for the IDA must be within or equal to

The Internal Standard (IS) response (peak area) must be within from the response (peak area) from the midpoint of the initial calibration.

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11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of a supervisor to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an Non-Conformance Memo (NCM). The NCM process is described in more detail in SOP WS-QA-0023. The NCM shall be filed in the project file and addressed in the case narrative.

Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

11.2. Water Sample Preparation

11.2.1. Visually inspect samples for the presence of settled and/or suspended sediment/particulates. If present or if the sample is biphasic add IDA prior to any sample decanting or centrifugation. If the sample requires decanting or centrifugation contact the client for guidance prior to such action. Decanting or filtering of the sample can lead to a low bias.

11.2.2. If authorized by the client to filter the sample, filter the water sample through a glass fiber filter (Whatman GF/F Cat No 1825 090 or equivalent). Gravity or vacuum can be used to pass the sample through the filter. Prepare a filtration blank with any samples requiring filtration. File an NCM noting the need for filtration.

Warning: The use of a vacuum system creates the risk of glassware implosion. Inspect all glassware prior to use. Glassware with chips, scratches, rub marks or cracks must not be used.

11.2.3. Weigh the sample container prior to extraction and then weigh the sample

container after extraction to determine the initial volume. Unless otherwise directed by client, use the entire sample volume.

11.2.4. Prepare additional aliquots of a field sample for the MS/MSD, if requested.

11.2.5. Prepare two 250 mL aliquots of LC/MS-grade water for the method blank and LCS.

11.2.6. Spike the LCS and MS/MSD (if requested) with 0.5 mL of the LCS/Matrix PFC Spike solution (Section 7.6).This will result in a sample concentration of 20 ng/L.

11.2.7. Add 0.5 mL of the IDA PFC solution (Section 7.7)into each sample and QC sample, for a fixed concentration of 50 ng/mL in the final sample vial.

11.3. Solid Phase Extraction (SPE) of Aqueous Samples

The automated Zymark Auto-Trace Workstation can be used as long as the program follows these conditions and passes the background check.

11.3.1. Condition the SPE cartridges (Waters WAX, 500 mg/6 cc) by passing the following without drying the column.

Note: *The cartridges should not be allowed to go dry until the final elution step with methanol. At all of the other transition steps, the solvent/sample level should be stopped at the top of the column before the next liquid is added.*

WARNING: The use of a vacuum system creates the risk of glassware implosion. Inspect all glassware prior to use. Glassware with chips, scratches, rub marks or cracks must not be used.

11.3.2. Wash with 5.0 mL [redacted].

11.3.3. Wash with 5.0 mL of 0.1N NaOH/water. Close valve when ~ 200 uL remains on top to keep column wet. After this step, the columns cannot go dry until the completion of loading and rinsing samples.

11.3.4. Appropriately label the columns and add the reservoir to the column.

11.3.5. Add samples to the columns and with vacuum, pull the entire 250 mL aliquot of the sample through the cartridge at a rate of approximately 2 to 5 drops per second.

11.3.5.1. If the SPE column should plug (flow rate <1 drop per minute) prior to the entire content of the sample container passing through the column do the following:

1. Stop adding sample to the reservoir.

2. Return any remaining sample volume back to the original container.
3. Weigh the original container and record this weight into the worksheet notes field within the TALS extraction batch.
4. Determine the full volume of sample fortified by using the "Gross Weight" – (remaining sample volume – default tare weight of a sample container (26.1 g)).
5. Enter this value into the "Initial Amount" field in the TALS extraction batch.
6. Proceed to Section 11.4, noting that additional vacuum or pressure might be needed to elute the SPE column.

11.3.6. After the entire sample has been loaded onto the column, rinse the sample bottle with two 5 mL aliquots of reagent water and pour onto the column reservoir.

11.3.7. After the final loading of the sample but before completely passed through the column, rinse the SPE column with 1 mL of water.

11.3.8. After the sample and water rinse have completely passed through the cartridge, allow the column to dry well with vacuum for 15 minutes.

11.4. SPE Column Wash of Aqueous Samples with Hexane

11.4.1. Load the first 5 mL of hexane to soak for five minutes and then elute to waste.

11.4.2. Load the second 5 mL of hexane and elute to waste (without a soaking period).

11.4.3. Allow the column to dry with vacuum for 5 to 10 minutes. Columns must be dried before continuing.

11.5. SPE Elution of Aqueous Samples – using 15 mL polypropylene test tubes as receiving tubes in the SPE manifold.

11.5.1. Rinse sample bottles with 4 mL [redacted] and transfer to the column reservoir onto the cartridge. Allow the solution to soak for 5 minutes and then elute into the 15 mL collection tube.

11.5.2. Repeat sample bottle to column reservoir rinse and cartridge elution with a second 4 mL aliquot of [redacted]. The total collection should be approximately 8 mL.

11.5.3. Proceed to Section 11.15.2 (Graphitized Carbon Cleanup) as needed. This

required for all DoD/DOE extracts.

11.5.4. Proceed to Section 11.6 for final volume.

11.6. Final volume for extract

11.6.1. Add 0.5 mL of IS 50 ng/mL concentration and 2 mL of water to the extract. This will create an extract with a final solvent composition of 80:20 methanol:water.

11.6.1.1. Seal the test tube tightly. Invert container several times and then vortex. Allow extract to settle for 10 minutes prior to moving to the next step.

11.6.2. Transfer a portion of the extract to a 300 uL polypropylene autosampler vial (7 dropwise or approximately ½ filled is sufficient). Archive the rest of the extracts for re-injection and dilution.

11.6.3. Seal the vial with a polypropylene screw cap. Note: Teflon lined caps cannot be used due to detection of low level concentration of PFAS.

11.7. Soil, Sediment and Tissue Sample Preparation and Extraction

11.7.1. Visually inspect soil samples for homogeneity.

11.7.1.1. Projects performed in accordance with the DOD/DOE QSM, version 5.1 or higher must have the entire sample homogenized prior to subsampling (see SOP WS-LA-0018).

11.7.2. Weigh a representative 5 g aliquot of soil, sediment or 1 g of tissue sample into a 50 mL HDPE wide-mouth bottle. Weigh additional sample amounts for the matrix spike and matrix spike duplicate analyses if they are requested.

11.7.3. For the method blank and LCS matrix, use 5 g each of Ottawa sand or 0.1 g of oil.

11.7.4. Spike the LCS and MS/MSD (if requested) with 1.0 mL of the LCS/Matrix PFAC Spike solution (Section 7.6). This will result in a sample concentration of 4.0 ng/g.

11.7.4.1. Spike non-concentrated samples at 0.5 mL of LCS/Matrix PFAC Spike Solution.

11.7.5. Add 1.0 mL of the ADA PFAC solution (Section 7.7) into each sample and QC sample, for a fixed concentration of 50 ng/mL in the final sample vial.

11.7.5.1. Spike non-concentrated samples at 0.5 mL of DA PC Solution.

11.7.6. Cap the bottles and allow the spike to settle into the sample matrix. Gently shake the bottles to mix the spike into the matrix.

11.7.7. Add 20 mL of [redacted] to each sample.

11.7.8. Shake each sample on an orbital shaker at room temperature for 3 hours.

11.7.9. Following the shaking, extract the samples in an ultrasonic water bath for an additional 12 hours.

11.7.10. After the completion of extraction, centrifuge each sample at 3500 rpm for 15 minutes.

11.7.11. Collect and decant the [redacted] extract to a new 50 mL centrifuge tube.

11.7.12. Add another 2 mL of [redacted] solution to the residue, briefly shake to mix and centrifuge at 3500 rpm for 15 minutes.

11.7.13. Combine the rinsate to the first corresponding tubes.

11.7.14. To the final [redacted] extract, add 2 mL of water to each.

11.7.15. Concentrate the [redacted] extract under nitrogen to less than 2 mL, and dilute with water to 15 mL final volume.

11.7.16. Acidify with 80 µL of glacial acetic acid, and mix the contents well with vortex mixer. Check the pH to ensure pH is between 6 to 8.

11.7.17. Centrifuge at 3500 rpm for 15 minutes.

11.8. Solid Extract Cleanup by SPE

Set up WA 150 mg/6 cc SPE columns for sample cleanup using vacuum manifold.

11.8.1. Condition the SPE cartridges by passing the following without drying the column.

Note: *The cartridges should not be allowed to go dry until the final elution step with methanol. At all of the other transition steps, the solvent/sample level should be stopped at the top of the column before the next liquid is added.*

WARNING: The use of a vacuum system creates the risk of glassware implosion. Inspect all glassware prior to use. Glassware with chips, scratches, rub marks or cracks must not be used.

11.8.2. Wash with 5.0 mL of [redacted].

11.8.3. Wash with 10 mL of 0.1 N NaOH/water. Close valve when ~ 500 µL remains on top of column to keep column wet. *After this step, the columns cannot go dry until the completion of loading and rinsing samples.*

11.8.4. Add extracts to the columns and with vacuum, pull the entire extracts through the cartridge at rate of approximately 3 to 5 drops per second.

11.8.5. Rinse the sample tube with 5 mL of water and add to the SPE column.

11.8.6. Dry the columns with vacuum for 15 minutes.

11.9. SPE Column Wash of Solid Extracts with Hexane

11.9.1. Load the first 5 mL of hexane to soak for five minutes, and elute to waste.

11.9.2. Load the second 5 mL of hexane and elute to waste (without a soaking period).

11.9.3. Allow the column to dry with vacuum for 10 minutes. Columns must be dried before continuing.

11.10. SPE Elution of Solid Extracts – using 15 mL polypropylene test tube as receiving tube in the SPE manifold.

11.10.1. Rinse extraction bottles with 4 mL of [redacted] and transfer to the column reservoir onto the cartridge. Allow the solution to soak for 5 minutes and then elute into the 15 mL collection tube.

11.10.2. Repeat extract bottle to column reservoir rinse and cartridge elution with a second 4 mL aliquot of [redacted]. The total collection should be approximately 8 mL.

11.10.3. Proceed to Section 11.15.2 (Graphitized Carbon Cleanup) as needed. This is required for all DoD/DOE extracts.

11.10.4. Proceed to Section 11.6 for final volume.

11.11. Product/Dispersion Samples

11.11.1. Check the solubility of the material in both methanol and water

11.11.1.1. If the material is soluble in water, dilute 0.5 mL of sample into 250 mL of DI water and proceed to Section 11.3 (follow water

extraction procedures). □ortify sample appropriately with IDA or P□C spike solution, see Section 11.2.

11.11.1.2. If the material is soluble in methanol, dilute 1 g (if solid) or 1 mL (if liquid) of material into 10 mL of methanol (□ eOH).

11.11.1.2.1. If the material does not completely dissolve, contact your immediate supervisor.

11.11.2. Take 100 □L of the 10 mL solution and dilute it to 10 mL in □ eOH.

11.11.3. Take a 1 mL aliquot of this solution (effective dilution of 1000x (1 mg for solid or 0.001 mL for liquid)) and fortify with 0.5 mL of labeled IDA solution (Section □.□).

11.11.4. DO NOT PASS E□TRACT THRO□GH SPE CARTIRIDGE (omit steps 11.9 – 11.11).

11.11.5. Proceed to Section 11.6 of this SOP for extract concentration.

11.12. TOP (Total Oxidizable Precursor) Assay for Aqueous Samples

11.12.1. Prepare 3 □250 mL HDPE containers with HPLC grade water to create the needed □C Samples (□ □, LCS/LCSD).

11.12.2. Prepare enough 125 mL HDPE containers as needed for all "Pre" and "Post" samples, including □C. Label each appropriately.

11.12.3. Spike the "Pre" and "Post" MB 125 mL containers with 25 µL of the reverse surrogate solution of □ 2□4□2 □TS (Section □8).

11.12.4. Spike the "Pre" and "Post" LCS/LCSD 125 mL containers with 0.5 mL of the LCS Spike solution (Section 7.6), both regular and "add□on", and 25 uL of the reverse surrogate solution (Section □8).

