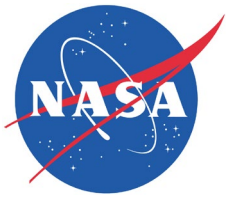
National Aeronautics and Space Administration



Final

Uniform Federal Policy for Sampling and Analysis Plan

Operable Unit 7
FORMERLY USED DEFENSE SITE
PROJECT 15
CONSTRUCTION DEBRIS LANDFILL
Remedial Action Work Plan

Goddard Space Flight Center
Wallops Flight Facility
Wallops Island, Virginia

February 2023

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CERTIFICATION

I certify that the information contained in or accompanying this document is true, accurate, and complete.

As to any portion of this document for which I cannot personally verify its accuracy, I certify under penalty of law that this document and all attachments were prepared in accordance with procedures designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who manage the system, or those persons directly responsible for gathering the information, or the immediate supervisor of such person(s), the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fines and imprisonment for knowing violations.

Signature: DAVID LIU Digitally signed by DAVID LIU Date: 2023.03.02 12:01:34 -05'00'

Name: David Liu

Title: NASA Project Coordinator

Executive Summary

This SAP outlines the site-specific organization, project management, objectives, planned activities, measurement, data acquisition, assessment, oversight, and data review procedures associated with the Operable Unit (OU) 7 Construction Debris Landfill (CDL) removal action located at the Goddard Space Flight Center's (GSFC) Wallops Flight Facility (WFF) in Wallops Island, Virginia. General protocols for sample collection, handling, and storage; chain-of-custody, laboratory and field analyses; data validation; and reporting are also addressed in this SAP. This SAP details the sample locations, rationale, and methods needed to achieve the goals and objectives of pre-excavation soil sampling at the CDL. Field activities will be conducted in accordance with the Standard Operating Procedures (SOPs) identified in this SAP and will meet the requirements of the Health and Safety Plan (HASP) to be submitted under separate cover. The grading design and erosion and sediment control plan will be submitted under separate cover.

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Acronyms and Abbreviations

µg/L	microgram per liter
ºC	Degree Celsius
%R	Percent recovery
AAOC	Administrative Agreement on Consent
Ca	Analytical Data Completeness
CASRN	Chemical Abstract Service Registry Number
CCV	Continuing Calibration Verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
COC	Chemical of Concern
Cs	Sampling Completeness
CTE	Central Tendency Exposure
DO	Dissolved Oxygen
DPT	direct push technology
DQI	Data Quality Indicator
DTW	deep temporary well
DV	Data Validation
DVM	Data Validation Manager
EDD	Electronic Data Deliverable
EE/CA	Engineering Evaluation/Cost Analysis
EICP	Extracted Ion Current Profile
ELCR	Excess Lifetime Cancer Risk
EPA	United States Environmental Protection Agency
ESV	Ecological Screening Value
FOL	Field Operations Leader
FS	Feasibility Study
FTMR	Field Task Modification Request
GAC	Granular Activated Carbon
GC/MS	Gas Chromatograph/Mass Spectrometry
GSFC	Goddard Space Flight Center
HASP	Health and Safety Plan
HDPE	High-density Polyethylene
HI	Hazard index
HSM	Health and Safety Manager
ICAL	Initial Calibration
ICP-AES	Inductively Coupled Plasma- Atomic Emission Spectroscopy
IDIQ	Indefinite Delivery Indefinite Quantity
IDQTF	Intergovernmental Data Quality Task Force
IDW	Investigation-Derived Waste
IS	Internal Standard
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LTM	Long-term monitoring
MB	Method Blank
MCL	Maximum Contaminant Level
MD	Matrix Duplicate
MDL	Method Detection Limit
mL	Milliliter