11.12.5. Remove the methanol solvent from all Post □C sample 125 mL containers (□ □ and LCS/LCSD) by using N2 evaporation.

11.12.6. Add 2g of [redacted] and 1.9 mL of [redacted] to each "Post" sample container.

11.12.□. Subsample 100 mL aliquots of water from each field sample and □C from the 250 mL containers into each of the corresponding 125 mL containers for both the "Pre" and "Post" samples. Spike all "Pre" and "Post" samples with 25uL of the reverse surrogate solution (Section □.8).

11.12.8. Set aside all "Pre" sample containers.

11.12.9. Cap each "Post" sample container, invert 2–3 times prior to placing container into water bath.

11.12.10. Add 2 g of [redacted] and 1.9 mL of [redacted] to each "Post" sample container.

11.12.11. Heat each "Post" sample container in a water bath (KD) at 85°C for 6 hours.

11.12.12. After digestion for 6 hours, place the "Post" sample containers in an ice bath for 30 minutes.

11.12.13. Adjust the pH of "Post" samples and associated QC aliquots to 7 with concentrated HCl. Use pH paper to determine the pH.

11.12.14. Spike both "Pre" and "Post" samples and their associated QC samples with 0.5 mL of PFC IDA solution (Section 6.1.1), both regular and add-on.

11.12.15. Use the following SPE procedure for both "Pre" and "Post" samples:

11.12.15.1. Set up WAX 150 mg/6 cc SPE columns for sample extraction using a vacuum manifold.

11.12.15.2. Establish a sample loading flow rate of 3–5 drops per second for each port of the vacuum manifold, for as many ports as will be used simultaneously during sample loading.

11.12.15.3. Wash/condition the SPE column with 5 mL of [redacted], then 5 mL water.

11.12.15.4. Load 100 mL of sample onto the SPE cartridge at a flow rate of 3–5 drops per second.

11.12.15.5. Add 5 mL rinse water

11.12.15.6. After the sample and water rinse have completely passed through the column, allow it to dry well using vacuum with a flow rate of 1 mL/minute for 15 minutes.

11.12.15.7. Wash the SPE column with 10 mL hexane rinse eluting all to waste.

11.12.15.8. Allow the column to dry well using vacuum for 5 minutes. Columns must be dry before continuing.

11.12.15.9. Elute the samples into 15 mL polypropylene test tubes in the SPE manifold by rinsing each 125 mL sample container with 5

mL of [redacted], and add to the SPE cartridge as eluent.

11.12.15.10. Repeat with another 5 mL of [redacted].

11.12.15.11. Collect the 10 mL of eluent and concentrate per Section 11.6.

11.13. TOP (Total Oxidizable Precursor) Assay for Soil Samples

11.13.1. Weigh representative 2 g aliquots of soil for each "Pre" and "Post" sample into a 50 mL centrifuge tube.

11.13.2. For the method blank and LCS matrix, use 2 g each of Ottawa sand for each "Pre" and "Post" QC sample.

11.13.3. Add 20 mL of [redacted] to each sample.

11.13.4. Shake each sample on an orbital shaker at room temperature for 3 hours.

11.13.5. Following the shaking, extract the samples in an ultrasonic water bath for an additional 12 hours.

11.13.6. After the completion of extraction, centrifuge each sample at 3500 rpm for 15 minutes.

11.13.7. Collect and decant the [redacted] extract to a new 50 mL centrifuge tube.

11.13.8. Add another 2 mL of [redacted] solution to the residue, briefly shake to mix and centrifuge at 3500 rpm for 15 minutes.

11.13.9. Combine the rinsate to the first corresponding tubes.

11.13.10. Proceed to Section 11.16.2 (Envi-carb clean up)

11.13.11. To the final [redacted] extract, add 0.5 mL of water to each.

11.13.12. Concentrate the [redacted] extract under nitrogen to less than 0.25 mL.

11.13.13. Dilute extract up to 50 mL with water in the centrifuge tube and vortex.

11.13.14. Prepare enough 125 mL HDPE containers as needed for all "Pre" and "Post" samples, including LC. Label each appropriately.

11.13.15. Spike the "Pre" and "Post" MB 125 mL containers with 25 µL of the reverse

surrogate solution of □ 2□4□2 □TS (Section □8).

11.13.16. Spike the "Pre" and "Post" LCS/LCSD 125 mL containers with 0.5 mL of the LCS Spike solution and 25 □L of the reverse surrogate solution (Section □.8).

11.13.1□. Remove the methanol solvent from all "Post" QC sample 125 mL containers (□ □ and LCS/LCSD) by using N2 evaporation.

11.13.18. Add 2 g of [redacted] and 1.9 mL of [redacted] to each "Post" sample container.

11.13.19. Transfer extract from the centrifuge tube to the appropriate 125 mL container.

11.13.20. Rinse the centrifuge container with an additional 50 mL of water and transfer to the appropriate 125 mL container.

11.13.21. Set aside all "Pre" sample containers.

11.13.22. Cap each "Post" sample container, invert 2□3 times prior to placing container into water bath.

11.13.23. Heat each "Post" sample container in a water bath (KD) at 85□C for 6 hours.

11.13.24. After digestion for 6 hours, place the "Post" sample containers in an ice bath for 30 minutes.

11.13.25. Adjust the pH of "Post" samples and associated QC aliquots to 7 with concentrated HCl. □se pH paper to determine the pH.

11.13.26. Spike both "Pre" and "Post" samples and their associated QC samples with 0.5 mL of P□C IDA solution (Section □.□).

11.13.2□. Use the following SPE procedure for both "Pre" and "Post" samples:

11.13.2□.1. Set up WA□ 150 mg/6 cc SPE columns for sample extraction using a vacuum manifold.

11.13.2□.2. Establish a sample loading flow rate of 3□5 drops per second for each port of the vacuum manifold, for as many ports as will be used simultaneously during sample loading.

11.13.2□.3. Wash/condition the SPE column with 5 mL of [redacted], then 5 mL water.

11.13.2□.4. Load 100 mL of sample onto the SPE cartridge at a flow rate of 3□5 drops per second.

11.13.2□.5. Add 5 mL rinse water

11.13.2□.6. After the sample and water rinse have completely passed through the column, allow it to dry well using vacuum with a flow rate of 1 mL/minute for 15 minutes.

11.13.2□.□Wash the SPE column with 10 mL hexane rinse eluting all to waste.

11.13.2□.8. Allow the column to dry well using vacuum for 5 minutes. Columns must be dry before continuing.

11.13.2□.9.Elute the samples into 15 mL polypropylene test tubes in the SPE manifold by rinsing each 125 mL sample container with 4 mL of [redacted], and add to the SPE cartridge as eluent.

11.13.2□.10. Repeat with another 4 mL of [redacted].

11.13.2□.11. Collect the 8 mL of eluent and bring to final volume per Section 11.6.

11.14. Other Types of Sample Cleanup

11.14.1. □reezing technique to remove lipids.

If samples contain lipids then freeze the methanolic extract and □C extracts at □20°C for at least 1 hour. Collect the solvent layer.

11.14.2. Cleanup with graphitized carbon will be applied to all samples as needed but is required for all DoD/DOE extracts.

11.14.2.1. Add 100 mg of graphitized carbon to each sample extract and □C extracts.

11.14.2.2. Shake vigorously and then let sit for 10 minutes.

11.14.2.3. Centrifuge each sample for 2 minutes at 1000 rpm.

11.14.2.4. Decant the solvent layer.

11.14.2.5. Proceed to Section 11.6.

11.15. A□□□ Sample Preparation

11.15.1. □C for A□□□ samples consists of a method blank, a laboratory control

sample and a sample or matrix duplicate only. No matrix spike or matrix spike duplicate is needed.

11.15.2. Perform a 1,000,000 □ serial dilution of the A□□□ sample. Dilute 1 mL of A□□□ sample to 1 L with laboratory supplied water. Then dilute 1mL of this dilution to 1 L with laboratory supplied water.

11.15.2.1. □e sure to retain all dilutions should the initial analysis warrant re□analysis at higher concentration.

11.15.3. Subsample 2.0 mL of this dilution and fortify with 0.5 mL IDA solution and 0.5 mL of IS (50 ng/mL) solution□then add □0 mL of methanol.

11.15.4. Transfer a portion of the sample to a 300 □L polypropylene autosampler vial (□ dr□p□wise or approximately □ filled is sufficient). Archive the rest of the sample for re□in□ection or dilution.

11.16. Instrument Analysis

Suggested operating conditions are listed in Tables 1□4 for the SCIE□ LC□ S systems□

□□□□ □ □□ □□□□ □ □□□□ □□□□□□ □□□□ □□□□□□ □ □□□□□□				
HPLC Conditions (***)***				
Column (Column temp = °C)				
Mobile Phase Composition	A = B =			
Gradient Program	**Time**	**%A**	**%B**	**Flow Rate - mL/min**
	Maximum pressure limit = 5,000 psi			
Injection Size	(fixed amount throughout the sequence).			
Run Time	~			
Mass Spectrometer Interface Settings (***)***				
MS Interface Mode	ESI Negative Ion. Minimum of 10 scans/peak.			
Ion Spray Voltage (kV)				
Entrance Potential (V)				
Declustering Potential (V)				
Desolvation Temp				
Curtain Gas				
Collision Gas				

Table 2 - Recommended Instrument Operating Conditions								
Mass Spectrometer Scan Settings ( )								
Compound	Comments	Reaction (MRM)	Dwell (sec)	Ent. Pot. (V)	Col. Energy (V)	Declu. Pot. (V)	Cell Exit Pot. (V)	Typ RT (Min)
PFBA	Native analyte	212.9 > 169	0.011					
13C4-PFBA	IDA	217 > 172	0.011					
PFBS	Native analyte	298.9 > 80	0.011					
PFBS_2	Native analyte	298.9 > 99	0.011					
13C3-PFBS	IDA	301.9 > 83	0.011					
PFPeA	Native analyte	262.9 > 219	0.011					
13C5-PFPeA	IDA	267.9 > 223	0.011					
4:2 FTS	Native analyte	327 > 307	0.011					
M2-4:2FTS	IDA or Reverse Surrogate for TOP	329 > 81	0.011					
PFHxA	Native analyte	313 > 269	0.011					
PFHxA_2	Native analyte	313 > 119	0.011					
13C2-PFHxA	IDA	315 > 270	0.011					
PFHpA	Native analyte	363 > 319	0.011					
PFHpA_2	Native analyte	363 > 169	0.011					
13C4-PFHpA	IDA	367 > 322	0.011					
PFPeS	Native analyte	349 > 80	0.011					
PFPeS_2	Native analyte	349 > 99	0.011					
PFHxS	Native analyte	399 > 80	0.011					
PFHxS_2	Native analyte	399 > 99	0.011					
18O2-PFHxS	IDA	403 > 84	0.011					
6:2 FTS	Native analyte	427 > 407	0.011					
M2-6:2FTS	IDA	429 > 81	0.011					
PFOA	Native analyte	413 > 369	0.011					
PFOA_2	Native analyte	413 > 169	0.011					
13C4-PFOA	IDA	417 > 372	0.011					
13C2-PFOA	IS	415 > 370	0.011					
PFHpS	Native analyte	449 > 80	0.011					
PFHpS_2	Native analyte	449 > 99	0.011					
PFNA	Native analyte	463 > 419	0.011					
PFNA_2	Native analyte	463 > 169	0.011					
13C5-PFNA	IDA	468 > 423	0.011					
PFOS	Native analyte	499 > 80	0.011					
PFOS_2	Native analyte	499 > 99	0.011					
PFNS	Native analyte	549 > 80	0.011					
PFNS_2	Native analyte	549 > 99	0.011					
PFDoS	Native analyte	699 > 80	0.011					
PFDoS_2	Native analyte	699 >99	0.011					
13C4-PFOS	IDA	503 > 80	0.011					

Table 2 - Recommended Instrument Operating Conditions								
Mass Spectrometer Scan Settings ( )								
Compound	Comments	Reaction (MRM)	Dwell (sec)	Ent. Pot. (V)	Col. Energy (V)	Declu. Pot. (V)	Cell Exit Pot. (V)	Typ RT (Min)
PFDA	Native analyte	513 > 469	0.011					
PFDA_2	Native analyte	513 > 169	0.011					
13C2-PFDA	IDA	515 > 470	0.011					
8:2 FTS	Native analyte	527 > 507	0.011					
10:2 FTS	Native analyte	627 > 607	0.011					
M2-8:2FTS	IDA	529 > 81	0.011					
PFOSA	Native analyte	498 > 78	0.011					
13C8-PFOSA	IDA	506 > 78	0.011					
N-MeFOSAA	Native analyte	570 > 419	0.011					
d3-MeFOSAA	IDA	573 > 419	0.011					
PFDS	Native analyte	599 > 80	0.011					
PFDS_2	Native analyte	599 > 99	0.011					
PFUdA	Native analyte	563 > 519	0.011					
PFUdA_2	Native analyte	563 > 169	0.011					
13C2-PFUdA	IDA	565 > 520	0.011					
N-EtFOSAA	Native analyte	584 > 419	0.011					
d5-EtFOSAA	IDA	589 > 419	0.011					
PFDoA	Native analyte	613 > 569	0.011					
PFDoA_2	Native analyte	613 > 169	0.011					
13C2-PFDoA	IDA	615 > 570	0.011					
PFTrDA	Native analyte	663 > 619	0.011					
PFTrDA_2	Native analyte	663 > 169	0.011					
PFTeDA	Native analyte	713 > 169	0.011					
PFTeDA_2	Native analyte	713 > 219	0.011					
13C2-PFTeDA	IDA	715 > 670	0.011					
Et-FOSA	Native analyte	526 > 169	0.011					
d5-EtFOSA	IDA	531 > 169	0.011					
Me-FOSA	Native analyte	512 > 169	0.011					
d3-MeFOSA	IDA	515 > 169	0.011					
Et-FOSE	Native analyte	630 > 59	0.011					
d9-EtFOSE	IDA	639 > 59	0.011					
Me-FOSE	Native analyte	616 > 59	0.011					
d7-MeFOSE	IDA	623 > 59	0.011					
PFHxDA	Native analyte	813 > 769	0.011					
PFHxDA_2	Native analyte	813 > 169	0.011					
13C2-PFHxDA	IDA	815 > 770	0.011					
PFODA	Native analyte	913 > 869	0.011					
PFODA_2	Native analyte	913 > 169	0.011					

Table 3 - Recommended Instrument Operating Conditions Mass Spectrometer Scan Settings (SCIEX 5500) for Fluorinated Replacement Chemicals								
Compound	Comments	Reaction (MRM)	Dwell (sec)	Ent. Pot. (V)	Col. Energy (V)	Declu. Pot. (V)	Cell Exit Pot. (V)	Typ RT (Min)
HFPO-DA	Native analyte	329.1 > 285	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
13C3-HFPO-DA	IDA	332.1 > 287	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
9Cl-PF3ONS (F53B major)	Native analyte	531 > 351	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
11Cl-PF3OUdS (F53B minor)	Native analyte	631 > 451	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
Dona	Native analyte	377 > 251	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
Dona_2	Native analyte	377 > 85	0.011	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]

Table 4 - Retention Times & Quantitation (SCIEX 5500)				
Native Compounds	**Typical Native RT (minutes)**	**IDA analog**	**Typical IDA RT (minutes)**	**Quantitation Method**
PFBA	[redacted]	13C4-PFBA	[redacted]	Isotope Dilution
PFPeA	[redacted]	13C5-PFPeA	[redacted]	Isotope Dilution
PFBS	[redacted]	13C3-PFBS	[redacted]	Isotope Dilution
PFHxA	[redacted]	13C2-PFHxA	[redacted]	Isotope Dilution
PFPeS	[redacted]	13C3-PFBS	[redacted]	Isotope Dilution
PFHpA	[redacted]	13C4-PFHpA	[redacted]	Isotope Dilution
PFHxS	[redacted]	18O2-PFHxS	[redacted]	Isotope Dilution
PFOA	[redacted]	13C4-PFOA	[redacted]	Isotope Dilution
PFHpS	[redacted]	13C4-PFOS	[redacted]	Isotope Dilution
PFNA	[redacted]	13C5-PFNA	[redacted]	Isotope Dilution
PFOS	[redacted]	13C4-PFOS	[redacted]	Isotope Dilution
PFNS	[redacted]	13C4-PFOS	[redacted]	Isotope Dilution
PFDA	[redacted]	13C2-PFDA	[redacted]	Isotope Dilution
FOSA	[redacted]	13C8-FOSA	[redacted]	Isotope Dilution
PFDS	[redacted]	13C4-PFOS	[redacted]	Isotope Dilution
PFUdA	[redacted]	13C2-PFUdA	[redacted]	Isotope Dilution
PFDoA	[redacted]	13C2-PFDoA	[redacted]	Isotope Dilution
PFTrDA	[redacted]	13C2-PFDoA	[redacted]	Isotope Dilution
PFDoS	[redacted]	13C4-PFOS	[redacted]	Isotope Dilution
PFTeDA	[redacted]	13C2-PFTeDA	[redacted]	Isotope Dilution
EtFOSA	[redacted]	d5-EtFOSA	[redacted]	Isotope Dilution
MeFOSA	[redacted]	d3-MeFOSA	[redacted]	Isotope Dilution
EtFOSE	[redacted]	d9-EtFOSE	[redacted]	Isotope Dilution
MeFOSE	[redacted]	d7-MeFOSE	[redacted]	Isotope Dilution
PFHxDA	[redacted]	13C2-PFHxDA	[redacted]	Isotope Dilution

Ta□le □- Retention Times & Quantitation (SCIEX 5500)				
Native Compounds	**Typical Native RT (minutes)**	**IDA analog**	**Typical IDA RT (minutes)**	**Quantitation Method**
PFODA	[redacted]	13C2-PFHxDA	[redacted]	Isotope Dilution
EtFOSAA	[redacted]	d5-EtFOSAA	[redacted]	Isotope Dilution
MeFOSAA	[redacted]	d3-MeFOSAA	[redacted]	Isotope Dilution
4□2 FTS	[redacted]	M2-4□2 FTS □I□ TOP then 13C-PFBS)	[redacted]	Isotope Dilution
6:2FTS	[redacted]	M2-6:2FTS	[redacted]	Isotope Dilution
8:2FTS	[redacted]	M2-8:2FTS	[redacted]	Isotope Dilution
HFPO-DA	[redacted]	13C3-HFPO-DA	[redacted]	Isotope Dilution
9Cl-PF3ONS (F53B major)	[redacted]	13C4-PFOS	[redacted]	Isotope Dilution
11Cl-PF3OUdS (F53B minor)	[redacted]	13C4-PFOS	[redacted]	Isotope Dilution
Dona	[redacted]	13C4-PFOS	[redacted]	Isotope Dilution
10:2 FTS	[redacted]	M2-8:2 FTS	[redacted]	Isotope Dilution

11.16.1. Post Spike Sample Analysis for AFFF samples

11.16.1.1. This section only applies to aqueous samples prepared by serial dilution instead of SPE that have reported value of <LOQ (RL) for any analyte.

11.16.1.2. Spike aliquots of the sample at the final dilution reported for the sample with all analytes that have reported of <LOQ in the final dilution. The spike must be at the LOQ concentration to be reported with the sample (the < LOQ value).

11.16.1.3. When analyte concentrations are calculated as <LOQ, the spike must recover within 70-130% of its true value.

11.16.1.4. It the recovery does not meet this criteria, the sample, sample duplicate and post spike sample must be reanalyzed at consecutively higher dilutions until the criteria is met.

11.16.2. Tune and calibrate the instrument as described in Section 10.

11.16.3. A typical run sequence is as follows:

- Rinse Blank (RB, not linked to anything)
- Start ICAL with CCVL but called IC in TALS (starts the 12 hour clock or time 0:00)
- Rest of ICAL

- ICB: link to midpoint of ICAL and samples
- ICV: link to midpoint of ICAL and samples (If ICAL good)
- CCB: link to midpoint of ICAL and samples
- PFOA RT marker
- Rinse Blank (RB, not linked to anything)
- 10 samples: link to midpoint of ICAL
- CCV: link to midpoint of ICAL
- 10 more samples: link to midpoint of ICAL
- CCV: link to midpoint of ICAL
- Etc.
- CCVL (within 12 hours from CCVL in ICAL, can be the ending CCV and starts 12 hours all over again): if this occurs link to the midpoint of the ICAL toggle it as opening closing CCV.
- CCV: link to midpoint of ICAL
- 10 samples: link to midpoint of ICAL
- CCV: link to midpoint of ICAL
- If no ICAL run that day
- CCB: link to CCVIS
- CCVL (starts 12 hour clock): link to CCVIS
- CCVIS: link to midpoint of ICAL
- 10 samples: link to CCVIS
- CCV: link to CCVIS
- 10 samples: link to CCVIS
- CCV: link to CCVIS
- Etc.
- If going over 12 hours in the sequence: CCVL (within 12 hours from CCVL at item 2 above, can be the ending CCV and starts 12 hours all over again): if this occurs link to the CCVIS toggle as opening and closing CCV.
- CCV: link to CCVIS
- 10 samples: link to CCVIS
- CCV: link to CCVIS

12. CALCULATIONS

12.1. If the concentration of the analyte ions e ceeds the working range as defined by the

calibration standards, then the sample must be diluted and reanalyzed. It may be necessary to dilute samples due to matri□.

12.2. E□tracts can be diluted up to 100□ without diluting out the IDA and thus preserving quantitation via isotope dilution. Dilutions greater than 100□ can be performed but additional IDA must be added. The quantitation will now be via internal standard as a result. Consult the client for authorization of such a dilution.

12.3. Results less than the reporting limit are flagged in the client report as estimated. Generally, the "J" flag is used to denote ≥ MDL and ≤ RL, but the specific flag may change based on client requirements.

12.4. Qualitative Identification

12.4.1. The retention times of PFAS with labeled standards should be the same as that of the labeled IDA's to within 0.05 min. For PFAS with no labeled standards, the RT must be within ± 0.3 minutes of the ICV and CCV standards.

***Note**: The IDA RT and native RT may be offset by 0.02 to 0.04 minutes.*

12.4.1.1. Criteria for samples analyzed in accordance with QSM 5.3: The peak RT must be within 0.4 mins of the CCV or midpoint of the ICAL. The target analyte must elute within 0.1 mins of the IDA for those analytes with their own IDA.

12.5. The ICAL established in Section 10 is used to calculate concentrations for the extracts.

12.6. Extract concentrations are calculated as below. The first equation applies to the linear fit, the second to the quadratic line fit.

Equation 3

$$\text{Concentration, ng/mL} = \frac{y - c}{b}$$

Equation 4

$$\text{Concentration, ng/mL} = \frac{-b + \sqrt{b^2 - 4a(c - y)}}{2a}$$

Where:

y = $\frac{\text{Area (analyte)}}{\text{Area (IS)}} \times \text{Concentration (IS)}$
x = concentration
a = curvature
b = slope
c = intercept

12.7. Water Sample Result Calculation:

Equation 5

$$\text{Concentration, ng/L} = \frac{C_{ex} V_t}{V_o}$$

Where:

C_{ex} = Concentration measured in sample extract (ng/mL)
V_t = Volume of total extract (mL)
V_o = Volume of water extracted (L)

12.8. Soil Sample Result Calculation:

Equation 6

$$\text{Concentration, } ng/g = \frac{C_{ex} V_t}{W_s D}$$

Where ng/g = µg/kg and:

C_{ex} = Concentration measured in sample extract (ng/mL)
V_t = Volume of total extract (mL)
W_s = Weight of sample extracted (g)
D = Fraction of dry solids, which is calculated as follows:

$$\frac{100 - \%\,\text{moisture in sample}}{100}$$ (for dry weight result)

12.9. IDA Recovery Calculation:

Equation 7

$$\%\,\text{Recovery} = \frac{A_t Q_{is}}{A_{is} Q_t RRF_{IDA}} X100$$

Where ng/g = µg/kg and:

RF_{IDA} = Response Factor for IDA compound
A_t = Area response for IDA compound
A_{IS} = Area Response for IS compound
Q_{IS} = Amount of IS added
Q_t = Amount of IDA added

12.10. Raw data, calibration summaries, QC data, and sample results are reviewed by the analyst. These must also be reviewed thoroughly by a second qualified person. See the Data Review Policy (WS-PQA-0012). These reviews are documented on the Data Review Checklist.