MPC	Measurement Performance Criteria
MS	Matrix Spike
MSA	Method of Standard Additions
MSD	Matrix Spike Duplicate
msl	Mean sea level
N/A	Not Applicable
NACA	National Advisory Committee for Aeronautics
NASA	National Aeronautics and Space Administration
NFG	National Functional Guidelines
NOAA	National Oceanic and Atmospheric Administration
NTCRA	Non-Time-Critical Removal Action
NTU	Nephelometric turbidity unit
ORP	Oxidation-Reduction Potential
OU	Operable Unit
PA	Preliminary Assessment
PAL	Project Action Limit
PDF	Portable Document Format
PDS	Post digestion spike
PFAS	Per-and Polyfluoroalkyl Substances
PID	Photoionization Detector
PM	Project Manager
POC	Point of Contact
PPE	Personal Protective Equipment
PQLG	Project Quantitation Limit Goal
PRAP	Proposed Remedial Action Plan
PRG	Preliminary Remedial Goal
QA	Quality Assurance
QAM	Quality Assurance Manager
QAPP	Quality Assurance Project Plan
QC	Quality Control
RAO	Removal Action Objective
RCRA	Resource Conservation and Recovery Act
RI	Remedial Investigation
RME	Reasonable Maximum Exposure
ROD	Record of Decision
RPD	Relative Percent Difference
RPM	Remedial Project Manager
SAP	Sampling and Analysis Plan
SFI	Supplemental Field Investigation
SGS	SGS North America – Orlando, Florida
SI	Site Inspection
SOP	Standard Operating Procedure
SSHO	Site-Specific Health and Safety Officer
SVOC	Semivolatile organic compound
TBD	To Be Determined
AECOM	AECOM, Inc.
TO	Task Order
UFP	Uniform Federal Policy
U.S.	United States

VDEQ	Virginia Department of Environmental Quality
VOC	Volatile organic compound
WFF	Wallops Flight Facility

SAP Worksheet #1 Title and Approval Page

(UFP-QAPP Manual Section 2.1- Worksheet #1)

Document Title: Uniform Federal Policy for Sampling and Analysis Plan, Operable Unit 7, Formerly Used Defense Site, Project 15 Construction Debris Landfill, Goddard Space Flight Center, Wallops Flight Facility, Wallops Island, Virginia

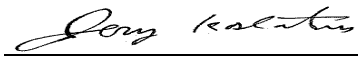
Lead Organization: National Aeronautics and Space Administration (NASA)

Preparer's Name and Organizational Affiliation: AECOM Technical Services, Inc. (AECOM)

Preparer's Address, Telephone Number, and email Address: 12420 Milestone Center Drive, Suite 150, Germantown, MD 20876, 301-250-2934, jerry.kashatus@aecom.com; prepared under Indefinite Delivery Indefinite Quantity (IDIQ) Contract 80KSC019D0010, Task Order (TO) 80GSFC20F0047

Preparation Date (Day/Month/Year): December 21, 2022

This Uniform Federal Policy (UFP) - Sampling and Analysis Plan (UFP-SAP) was prepared and submitted in accordance with the requirements of the 2021 Resource Conservation and Recovery Act (RCRA) Administrative Agreement and Order on Consent (AAOC) between NASA and the United States (U.S.) Environmental Protection Agency (EPA) (EPA Docket Number RCRA-03-2021-0022TH).

Investigative Organization's Project Manager (PM):	 2/28/23 Signature/Date Jerry Kashatus, AECOM PM
Investigative Organization's Project Quality Assurance (QA) Manager (QAM):	 2/28/23 Signature/Date Naoum Tavantzis, AECOM/Project Chemist
Lead Organization's PM:	John Brodt Digitally signed by John Brodt Date: 2023.03.02 09:19:18 -05'00' Signature/Date John Brodt, NASA Remedial Project Manager (RPM)

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SAP Worksheet #2 Distribution List

(UFP-QAPP Manual Section 2.3.1-Worksheet #3)

SAP Recipients	Title	Organization	Telephone Number	Email Address
John Brodt	NASA RPM	NASA	216-433-6028	John.p.brodt@nasa.gov
Susan Dunn	Facility Point of Contact (POC)	NASA-Bluestone Environmental Group, Inc.	757-824-1832	Susan.K.Dunn@nasa.gov
Jennifer Joyal, P.E.	Program Manager	AECOM	407-276-4550	Jennifer.Joyal@aecom.com
Kathy Smith	Laboratory PM	Pace Laboratories	803-791-9700	Kathy.Smith@pacelabs.com
Jerry Kashatus	AECOM Facility Coordinator/ PM	AECOM	240-370-3285	Jerry.Kashatus@aecom.com
Naoum Tavantzis	AECOM QAM/Project Chemist	AECOM	919-461-1178	naoum.tavantzis@aecom.com
Pete Wray	AECOM Health and Safety Manager (HSM)	AECOM	301-781-5872	Pete.Wray@aecom.com
Kyle Newman	VDEQ RPM	State Regulator	804-698-4452	Kyle.Newman@deq.virginia.gov
Lorie Baker	EPA RPM	Federal Regulator	215-814-3355	Baker.Lorie@epa.gov
Tara Bhat	AECOM Data Manager	AECOM	301-944-2610	tara.bhat@aecom.com
Naoum Tavantzis	AECOM Data Validation Manager (DVM)	AECOM	919-461-1178	naoum.tavantzis@aecom.com
Gus Remenicky	AECOM Field Operations Leader (FOL) Site-Specific Health and Safety Officer (SSHO)	AECOM	302-304-4188	gus.remenicky@aecom.com

Note: Managers for individual organizations shall ensure that support staff have access to the current SAP prior to conducting work.