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.□. □ethod □etection □imit

The laboratory must generate a □alid method detection limit for each analyte of interest. The □ □□ must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is gi□en in □□ □□□ □art 13□□□ppendix □□and further defined in □□□□ □□□□□□□□□and policy □ □□□□□□□□3 □ □□s are a□ailable in the □uality □ssurance □epartment.

13.3. □nitial □emonstration of □apability □□□□□□

□ach analyst performing this procedure must successfully analy□e four □□□ □□ samples using current laboratory □□□ control limits. □□□□s are appro□ed by the □uality □ssurance □anager and the Technical □irector. □□□□ records are maintained by the □□ staff in the central training files.

13.□. The laboratory must generate a □alid method detection limit for each analyte of interest. The □ □□ must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is gi□en in □□ □□□ □art 13□□□ppendix □□and further defined in □ □□□□□□□□□□nd policy □ □□□□□□□□3.

□□□PO□□□T□ON PRE□ENT□ON

1□.1. □ll waste will be disposed of in accordance with □ederal□□tate and □ocal regulations.

1□.□. □olid phase extraction used for water samples greatly reduces the amount of sol□ent used compared to liquid□liquid extraction.

1□.3. □tandards and reagents are purchased and prepared in □olumes consistent with laboratory use to minimi□e the □olume of expired standards and reagents requiring disposal.

1□.□. □ here reasonably feasible□technological changes ha□e been implemented to minimi□e the potential for pollution of the en□ironment. □mployees will abide by this method and the policies in □ection 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

1□.□. □o not allow waste sol□ent to □ent into the hoods. □ll sol□ent waste is stored in capped containers unless waste is being transferred.

1□.□. Transfer waste sol□ent from collection cups □tri□pour and similar containers□to □ugs and/or carboys as quic□ly as possible to minimi□e e□aporation.

□□□□ A□TE MANA□EMENT

The following waste streams are produced when this method is carried out□

1□.1. □ssorted test tubes□auto□ials□syringes□filter discs and cartridges. □ump the solid waste into a yellow contaminated lab trash buc□et. □ hen the buc□et is full or after no more than one year□tie the plastic bag liner shut and put the lab trash into the ha□ardous waste – landfill steel collection drum in the □3 closet. □ hen the drum is full or after no more than □□days□mo□e it to the waste collection area for shipment.

1□.□. □xtracted soil samples□used sodium sulfate□paper funnel filters□glass wool□thimbles□ and extracted solids saturated with sol□ents. □ump these materials into an orange contaminated lab trash buc□et. □ hen the buc□et is full or after no more than one year□ tie the plastic bag liner shut and put the lab trash into the incineration steel collection drum in the □3 closet. □ hen the drum is full or after no more than □□days□mo□e it to the waste collection area for shipment.

1□.3. □ aste □ ethanol. □ollect the waste sol□ents in tripours during use. □mpty the tripours into a 1□liter to □□liter carboy at the fume hood. □ hen the carboy is full□or at the end of your shift□whiche□er comes first□empty the carboy into the steel flammable sol□ent drum in the □3 closet. □ hen the drum is full to between four and six inches of the top□ or after no more than □□days□mo□e the steel flammable sol□ent drum to the waste collection area for shipment.

1□.□. □ ixed water/methanol waste from soil extraction. □ollect the waste in the □□□□ waste carboy. □ hen full□or after no more than one year□dump into the blue plastic □□□□ collection drum in the □3 closet. □ hen the drum is full to between four and six inches of the top or after no more than □□days□ m□eit to the waste collection area for shipment.

1□.□. □queous acidic waste from the □□□ □instrument contaminated with methanol. This is collected in a 1□gallon carboy at the instrument. □ hen the carboy is full□or after no more than one year□it is emptied into the blue plastic □□□□ collection drum in the □3 closet. □ hen the drum is full to between four and six inches of the top or after no more than □□days□ m□eit to the waste collection area for shipment.

1□.□. □uto□ials contaminated with methanol. □s the auto□ials are remo□ed from the instrument after analysis□they are collected in open containers at the instrument. □fter all auto□ials are remo□ed□the open container must be dumped into a closed satellite collection container in a fume hood□as the punctured septa in the auto□ial can allow methanol and other contaminants to e□aporate into the atmosphere. The satellite collection containers are transferred to the waste disposal area when full or after no more than one year□ where they are disposed through the □ial eater.

□□□REFERENCE□

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1□.□. □T□ □en□er □ hite □aper □□□□□ □□□□003, "Addendum A to Method Validation Study for □nalysis of □mmonium □erfluorooctanate in □oil □ atrices by □igh □erformance □iquid Chromatography/Mass Spectrometry (HPLC/MS/MS)", Mark Dymerski, □ugust □□□□□3.

1□.□. □T□ □en□er □ hite □aper □□□□□ □□□□004, "Method Validation Study for Analysis of □erfluorooctanoic □cid in □ aters by □igh □erformance □iquid □hromatography/Tandem □ ass □pectrometry (HPLC/MS/MS)", Mark Dymerski, □anuary □□□□□□.

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1□.1□.□.□. □epartment of □enfense □□o□□/□epartment of □nergy □□□□□□onsolidated □uality □ystems □ anual □□□□ □for □n□ironmental □aboratories□□ersion □.1.1□dated □□1.□

1□.11. □.□. □epartment of □enfense □□o□□/□epartment of □nergy □□□□□□onsolidated □uality □ystems □ anual □□□□ □for □n□ironmental □aboratories□□ersion □.3 dated □□1.□

1□□ METHOD MOD□F□CAT□ON□

1□.1. □ odifications from □ ethod □3□ are detailed below□

1□.1.1. Target analyte results are quantitated □ia isotope dilution.

1□.1.□. Two ion transitions □precursor to quant ion and precursor to confirmation ion□ are monitored for those analytes that ha□e two transitions. □on ratios are monitored as well for these analytes.

1□.1.3. □ ater sample containers are not preser□ed with Tri□ma.

1□.1.□. The method has been modified to address soil/solid matrices. The extraction holding time is set at 1□ days.

1□.1.□. The analyte list has been expanded. The number of labeled analytes has been expanded as well to impro□e quantitation.

1□.1.□. The reporting limits differ as they are all set at one consistent □alue.

1□.1.□. □alibration le□els differ from the referenced method.

1□.1.□. □ ore labeled analytes are fortified into the samples prior to the extraction process. □ ost target analytes are quantitated against a labeled analyte.

1□.1.□. There is no symmetry requirement.

1□.1.1□. □alibration□both initial and continuing□has different acceptance criteria due to the longer list of analytes□and the use of isotope dilution quantitation.

1□.1.11. The eluents and □□□□ configuration differs. □s a result the final extract is in □□□□□ methanol□water.

1□.1.1□. The □□□ and □ □/□ □□ are spi□ed at one concentration and do not rotate between a low to high le□els.

1□.1.13. □amples are not chec□ed for residual chlorine or p□.

1□.1.1□. □ different □□□ cartridge □□ aters □□□□□□ □ □□□is used for the extraction process. □s a result sol□ents and elution procedures are different.

1□□ ATTACHMENT□

1□.1. □ttachment 1 – □nalysis of □erfluorinated □ompounds □□□□ □□ in □ ater □ia □n □ine □olid □hase □xtraction □□□□□.

RE□□□ON H□□TOR□

□e□isions to □ttachment 1 are documented in the attachment.

□e□isions prior to □□/□1/□□1□ ha□e been remo□ed and are a□ailable in pre□ious □ersions of this □□□.

1□.1. □ □□□□□□□□ e□ision 3.□□□ffecti□e □□/□3/□□1□

1□.1.1. Removed Section 6.9.1, "[redacted]"

1□.1.□. Removed Section 6.9.4, "[redacted]"

1□.1.3. Removed Section 6.9.5, "[redacted]"

1□.1.□. Removed Section 6.9.6, "[redacted]"

1□.1.□. Revised Section 9.8.2.3, "For samples analyzed in in accordance with □ersion □.1 of the □□□/□□□ □□□ the □□ reco□ery criteria is □□1□□□. □f □□ or field samples do not meet these criteria then re□extraction is required"

1□.1.□. Added Section 9.8.2.4, "For samples analyzed in accordance with version □.3 ofthe □□□/□□□ □□□ □□□ reco□ery are not calculated. The areas of the □□ must be within □□1□□□ of the areas in the □□□ or initial □□□ if an □□□ is not analyzed on the same day"

1□.1.□. □dded □ection □11.□, "□or samples analy□ed in accordance with the □o□/□□□ □□□ □ersion □3□if the quantitation ion pea□does not meet the maximi□ation criteria the pea□shall be included in the summed integration. The result should be flagged "estimated, high bias". As there not a default qualifier for this in the TALS formatter for , use the "see case narrative" flag and □□□ the issue."

1□.1.□. □dded □ection 1□3.1□"□ ass □alibration is performed by instrument manufacturer ser□ice representatives in accordance with the manufacturer's procedures during installation, and annually thereafter."

1□.1.□. Section 10.3.2 revised, "to maintain the sensitivity and selectivity f the method" to "during troubleshooting" and "calibrated if necessary" to "updated as needed".

1□.1.1□. Added Section 10.3.3.1, "For samples run in accordance with the □o□/□□□

□□□ □ersion □.3 □the instrument must ha□e a □alid mass calibration prior to sample analysis. This is □erified through the acquisition of a full scan continuum mass spectrum of a □□□□ stoc□ standard. □ll masses must be verified to be within ± 0.5 amu of true value."

1□.1.11. Revised Section 10.8.2.5 to, "Criteria for samples analyzed in accordance with □□□ □.1 or higher□"

1□.1.1□. Revised Section 10.10.4 to, "Criteria for samples analyzed in accordance with □□□ □.1 or higher□"

1□.1.13. Section 10.11.3 revised, "Criteria for samples analyzed in accordance with □□□ □.1 or higher□"

1□.1.1□. Section 10.12.4 revised to, "Criteria for samples analyzed in accordance with QSM 5.1 or higher:"

1□.1.1□. □ection 11.□.1.1 re□ised to, "□ro□ects performed in accordance with the □□□/□□□ □□□ □□ersion □.1 or higher must ha□e the entire sample homogeni□ed prior to subsampling □see □□□□ □□□□□□ □1□."□

1□.1.1□. Added Section 12.4.1.1, "□riteria for samples analy□ed in accordance with □□□ □.3□The pea□□T must be within □.□mins of the □□□ or midpoint of the □□□□. The target analyte must elute within □.1 mins of the □□□ for those analytes with their own □□□."

1□.1.1□. Added Section 6.10, "□.□. □epartment of □enfense □□o□/□epartment of □nergy □□□□□□onsolidated □uality □ystems □anual □□□□ □for □n□ironmental □aboratories□□ersion □.1.1□dated □□1□."

1□.1.1□. Added Section 6.11, "□.□. □epartment of □enfense □□o□/□epartment of □nergy □□□□□□onsolidated □uality □ystems □anual □□□□ □for □n□ironmental □aboratories□□ersion □.3 dated □□1□."

1□.1.1□. □ditorial changes

1□.□. □ □□□□□□□□□e□ision 3.□□ffecti□e □□/13/□□1□

1□.□.1. □dded □t/□e □□□□ and □t/□e□□□□ and related labeled analogs to all calibration and instrument specification tables.

1□.□.□. □emo□ed all references to □aters □□□ □systems.

1□.□.3. □emo□ed all references and procedures for concentrating extracts.

1□.□.□. □emo□ed all references and requirements for the state of □ew □ersey as there is now a separate □□□.

1□.□.□. Added Sections 12.2, "□xtracts can be diluted up to 1□□□ without diluting out the □□□ and thus preser□ing quantitation □ia isotope dilution. □ilutions greater than 1□□□ can be performed but additional □□□ must be added. The quantitation will now be □ia internal standard as a result. □onsult the client for authori□ation of such a dilution."

1□.□.□. □dded □ection 12.3, "□esults less than the reporting limit are flagged in the client report as estimated. Generally, the "J" flag is used to denote ≥ MDL and ≤ RL, but the specific flag may change based on client requirements."

1□.□.□.Added Section 17.1.1, "Target analyte results are quantitated □ia isotope dilution."

1□.□.□. □dded □ection 17.1.2, "Two ion transitions □precursor to quant ion and precursor to confirmation ion□are monitored for those analytes that ha□e two transitions. □on ratios are monitored as well for these analytes."

1□.□.□. □ditorial changes.

1□.3. □ □□□□□□□□□□e□ision 3.□□□ffecti□e □□/1□/□□1□

1□.3.1. □ection 1.1 updated □□□ numbers for "□erfluoro□1□pentanesulfonic acid" and "□erfluoro□nonanesulfonic acid".

1□.3.□. □ection 1.□updated table with correct compound names□abbre□iations□and □□□ numbers.

1□.3.3. Section 1.2 added note, "[1□]□n some literature□the acronym □□□□□ refers to the ammonium salt□□□□ □□□□□□□□□□□□ □nd □□□□ refers to the parent acid. □n □ ethod □3□1□□□□□□ refers to the parent acid. □□□□ is the acronym present on the laboratory raw data."

1□.3.□. Section 7.4 added "*" to "EtFOSAA" and "MeFOSAA" to indicate that both linear and branched isomers are used.

1□.3.□. Section 7.4.1.1 revised last sentence to, "□se the following naming con□ention□"_TFOA_Instrument_Date." Example: _TFOA_A10_15Mar2019."

1□.3.□. Sections 15.3, 15.4, and 15.5 revised, "When full to no less than six inches of the top" to "When the drum is full to between four and six inches of the top".

1□.3.□. □ditorial changes.

1□.□. □ □□□□□□□□□ □□□□ e□ision 3.□□□ffecti□e □□/□□/□□1□

1□.□.1. Added Section 11.3.6, "□fter the entire sample has been loaded onto the column□rinse the sample bottle with two □m□ aliquots of reagent water and pour onto the column reser□oir."

1□.□.□. □ditorial changes.

1□.□. □ □□□□□□□□□ □□□□ e□ision 3.□□□ffecti□e □□/13/□□1□

1□.□.1. Section 6.4 added, "The a□erage weight of the □□□□ bottles with □□□□ screw caps are calibrated once a year. The calibration is performed by weighing 1□bottles with caps and di□iding by 1□to get the a□erage weight. The a□erage weight is used in section □11.3.□1.d□."

1□.□.□. Section 7.4.1 revised, "an" to "every" and removed "or when a new column is installed".

1□.□.3. Add Section 7.4.1.1, "□ttach this document to the □□□ from the associated □□□□ by scanning the document and associating it to the file as a document type of □igh □es □ □Tune in T□□□. □se the following naming con□ention□ "□□batchnumberT□□□□".".

1□.□.□. □dded Section 8.2.1, "□ro□ects performed for the state of □ew □ersey ha□e an analytical holding time □□ days from the extraction date."

1□.□.□. Added Section 8.2.2, "□or pro□ects performed for the state of □ew □ersey a field reagent blan□ □□□□□□must be collected with each sample set. □cceptance limits are □□□ for each analyte."

1□.□.□. Added Section 9.4.1, "□ro□ects performed for the state of □ew □ersey□□□□ □mid and high spi□e□reco□ery limits are □□□13□□. □ow le□el □□□ reco□ery limits are □□□1□□□. The spi□e le□el must rotate between low□medium and high."

1□.□.□. Added Section 9.5.1, "□ro□ects performed for the state of □ew □ersey□ □ □/□ □□ □mid and high spi□e□reco□ery limits are □□□13□□. □ow le□el □ □/□ □□ reco□ery limits are □□□1□□□. The spi□e le□el must rotate between low□medium and high."

1□.□.□. Added Section 9.10, "TOP Oxidation Efficiency" and its associated subsections.

1□.□.□. Added Section 9.11, “Ion Ratio” and associated subsections.

1□.□.1□. Added Section 10.8.2.6, “□ro□ects performed for the state of □ew □ersey□ □ □/□ □□ □mid and high spi□e □reco□ery limits are □□□13□□. □ow le□el □ □/□ □□ reco□ery limits are □□□1□ □□. The spi□e le□el must rotate between low□medium and high.”

1□.□.11. Section 10.11.3 added, “and the state of New Jersey”.

1□.□.1□. Added Section 10.12.5, “□ro□ects performed for the state of □ew □ersey□□ll analyte concentrations in the □□□ must be within □3□□ of their true □alue. □ll analyte concentrations in the low le□el □□□ must be within □ □□□ of their true □alue.”

1□.□.13. Added Section 11.3.5.1, “□f the □□□ column should plug □flow rate □1 drop per minute□prior to the entire content of the sample container passing through the column do the following□” and its associated subsections.

1□.□.1□. Sections 11.14.15.8 and 11.15.27.8 removed, “with a flow rate of 1 mL/minute”.

1□.□.1□. Section 11.18.3 removed, “(as needed)” from the □□□□ □T mar□er.

1□.□.1□. Throughout SOP revised, “1 mL/minute” to “3□5 drops per second”.

1□.□.1□. □ditorial changes.

1□.□. □ □□□□□□□□ □□□□e□isi□n 3.3□ □ffecti□e 1□/□3/□□1□

1□.□.1. Added Section 6.9, “[redacted]”

1□.□.□. Tables □and □re□ised comment for □ □4:2 FTS to, “IDA or Reverse Surrogate for TOP”.

1□.□.3. Tables 4 and 7 revised header from “IS Analog” to “IDA Analog”, and revised “4:2 FTS” entry to “□ □□□□□□T□ □□if T□□then 13□□□□□□□”.

1□.□.□. □ditorial changes.

1□.□. □ □□□□□□□□ □□□□e□isi□n 3.□□□ffecti□e □□/□□/□□1□

1□.□.1. Section 1 added, “1□□1□□□□□□□perfluorododecane sulfonate” and “Perfluoro□1□dodecansulfonic acid” entries to table.

1□.□.□. Section 1.2 revised table entry for “Adona” to “Dona”.

1□.□.3. Section 7.4 added, “PFDoS” and “10:2 FTS” entries to table.

1□.□.□. □ection □.□ re□ised, “Adona” entry to “Dona”.

1□.□.□. Table □ added, “PFDoS”, “PFDoS_2”, and “10:2 FTS” entries to table.

1□.□.□. Table 3 revised, “Adona” and “Adona_2” entries to “Dona” and “Dona_2”.

1□.□.□. Table □ added, “PFDoS” and “10:2 FTS” entries to table.

1□.□.□. Table 4 revised, “Adona entry to “Dona”.

1□.□.□. □ditorial changes.

1□.□. □ □□□□□□□□□ □□e□ision 3.1□□ffecti□e □□/□1/□□1□

1□.□.1. Section 11.2.1 revised to, “□isually inspect samples for the presence of settled and/or suspended sediment/particulates. □f present or if the sample is biphasic add □□□ prior to any sample decanting or centrifugation. □f the sample requires decanting or centrifugation contact the client for guidance prior to such action. □ecanting or filtering of the sample can lead to a low bias.”

1□.□.□. □ditorial changes.

1□.□. □ □□□□□□□□□ □□e□ision 3.□□□ffecti□e □□/13/□□1□

1□.□.1. □ection 1.1 updated table with □□□e□ and □□□□ analytes.

1□.□.□. □dded □ection □.□□ which details the analytes that can be co□ered by the method under special request.

1□.□.3. Added Section 3.13, “□□□□□ □queous □ilm □orming □oam”.

1□.□.□. Section 6.19 added, “Create all eluents in □eagent module□label eluent containers with T□□□ label and place □nd label into maintenance log when put into use” to table.

1□.□.□. Section 7.1.2 added, “[redacted] The resultant solution is filtered through a □.□□um filter before use. This solution has □olatile components□thus it should be replaced every 7 days or sooner.”

1□.□.□. Section 7.1.3 added, “[redacted]”

1□.□.□. □ection □1.□added, "[redacted]."

1□.□.□. □ection □1.11 added, "Prepared by diluting 400mL of 1N NaOH into 3.6L of water for a total volume of 4L."

1□.□.□. □ection □.□updated table with □□□e□and □□□□analytes.

1□.□.1□. □ection □.□□added table to detail □□□□ for □luorinated □eplacement □ompounds.

1□.□.11. Added Section 8.1.1, "Water samples collected from a known chlorinated source should be preserved with Trizma."

1□.□.1□. Added Section 9.9.3, "If the IS does not meet criteria, re□analy□e the extract. □f the □□ meets criteria in the second analysis □report that analysis. □f the □□ does not meet criteria in the second analysis □report the first analysis with narration."

1□.□.13. Added Section 11.14.6, "Add 2g of [redacted] and 1.□m□ of [redacted] to each "Post" sample container."

1□.□.1□. Removed Section 11.14.8, "Add 2g of [redacted] and 1.□m□ of [redacted] to each "Post" sample container."

1□.□.1□. Added Section 11.14.9, "Cap each "Post" sample container, invert 2□3 times prior to placing container into water bath."

1□.□.1□. Added Section 11.5 and associated subsections, which detail the "TOPS □Total □xidi□able □recursor□Assay for Soil Sample".

1□.□.1□. □ection 11.□ updated Table labeling□added □□□e□and □□□□analytes throughout Tables where applicable□and updated Table □to reflect current retention times and quantitation.

1□.□.1□. Section 11.8 added Table 6, "Recommended Instrument Operating □onditions □ ass □pectrometer □can □ettings □[redacted]□ for □luorinated □eplacement □hemicals"

1□.□.1□. □ection 11.1□3 remo□ed outdated run sequence and replaced with current run sequence.

1□.□.□□.□ditorial changes.

1□.1□.□ □□□□□□□□□□ e□ision □.□□□ffecti□e 11/□□/□□1□

1□.1□.1. □ection 1.□□table updated to reflect ranges after remo□ing □ e□□□□ and □t□□□□ from the □□□ in the pre□ious re□ision.

1□.1□.□. □ection □3.□□last sentence changed to read, "Reprepare and reanalyze all field and QC samples associated with the contaminated method blank."

1□.1□.3. Section 9.7, first sentence changed to read, "□nitial calibration □erification □□□□□– □ second source standard is analy□ed with the initial calibration cur□e.

1□.1□.□. Section 1.3.1 revised to read, "□nce the optimal mass assignments □within □□.□amu of true□are made immediately following the initial tune□the lowest le□el standard from the initial calibration cur□e is assessed to ensure that a signal to noise ratio greater than 1□to 1 □□/□ □1□□1□is achie□ed for each □□□□analyte. The first le□el standard from the initial calibration cur□e is used to e□aluate the tune stability on an ongoing basis. The instrument mass windows are set initially at □□.□amu of the true □alue□therefore□continued detection of the analyte transition with □/□ □1□□1 ser□es as □erification that the assigned mass remains within □□.□amu of the true □alue□which meets the □o□/□□□ □□□ tune criterion. □or □□□ wor□ □he instrument sensiti□ity chec□ □section 1□.1□.□□is also e□aluated to ensure that the signal to noise criteria is met."

1□.1□.□. □ditorial changes.

1□.11. □ □□□□□□□□□□ e□ision □.□□□ffecti□e 11/□□/□□1□

1□.11.1. Revised Section 4.5 to "□oth branched and linear □□□□isomers can potentially be found in the en□ironment. □inear and branched isomers are □nown to exist for □□□□□□□□□□□□x□□□□□□□□t□□□□□□and □ e□□□□□ based upon the literature. □f multiple isomers are present for one of these □□□□they might be ad□acent pea□s that completely resol□ed or not□but usually with a deflection point resol□ed during pea□integration. The later of these pea□s match the retention time of its labeled linear analog. □n general□ earlier pea□s are the branched isomers and are not the result of pea□ splitting.