SAP Worksheet #3 Project Personnel Sign-Off Sheet

(UFP-QAPP Manual Section 2.3.2- Worksheet #4)

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

1. In the case of regulatory agency, personnel with oversight authority, approval letters or emails will constitute verification that applicable sections of the SAP have been reviewed. Copies of regulatory agency approval letters / emails will be retained in the project files as project records
2. Emails will be sent to the NASA, AECOM, and subcontractor project personnel who will be requested to verify by email that they have read the applicable SAP / sections and the date on which they were reviewed. Copies of the verification email will be included in the project files

   A copy of the signed worksheet below will be retained in the project files and identified as a project document in **Worksheet #12**.

Key personnel will be instructed to read the SAP prior to attending the internal kick-off meeting for field activities. The AECOM PM will track when the reviews have been completed, obtain signatures, and ensure that the completed sign-off sheet is included in the central project file.

Name[1]	Organization/Title	Telephone Number	Signature	Date SAP Read
NASA				
John Brodt	NASA/RPM	216-433-6028	See **Worksheet #1** for signature	
Susan Dunn	NASA-Bluestone Environmental/ Facility POC	757-824-1832	Email receipt	
Regulatory Agencies				
Kyle Newman	VDEQ/ RPM	804-698-4452	See Concurrence Letter for signature	
Lorie Baker	EPA Region 3/ RPM	215-814-3355	See Concurrence Letter for signature	
AECOM				
Naoum Tavantzis	AECOM/ QAM and Project Chemist	919-461-1178	See **Worksheet #1** for signature	
Tara Bhat	AECOM/ Data Manager	301-944-2610	Email receipt	
Naoum Tavantzis	AECOM/ DVM	919-461-1178	Email receipt	
Pete Wray	AECOM/ HSM	301-781-5872	Email receipt	
Jerry Kashatus	AECOM/ Facility Coordinator and PM	210-370-3285	See **Worksheet #1** for signature	

Name[1]	Organization/Title	Telephone Number	Signature	Date SAP Read
Gus Remenicky	AECOM/ FOL/SSHO	302-304-4188	Email receipt	
Subcontractor				
Kathy Smith	Laboratory PM	803-791-9700	Email receipt	

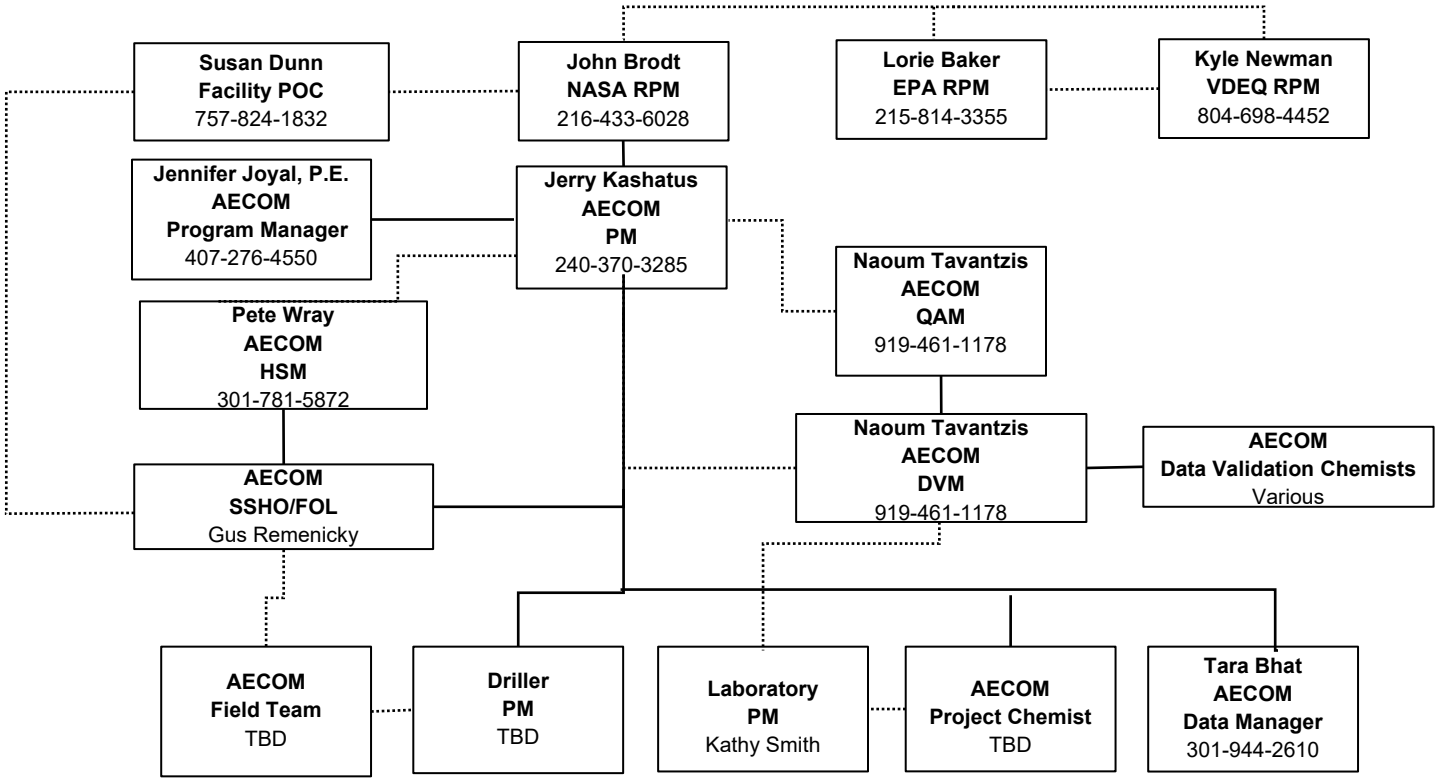
Footnote:

1 - Persons listed on this worksheet will be responsible for distributing the SAP to the appropriate people within their organization.

SAP Worksheet #4 Project Organizational Chart

(UFP-QAPP Manual Section 2.4.1- Worksheet #5)

Lines of Authority ———————— Lines of Communication - - - - - - - - - - - - -



SAP Worksheet #5 Communication Pathways

(UFP-QAPP Manual Section 2.4.2-Worksheet #6)

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathway To/From, etc.)
Draft SAP Review/Revisions	AECOM PM NASA RPM	Jerry Kashatus John Brodt	240-370-3285 216-433-6028	Within two days of completing the draft SAP, the SAP will be submitted to NASA by the AECOM PM. NASA comments will be resolved to the satisfaction of the NASA PM and the SAP will be submitted to NASA for signature.
Regulatory Agency Interface	NASA RPM	John Brodt	216-433-6028	When due according to the project schedule, the draft SAP will be submitted to the regulatory agency by the NASA PM or designee.
Field Progress Reports	AECOM FOL	Gus Remenicky	302-304-4188	The AECOM FOL will provide email or verbal reports to the AECOM PM on a schedule arranged by the PM.
Stop Work Due to Safety Issues	AECOM FOL AECOM PM NASA RPM Facility POC	Gus Remenicky Jerry Kashatus John Brodt Susan Dunn	302-304-4188 240-370-3285 216-433-6028 757-824-1832	If AECOM is the responsible party for a stop work command, the AECOM FOL will inform on-site personnel, subcontractor(s), and Facility POC, and the identified team members within 1 hour (verbally or by email). If a subcontractor is the responsible party, the subcontractor must inform the AECOM FOL within 15 minutes, and the AECOM FOL will then follow the procedure described above.
SAP Changes Prior to Field/ Laboratory Work	AECOM FOL AECOM PM NASA RPM Facility POC	Gus Remenicky Jerry Kashatus John Brodt Susan Dunn	302-304-4188 240-370-3285 216-433-6028 757-824-1832	The AECOM PM will submit an email to the NASA PM within 5 days requesting the modification. If necessary, the AECOM PM will discuss out of scope items with the NASA PM. SAP amendments will be submitted by the AECOM PM to the NASA PM for review and approval.
SAP Changes in the Field	AECOM FOL AECOM PM	Gus Remenicky Jerry Kashatus	302-304-4188 240-370-3285	The AECOM FOL will verbally inform the AECOM PM within 24 hours of realizing the need for a change. The AECOM PM will document the change via an email to the NASA PM within 5 days.
Field Corrective Actions	AECOM FOL AECOM PM	Gus Remenicky Jerry Kashatus	302-304-4188 240-370-3285	The AECOM FOL will initiate corrective actions and will notify the AECOM PM verbally within 1 business day of taking action.
Sample Receipt Variances	Laboratory PM AECOM FOL AECOM PM	Kathy Smith Gus Remenicky Jerry Kashatus	803-791-9700 302-304-4188 240-370-3285	The Laboratory PM will report variances to the AECOM FOL or PM within 24 hours of identifying a variance and those individuals will attempt to resolve the variance with the laboratory. If the variance cannot be resolved, the AECOM PM will notify the NASA PM within 1 business day of the variances being brought to his or her attention. If necessary, the NASA PM will take corrective action commensurate with the deficiency.