□t this time only □□□□□□□□□ and □□□x□are commercially a□ailable as technical mixtures. These reference standards of the technical mixtures for these specific □□□□are used to ensure that all appropriate pea□s are included during pea□integration."

1□.11.□. □ections □.□and □.□.1.1□or□rected the in□sample contributions to □3□ng/□

and □.□1 □ug/□g.

1□.11.3. Removed Section 7.1.14, "Methanol□□ ater□□□□□□ □ol./□ol. □prepared by mixing □□□m□ methanol and □□□m□ reagent water. □tored in polypropylene bottle and sealed with polypropylene screw cap." Reagent was added incorrectly.

1□.11.□. □ection □.□.□□corrected the factor to □.□□□from 1.□□□.

1□.11.□. Added Section 7.4.1, "□ technical □qualitati□e□grade □□□□ standard which contains both linear and branched isomers is used as a retention time □□T□ mar□er. This is used to integrate the total response for both linear and branched isomers of □□□□ in en□ironmental samples while relying on the initial calibration with the linear isomer quantitati□e standard This technical □qua□itati□e□grade □□□□ standard is analy□ed initially□after an initial calibration when a new column is installed or when significant changes are made to the HPLC parameters."

1□.11.□. Section 9.7, added "Rerun the initial calibration" as the last bullet item.

1□.11.□. □dded □ection 10.3.1, "The first le□el standard from the initial calibration cur□e is used to e□aluate the tune criteria. The instrument mass windows are set at □□.□amu□therefore□detection of the analyte ser□es as □erification that the assigned mass is within □□.□amu of the true □alue□which meets the □o□/□□□□□□□ tune criterion.

1□.11.□. Section 10.10.1, appended "containing both IDA and IS" to the end of the paragraph.

1□.11.□. Sections 11.6.3 and 11.12.2.3, changed "78:22 methanol:water" to "methanol".

1□.11.1□.□ections 1.1 and □.□□remo□ed □t□□□□ and □e□□□□ from tables due to low □olume of requests for those analytes.

1□.11.11. Removed Section 2.2.1, "Optional cleanups may include sample freezing and/or cleanup by □□□ cartridge□unless □t□□□□ and □e□□□□ are requested."

1□.11.1□.□emo□ed □t□□□□/□e□□□□ specific comments in □arious sections throughout the document.

1□.11.13. Section 7.4 Note added, "The concentration of the calibration solutions for non□concentrated extracts is 1/□□th the levels indicated above."

1□.11.1□. □ection □.□□changed 1□□□ng/m□ to □□□ng/m□ and replaced final sentence with “The internal standard solution used for the non□concentrated extracts is at a concentration of 50 ng/mL.”

1□.11.1□. Removed Section 11.2.8, “□f □t□□ □□ and/or □ e□□ □□ are requested□add 1□□□u of □□ and then ad□ust the final □olume □□□□of these aliquots to □.□ m□ with □ e□□. □□ samples□□□□□□ □□and □ □□ will require concentration □ia nitrogen to ad□ust the □□ to □.□m□. □ortex each sample. Then□transfer a portion of the extract to a 3□□u□ polypropylene autosampler □ial □□drop□wise or approximately □ filled is sufficient□ □rchi□e the rest of the extracts for re□in□ection and dilution.”

1□.11.1□. Added Section 11.5.4, “□roceed to □ection 11.1□.□□□□raphiti□ed □arbon □leanup□as needed. This is required for all □o□/□□□ extracts.”

1□.11.1□. Added Section 11.7.1.1, “Seal the test tube tightly. □n□ert container se□eral times and then □ortex. □llow extract to settle for 1□minutes prior to mo□ing to the next step.”

1□.11.1□. Inserted Section 11.8.1.1, “Projects performed under the auspices of the □o□/□□□ must ha□e the entire sample homogeni□ed prior to subsampling in accordance with □□□ □.1 criteria.”

1□.11.1□. Section 11.11.4, added “□□raphiti□ed □arbon □leanup□as needed. This is required for all □o□/□□□ extracts.”

1□.11.□□ Section 11.14.6, added “Spike all “Pre” and “Post” samples with 25uL of the re□erse surrogate solution □□ection □.□).”

1□.11.□1. Section 11.15.2, revised to read, “□leanup with graphiti□ed carbon will be applied to all samples as needed but is required for all □o□/□□□ extracts.”

1□.11.□□ Added Section 11.15.2.5, “Proceed to □ection 11.□□11.□□or 11.1□as applicable.”

1□.11.□3. □emo□ed □ections 11.1□3 through 11.1□.□.

1□.11.□□ Added Section 11.16, “AFFF Sample Preparation”.

1□.11.□□. ection 11.1□□ emo□ed □t□□□□□ e□□□□d□□□t□□□□and d3□ e□□□□ from all tables.

1□.11.□□. ection 11.1□□ hanged masses for □ □□□□□T□□□ □□□□□T□□and □ □□ □□□T□. □nitially assigned daughter masses were bleeding through from the nati□e analog.

1□.11.□□.ection 11.1□□ ll tables on □ □ nterface □ ode □ine □added "Minimum of 10 scans/peak."

1□.11.□□Added Section 11.17.1, "Post Spike Sample Analysis for AFFF Samples".

1□.11.□□.dded □ection 11.8.4.1 "Spike non□concentrated samples at □.□m□ of LCS/Matrix Spike Solution."

1□.11.3□.Added Section 11.8.5.1, "Spike non□concentrated samples at □.□m□ of □□ PFC Solution."

1□.11.31. □ditorial changes.

1□.1□.□ □□□□□□□□□e□ision □.□□□ffecti□e □□/□/□1□

1□.1□.1. □ection 1.1 table□added 1□1□□□□□□□perfluorohexane sulfonate *(4:2)*.

19.12.2. Section 1.1, removed "Sample results for PFOA may also be reported as APFO, at the request of the client. (See Section 12.7)."

19.12.3. Section 1.2 and 11.8.2, updated tissue extracted mass and RL.

19.12.4. Section 2.5, removed "and assumes a proportional relationship between the initial calibration and the analyte in the extract. The ratio of the peak response to mass or concentration injected is used to prepare a calibration curve."

19.12.5. Added Section 6.6, "Extract concentrator or nitrogen manifold with water bath heating to 50-55°C".

19.12.6. Added Section 7.1.14, "Methanol-Water, 78:22 vol./vol., prepared by mixing 780 mL methanol and 220 mL reagent water. Stored in polypropylene bottle and sealed with polypropylene screw cap."

19.12.7. Section 7.2.1.1, revised "roughly 0.15 pg/L" to "roughly 0.15 ng/L".

19.12.8. Section 7.4 table, added:

4:2 FTS	0.5	1.0	2.0	20	50	200	400

19.12.9. Section 7.4 table, revised Labeled Isotope Dilution Analytes (IDA) Section.

19.12.10. Section 7.4 table, added:

Internal Standard (IS)							
13C2-PFOA	50	50	50	50	50	50	50

19.12.11. Section 7.4, removed “FOSAA may be added to the mix and are added at the same concentration as FOSA.”

19.12.12. Added Section 7.9, “Internal Standard Solution, 1000 ng/mL. The internal standard solution is prepared by diluting 13□2-PFOA to produce a solution containing this compound at a concentration of 1000 ng/mL in methanol. This is added to all extracts prior to analysis. □on-concentrated extracts are fortified with a 5X dilution of this solution.”

19.12.13. Section 8.1, changed “250 mL” to “8 oz.”

19.12.14. Added Sections 9.3.6, 9.8.2.3, 10.10.4, 10.8.2.5, 10.11.3, and 10.12.4 to address DOD □S□ 5.1 Table □-15 criteria.

19.12.15. Added Section 9.9, “Internal Standard.”

19.12.16. □pdated all tables to indicate target analyte quantitation via isotope dilution. Internal standard quantitation is only used to quantitate the IDA recoveries.

19.12.17. Added Section 10.8.2.4, 10.12.2, and 10.12.2.1 to incorporate IS criteria into calibrations.

19.12.18. Section 11.2.1, “Evaluate if the sample can be decanted or centrifuged; if not, contact the client for guidance. Filtering the sample can lead to a low bias.”

19.12.19. Added Section 11.2.3.1, “Alternatively, weigh the sample container prior to extraction and then weigh the sample container after extraction to determine the initial volume.”

19.12.20. Added Section 11.5.3, “Note: If the extracts will not be concentrated elute extract with a total of 8 mL of [redacted].”

19.12.21. Added Section 11.6.2.3, “Add 300 uL of the 78:22 methanol:water solution and mix the contents well using a vortex mixer.”

19.12.22. Added Section 11.6.2.4, “Add 100 uL of Internal Standard (IS) solution to each extract and vortex to mix.”

19.12.23. Added Section 11.7, “Final volume for non-concentrated extract”.

19.12.24. Revised Section 11.11, “SP□ □lution of Solid Extracts”.

19.12.25. Revised Section 11.12, “Extract Concentration for Solid Samples”.

19.12.26. Removed Section 12.8, “If results are to be reported as ammonium

perfluorooctanoate (APFO), instead of PFOA, apply a multiplier of 1.0406 to the sample results to correct for the molecular weight differences between PFOA and APFO or this adjustment can be made during the preparation of the standards used for calibration. (Use one, not both.)"

19.12.27. Removed Section 13.4 – it was a copy of Section 13.2.

19.12.28. □arious revisions to fulfill requirements based on DOD/DO□ □S□ 5.1.

19.12.29. □ditorial changes.

19.13. WS-L□-0025, Revision 2.6, □ffective 08/15/2017

19.13.1. Section 7.4, added □ PF□S, □ PFTeDA, and □ PF□xDA to the table.

19.13.2. Section 11.15, added 13□-PF□S to the Recommended Instrument Operating □onditions table for [redacted].

19.13.3. Section 11.15 Recommended Instrument Operating □onditions table, changed the mass transitions for native PFTeDA from 713 □669 (quant) and 713 □169 (qualifier) to 713 □169 (quant) and 713 □219 (qualifier).

19.13.4. □ditorial changes.

19.14. WS-L□-0025, Revision 2.5, □ffective 07/10/2017

19.14.1. Revised Section 11.6.1 to read "Prior to concentrating each sample, add 100 uL of water."

19.14.2. Revised Section 11.6.2 to read "Concentrate each sample under a gentle stream of nitrogen until the methanol is evaporated and the 100 uL of water remains.

11.6.2.1 This blow down must take a minimum of 3.5 hours.

11.6.2.2 □xtracts can not remain in the water bath longer than 5 minutes once concentrated."

19.14.3. Revised Section 11.6.3 to read "Add 400 uL of methanol to each extract, soak, and vortex to mix well. This will create an extract with a final solvent composition of 80:20 methanol:water."

19.14.4. Revised Section 11.11.1 to read "Prior to concentrating each sample, add 200 uL of water."

19.14.5. Revised Section 11.11.2 to read "Concentrate each sample under a gentle

stream of nitrogen until the methanol is evaporated and the 200 uL of water remains."

11.11.2.1 This blow down must take a minimum of 3.5 hours.

11.11.2.2 □xtracts can not remain in the water bath longer than 5 minutes once concentrated."

19.14.6. Revised Section 11.11.3 to read "Add 800 uL of methanol to each extract, soak, and vortex to mix well. This will create an extract with a final solvent composition of 80:20 methanol:water."

1. SCOPE AND APPLICATION

1.1. This procedure describes the analysis of water samples via in line solid phase extraction (SP□) for the following compounds using liquid chromatography / tandem mass spectrometry (L□/□ S/□ S) on a [redacted].

Compound Name	Abbreviation	CAS #
Perfluoroalkylcarboxylic acids (PFCAs)		
Perfluoro-n-heptanoic acid	PF□pA	375-85-9
Perfluoro-n-octanoic acid	PFOA	335-67-1
Perfluoro-n-nonanoic acid	PF□A	375-95-1
Perfluorinated sulfonic acids (PFSAs)		
Perfluoro-1-butanesulfonic acid	PF□S	375-73-5
Perfluoro-1-hexanesulfonic acid	PF□xS	355-46-4
Perfluoro-1-octanesulfonic acid	PFOS	1763-23-1

1.2. The working range of the method is listed below. The linear range can be extended by diluting the extracts.

Matrix	Nominal Sample Size	Reporting Limit	Working Range
Water	1.0 mL	2.0 ng/L	2 to 200 ng/L

2. SUMMARY OF METHOD

2.1. A 1 mL aliquot of sample is diluted to a 40:60 methanol:water extract and analy□ed by L□/□ S/□ S. PFAS are separated from other components on a □18 column with a solvent gradient program using [redacted].

3. DEFINITIONS

Refer to Section 3 of the main body of this SOP for a summary of definitions.

4. INTERFERENCES

Refer to Section 4 of the main body of this SOP for interferences.

5. SAFETY

Refer to Section 5 of the main body of this SOP for safety information.

6. EQUIPMENT AND SUPPLIES

Refer to Section 6 of the main body of this SOP for supplies, other than those listed below specific to the in line SPE analysis.

6.□. □m□ auto sampler □ials, clear □lass, □hermo Scientific □ation surestop □ial, part no. □5□□□□□, ore□ui□alent.

6.□. □ial caps, □hermo Scientific □ational □□□S blue cap, pre slit □E□S□□ septa, part no. □5□□□55□ or e□ui□alent.

6.3. Eppendorf □□□□u□ ep□□PS, part no. □□□4□□□54 or e□ui□alent.

6.4. Eppendorf □□□u□ ep□□PS, part no. □□□4□□□3□or e□ui□alent.

6.5. 5□ m□ □raduated plastic centrifu□e tubes, S□P Science □i□i□□□ES part no. □□□5□□□ □63 ore□ui□alent.

6.6. □□□u□ Pipette□Eppendorf Research Plus.

6.□. □□□u□ Pipette□Rainin E□P3□Plus.

6.□. □5□ m□ □□PE bottles with PPE screw caps, ESS part no. □5□□□□□□□ or e□ui□alent.

6.□. □nalytical columns

6.□.□. [redacted] or e□ui□alent.

6.□.□. P□□S □solator column, [redacted] or e□ui□alent.

6.□□. [redacted]. □he system utili□es □hrom Pea□Re□iew, □ersion □.□or e□ui□alent.

6.□□. [redacted] □P□□ e□uipped with [redacted] pumps and one [redacted] de□assin□unit or e□ui□alent.

□. REA□ENTS AND STANDARDS

Refer to Section □of the main body of this SOP for rea□ents and standards, other than those listed below specific to the in line SPE analysis.

□.□. Rea□ent □rade chemicals shall be used in all tests whene□er a□ailable. □nless otherwise indicated, it is intended that all rea□ents shall conform to the specifications of the □ommittee on the □nalytical Rea□ents of the □merican □hemical Society, where such specifications are a□ailable. Other □rades may be used, pro□ided it is first ascertained that the rea□ent is of sufficiently hi□h purity to permit its use without lessenin□the accuracy of the determination.

□.□.□. □mmonium acetate, □isher Optima □□□ S □rade □□□m□ in water□, part no. □□□4□5□, ore□ui□alent.

□.□.□. □ ethanol, □a□er □P□□ □rade, part no. □□□3□□3.

□.□.3. □ ater, □anopure or □ illipore or □isher Optima □□□ S □rade, part no. □ 6□4, must be free of interference and tar□et analytes.

□.□. □alibration Standards

□he calibration stoc□solution is prepared by dilutin□the appropriate amounts of the stoc□solutions □Section □.□of the main body of this SOP□in 4□6□ m□ethanol□water. □he calibration stoc□solution is diluted with methanol to produce initial calibration standards. □hese are the normal calibration le□els used. □ different ran□e can be used if needed to achie□e lower reportin□limits or a hi□her linear ran□e.

□.3. □nitial □alibration □□□□□□□e□els □n□□□□

C□□ □□□□□	CS□□	CS□□	CS□□	CS□4	CS□5	CS□6	CS□□	CS□□
P□□□□□□□□□□□□□□□□□□□ □□□□□PFCA□□								
P□□p□	□.□	□.□	5.□	□□	□□	5□	□□□	□□□
P□O□	□.□	□.□	5.□	□□	□□	5□	□□□	□□□
P□□□	□.□	□.□	5.□	□□	□□	5□	□□□	□□□
P□□□□□□□□□□□ □□□□□□□ □□□□PFSA□□								
P□□S	□.□	□.□	5.□	□□	□□	5□	□□□	□□□
P□□□S	□.□	□.□	5.□	□□	□□	5□	□□□	□□□
P□OS	□.□	□.□	5.□	□□	□□	5□	□□□	□□□
L□□□□□I□□□□□D□□□□□A□□□□□IDA□								
□3□4□P□□p□	5□	5□	5□	5□	5□	5□	5□	5□
□3□4□P□O□	5□	5□	5□	5□	5□	5□	5□	5□
□3□5□P□□□	5□	5□	5□	5□	5□	5□	5□	5□
□□O□P□□□S	5□	5□	5□	5□	5□	5□	5□	5□
□3□4□P□OS	5□	5□	5□	5□	5□	5□	5□	5□
□3□3□P□□S	5□	5□	5□	5□	5□	5□	5□	5□

Note: *The above calibration levels are provided only as an example. The actual ICAL level used for each analytical batch will depend upon the LOQ requirements of the program.*

7.4. LCS/Matrix PFC Spike Solution, 100 ng/mL.

The PFC spike solution is prepared by diluting all PFAS to produce a solution containing each PFAS at 100 ng/mL in methanol.

7.5. PFC Isotope Dilution Analyte (IDA) Spike Solution, 1 ng/mL.

The PFC IDA solution is prepared by diluting all labeled PFAS to produce a solution containing each at 1 ng/mL in methanol.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Water samples are collected in pre-cleaned 250 mL HDPE containers. Other containers may also be suitable. Samples are chilled to 0 - 6 °C for shipment to the laboratory.

8.2. Samples are logged in following normal laboratory procedures and are stored under refrigeration at 0 - 6 °C. Water samples must be analyzed within 28 days of collection.

9. QUALITY CONTROL

Refer to Section 9 of the main body of this SOP for Quality Control information.

9.1. If potable water samples from the state of New York (NY) are analyzed via this method the control limits for LCS and IDA for PFOS and PFOA recoveries are 70-130%. If these limits are not met, refer to Section 9 of the main body of this SOP for corrective action.

9.2. If POST (treatment) samples have positive detections, review the associated PRE and MID (treatment) samples for similar detections. Re-preparation and re-analysis may be needed.

9.3. If PFBS is detected in the method blank greater than the RL, evaluate data for impact. PFBS is a known laboratory artifact. Re-preparation and re-analysis may be needed.

10. CALIBRATION

Refer to Section 10 of the main body of the SOP for calibration information.

11. PROCEDURE

Refer to Section 11 of the main body of this SOP for procedures, other than those listed below specific to the in line SPE analysis.

11.1. Water Sample Preparation

11.1.1. Visually inspect samples for the presence of settled and or suspended sediment/particulate. Evaluate if the sample can be decanted or centrifuged; if not, contact the client for guidance. Filtering the sample can lead to a low bias.

If authorized by the client to filter the sample, filter the water sample through a glass fiber filter (Whatman GF/F Cat No 1825 090 or equivalent).

Gravity of vacuum can be used to pass the sample through the filter. Prepare a filtration blank with any samples requiring filtration. File an NCM noting the need for filtration.

[illegible]

11.1.2. Prepare an LCS and method blank by adding 250 mL of HPLC grade water into a 250 mL HDPE bottle.

11.1.3. If requested, find the client assigned sample for MS/MSD.

11.1.4. Spike directly into the sample bottles for the LCS and MS/MSD (if requested) with 0.050 mL (50 uL) of the LCS/Matrix PFC Spike solution (Section 7.4). This will result in a sample concentration of 20 ng/L. Shake well to disperse spike.

11.1.5. Measure 1 mL of each sample using an Eppendorf pipette and pour into a labeled 2.0 mL injection vial. This includes the LCS and method blank samples as well.

11.1.6. Be sure to "prepare" the pipette by collecting two 1 mL aliquots and disposing of them, and then collect the aliquot for testing.

11.1.7. Add 83 uL of surrogate solution (PFC IDA Spike Solution, Section 7.5) into each vial for each sample and QC sample. This will result in an extract concentration of 50 ng/L for the surrogate.

11.1.8. Add 577 uL of methanol to each sample for a final solvent composition of 60 methanol:water.

11.1.9. Seal the vial with a polypropylene screw cap. Note: Teflon lined caps can not be used due to detection of low level concentration of PFAS.

11.1.10. Vortex to mix the mixture well.

11.2. Instrument Analysis

11.2.1. Suggested operation conditions are listed in Tables 1A-1C below:

Table 1A: [illegible]
HPLC Conditions ([redacted])
Column (Column temp = [redacted]°C)	[redacted]
Mobile Phase Composition	A = [redacted] B = [redacted]

Table 1A - Routine Instrument Operating Conditions					
HPLC Conditions ([redacted])					
Gradient Program	**Time (min)**	**%A**	**%B**	**Curve**	**Flow Rate (mL/min)**
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	Maximum Pressure limit = 5,000 psi				
Injection Size	[redacted] (fixed amount throughout the sequence)				
Run Time	[redacted]				
MS Interface Mode	ESI Negative Ion. Minimum of 10 scans/peak.				
Ion Spray Voltage (kV)	[redacted]				
Entrance Potential (V)	[redacted]				
Declustering Potential (V)	[redacted]				
Desolvation Temp	[redacted]				
Curtain Gas (nitrogen) Flow	[redacted]				
Collision Gas (nitrogen) Flow	[redacted]				

Table 1B - Routine Instrument Operating Conditions						
Mass Spectrometer Scan Settings ([redacted])						
Compound	**Comments**	**Reaction (MRM)**	**Dwell (sec)**	**Ent. Pot. (V)**	**Col. Energy (V)**	**Declu. Pot. (V)**
PFBS	Perfluorobutanesulfonate	299 > 80	[redacted]	[redacted]	[redacted]	[redacted]
13C3-PFBS	IDA	302 > 83	[redacted]	[redacted]	[redacted]	[redacted]
PFHpA	Perfluoroheptanoic acid	363 > 319	[redacted]	[redacted]	[redacted]	[redacted]
13C4-PFHpA	IDA	367 > 322	[redacted]	[redacted]	[redacted]	[redacted]
PFHxS	Perfluorohexanesulfonate	399 > 80	[redacted]	[redacted]	[redacted]	[redacted]
18O2-PFHxS	IDA	403 > 84	[redacted]	[redacted]	[redacted]	[redacted]
PFOA	Perfluorooctanoic acid	413 > 369	[redacted]	[redacted]	[redacted]	[redacted]
13C4PFOA	IDA	417 > 372	[redacted]	[redacted]	[redacted]	[redacted]
PFNA	Perfluorononanoic acid	463 > 419	[redacted]	[redacted]	[redacted]	[redacted]
13C5-PFNA	IDA	468 > 423	[redacted]	[redacted]	[redacted]	[redacted]
PFOS	Perfluorooctanesulfonate	499 > 80	[redacted]	[redacted]	[redacted]	[redacted]
13C4-PFOS	IDA	503 > 80	[redacted]	[redacted]	[redacted]	[redacted]

Table 1C				
Native Compounds	**Typical Native RT (minutes)**	**IDA analog**	**Typical IDA RT (minutes)**	**Quantitation Method**
PFBS	[redacted]	13C3-PFBS	[redacted]	Isotope Dilution
PFHpA	[redacted]	13C4-PFHpA	[redacted]	Isotope Dilution
PFHxS	[redacted]	18O2-PFHxS	[redacted]	Isotope Dilution
PFOA	[redacted]	13C4-PFOA	[redacted]	Isotope Dilution
PFNA	[redacted]	13C5-PFNA	[redacted]	Isotope Dilution
PFOS	[redacted]	13C4-PFOS	[redacted]	Isotope Dilution

11.2.2. Tune and calibrate the instrument as described in Section 10.

11.2.3. A typical run sequence is as follows:

- Primer (A number of primers are injected for conditioning of the instrument before analysis, especially when the instrument was idled or changed from a different analysis).
- Blank
- Calibration Curve
- ICB
- ICV
- PFOA RT marker (as needed)
- Rinse Blank (RB, not linked to anything)
- MB
- LCS
- LCSD (if applicable)
- Sample 1
- Sample 1 MS (if applicable)
- Sample 1 MSD (if applicable)
- Sample 2 (up to sample 10 before next CCV)
- CCV
- Up to 10 samples.
- End sequence with CCV

12. CALCULATIONS

Refer to Section 12 of the main body of this SOP for calculation information.

13. METHOD PERFORMANCE

Refer to Section 13 of the main body of this SOP for method performance information.

1□. POLLUTION PRE□ENTION

Refer to Section 1□ of the main body of this SOP for pollution pre□ention information.

1□. □ ASTE MANA□EMENT

Refer to Section 1□ of the main body of this SOP for □aste mana□ement information.

1□. REFERENCES

Refer to Section 1□ of the main body of this SOP for reference information.

1□. METHOD MODIFICATIONS

1□.1. Refer to Section 1□ of the main body of this SOP for modifications from □ ethod □3□□ e□cept as detailed belo□ □

1□.1.1. □ ater samples are prepared at 1.□m□□not 2□□m□.

1□.1.2. □ ater sample containers are not preser□ed □ith □ri□ma. □oldin□time has been chan□ed to 2□days for analysis.

1□.1.3. □he eluents and □P□□ confi□uration differs. □s a result the final e□tract is in □□□□□methanol□□ater.

1□. ATTACHMENTS

□here are no attachments to this □ppendi□.

1□. RE□ISION HISTOR□

Re□isions prior to □□1□2□1 □ha□e been remo□ed and are a□ailable in pre□ious □ersions of this SOP.

1□.1. □ S□□□□□□□2□□□ttachment 1□Re□ision 3.□□□ffecti□e □□23□2□1□

1□.1.1. □o chan□es to the attachment □ith this re□ision.

1□.2. □ S□□□□□□□2□□□ttachment 1□Re□ision 3.□□□ffecti□e □□□□□2□1□

1□.2.1. □o chan□es to the attachment □ith this re□ision.

1□.3. □ S□□□□□□□2□□□ttachment 1□Re□ision 3.□□□ffecti□e □□1□2□1□

1□.3.1. □o chan□es to the attachment □ith this re□ision.

1□.□. □ S□□□□□□□2□□□ttachment 1□Re□ision 3.□□□ffecti□e □2□2□2□1□

1□.□.1. □o chan□es to the attachment □ith this re□ision.

1□.□. □ S□□□□□□ □2□□ttachment 1□Re□ision 3.□□□ffecti□e □2□13□2□1□

1□.□.1. Removed Section 3.6, "□ P□O□□ Perfluoro□n□□1□2□3□□□13□□□octanoic acid. □arbon□13 labeled PFOA".

1□.□.2. Remo□ed Section 3.7, "□ P□OS□ Perfluoro□1□□1□2□3□□□ 13□□□octanesulfonic acid. □arbon□13 labeled PFOS".

1□.□.3. Section 7.2.3 removed, "MPFOS".

1□.□.□. Section 7.3 removed, "PFCA and PFSA".

1□.□.□. Section □3 added "13□3□P□□S" entry to table.

1□.□.□. Section 10.11.3 revised to, "Projects performed under the auspices of the □o□□□O□ □S□ □□ersion □.1□and the state of □e□ □ersey must meet these criteria for the □□□□□nalyte concentrations must be □ithin □3□□ of their true values for all analytes, IDA and target."

1□.□.□. □able 1□□re□ised P□□S □□A from "18O2□PFHxS" to "13C3□PFBS" and updated entry □alues.

1□.□.□. Table 1C, revised "IS Analog" to "IDA Analog", revised the PFBS IDA from "18O2□PFHxS" to "13C3□PFBS", and updated entry values.

1□.□.□. □ditorial chan□es.

1□.□. □ S□□□□□□ □2□□□ttachment 1□Re□ision 3.3□□ffecti□e 12□□32□1□

1□.□.1. □o chan□es to the attachment □ith this re□ision.

1□.□. □ S□□□□□□ □2□□□ttachment 1□Re□ision 3.2□□ffecti□e □□2□□2□1□

1□.□.1. □o chan□es to the attachment □ith this re□ision.

1□.□. □ S□□□□□□ □2□□□ttachment 1□Re□ision 3.1□□ffecti□e □□21□2□1□

1□.□.1. □o chan□es to the attachment □ith this re□ision.

1□.□. □ S□□□□□□ □2□□□ttachment 1□Re□ision 3.□□□ffecti□e □□□13□2□1□

1□.□.1. □pdated labelin□and formattin□of □ables 1□□1□.

1□.□.2. □dded section 11.2.3□detailin□a typical run se□uence.

1□.1□.□ S□□□□□□ □2□□ttachment 1□Re□ision 2.□□□ffecti□e 11□2□2□1□

1□.1□.1. □o chan□es to the attachment □ith this re□ision.

1□.11. □ S□□□□□□ □2□□ttachment 1□Re□ision 2.□□□ffecti□e 11□□□2□1□

1□.11.1. Section 11.2.1□Routine □nstrument Operatin□ □onditions table □[redacted]), added "Minimum of 10 scans/peak".

1□.12. □ S□□□□□□ □2□□ttachment 1□Re□ision 2.□□□ffecti□e □□22□2□1□

1□.12.1. Section 6.5, removed "□he □items abo□e are to be maintained in the dra□er labeled "Segregated Supplies for in line SP□ □nalysis" in the LC/MS instrument room."

1□.12.2. □dded Sections □.1 – □.3

1□.12.3. □pdated Section 11.1.

1□.12.□. □ditorial chan□es.

1□.13. □ S□□□□□□ □2□□ttachment 1□Re□ision 2.□□□ffecti□e □□11□2□1□

1□.13.1. □o re□isions to this attachment.

1□.1□.□ S□□□□□□ □2□□ttachment 1□Re□ision 2.□□□ffecti□e □□1□□2□1□

1□.1□.1. □o re□isions to this attachment.

1□.1□.□ S□□□□□□ □2□□ttachment 1□Re□ision 2.□□□ffecti□e □□2□2□1□

1□.1□.1. □o re□isions to this attachment.

1□.1□.□ S□□□□□□ □2□□ttachment 1□Re□ision 2.3□ ffecti□e □□1□□2□1□

1□.1□.1. Changed all mentions of "direct aqueous injection (DAI)" to "in line solid phase extraction (SPE)."

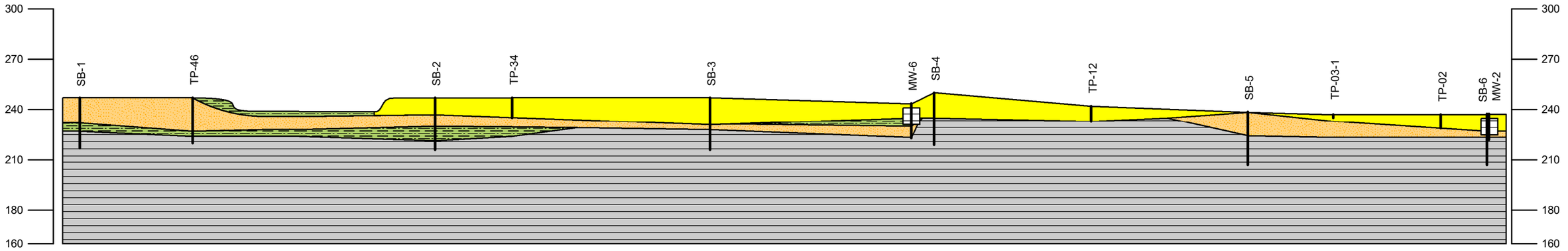
1□.1□.2. □nserted Section 1□.1□and chan□ed formattin□of the modifications to □ ethod □3□ to Section 1□2 and subheadin□s.

Attachment 1
Figure 6-1b
Remedial Investigation Report (RI) (Geosyntec 2018)

Cross-Section A-A'

North
A

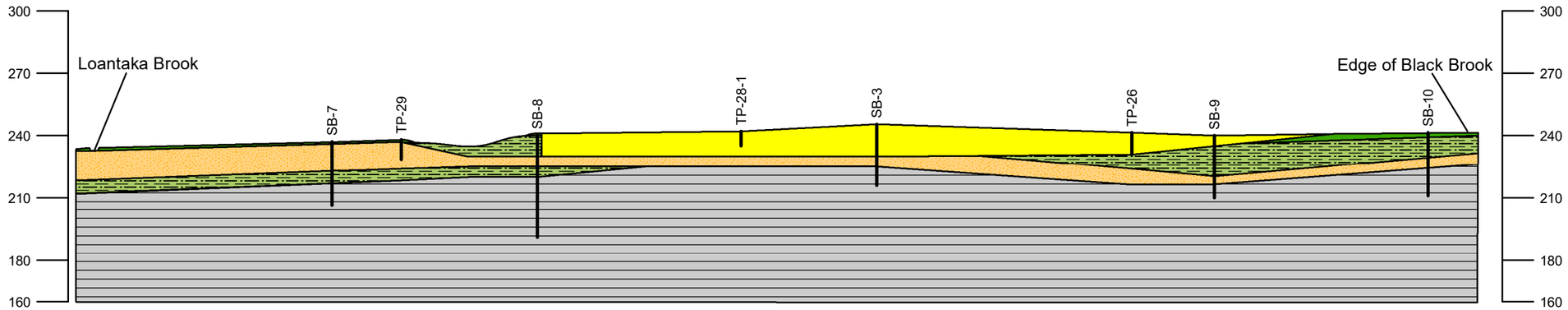
South
A'

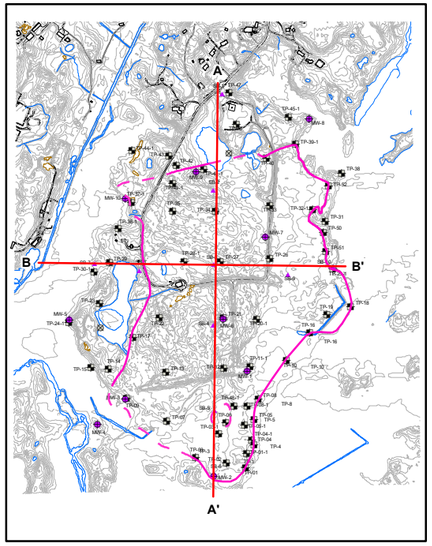


Cross-Section B-B'

West
B

East
B'

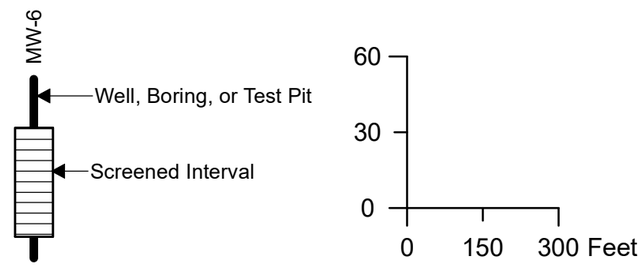




5x vertical exaggeration.
Geologic information interpreted from cross-sections provided by Arcadis dated January 2012.

Geologic Legend

- Topsoil
- Landfilled Material
- Sand
- Silt
- Clay



K:\Malvern\Finals & Ib Conceptual Site Model 2017 XS extended.mxd

Conceptual Site Model Cross-Sections

ROLLING KNOLLS LANDFILL SUPERFUND SITE
CHATHAM, NEW JERSEY

Geosyntec consultants

Princeton, NJ | August 2017

Figure
6-1b