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathway To/From, etc.)
Laboratory Quality Variances	Laboratory PM AECOM QAM/Project Chemist AECOM PM	Andrea Colby Naoum Tavantzis Jerry Kashatus	609-495-5321 919-461-1178 240-370-3285	The Laboratory PM will notify (verbally or via email) the AECOM QAM/Project Chemist within 1 business day of when an issue related to laboratory data is discovered. The AECOM QAM/Project Chemist will notify (verbally or via email) the data validation (DV) staff and the AECOM PM within 1 business day. The Laboratory PM will ensure that all quality variances are presented in the Case Narrative of the Analytical Laboratory Report.
Analytical Corrective Actions	Laboratory PM AECOM FOL AECOM QAM/Project Chemist AECOM PM NASA PM	Andrea Colby Gus Remenicky Naoum Tavantzis Jerry Kashatus John Brodt	609-495-5321 302-304-4188 919-461-1178 240-370-3285 216-433-6028	If the impact of an identified deficiency is limited to this project, it will be resolved between the Laboratory PM and AECOM PM and support staff and will be documented in the project report. If the deficiency is systemic and potentially affects other projects, the AECOM PM will verbally advise the NASA PM within 24 hours of notification from the QAM/Project Chemist. The NASA PM and AECOM PM will work together to determine the appropriate corrective action for the identified deficiency. Corrective actions may include a consult with the AECOM QAM/Project Chemist and coordination with the laboratory.
Reporting Data Validation Issues	AECOM DVM AECOM DV Chemist AECOM PM	Naoum Tavantzis Various Jerry Kashatus	919-461-1178 Various 240-370-3285	AECOM DV Chemist will document data qualifications in the data validation report and database. For serious deficiencies the DVM will notify AECOM PM verbally or via email within 48 hours of recognizing that a significant laboratory quality deficiency has been detected that could affect this project and/or other projects.
Data Validation Corrective Action	AECOM DVM AECOM DV Chemist AECOM PM	Naoum Tavantzis Various Jerry Kashatus	919-461-1178 Various 240-370-3285	If a data validation issue cannot be resolved between the AECOM DV Chemist and DVM and the laboratory or the issue appears to be systemic, the AECOM PM will verbally advise the NASA PM within 24 hours of notification from the DV Chemist. The NASA PM and AECOM PM will work together to determine the appropriate corrective action for the identified deficiency. This may include a consult with the AECOM QAM/Project Chemist.
Project Report Review	AECOM PM AECOM support staff NASA PM	Jerry Kashatus Various John Brodt	240-370-3285 Various 216-433-6028	Internal reviews will be conducted by the AECOM PM and support staff. Comments will be resolved internally to the satisfaction of the AECOM PM. When satisfied with resolution of all comments, the report will be submitted by the AECOM PM or designee to the NASA PM in accordance with the project scope of work requirements. Upon resolution of NASA comments, the report will be submitted to the NASA PM for transmittal to the regulators for review. Comments will be resolved to the satisfaction of the NASA PM, revisions will be made, and the report will be finalized for final distribution and submittal.

SAP Worksheet #6 Personnel Responsibilities and Qualifications Table

(UFP-QAPP Manual Section 2.4.3- Worksheet #7)

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
John Brodt	RPM	NASA	Oversees implementation of the NASA Wallops Flight Facility (WFF) restoration program and AAOC.	To be provided upon request
Lorie Baker	RPM	EPA Region 3	Participates in scoping, data review, evaluation, and approves the SAP.	To be provided upon request
Kyle Newman	RPM	VDEQ	Participates in scoping, data review, evaluation, and approves the SAP	To be provided upon request
Susan Dunn	Facility POC	NASA-Bluestone Environmental Group, Inc	Point of contact for NASA WFF and related information pertaining to this investigation. Field work coordination.	To be provided upon request
Jerry Kashatus	PM	AECOM	Daily project management and administration. Ensures that health and safety requirements are implemented. Oversees project, financial, schedule, and technical day to day management of the project. Oversees project implementation, including scoping, data review, and evaluation for this project. Coordinates and oversees review of AECOM project deliverables.	Master of Science, Geology; over 35 years of professional experience in environmental industry.
Pete Wray	HSM	AECOM	Ensures that health and safety aspects of the AECOM Health and Safety Program are implemented. Oversees review of health and safety documents and approves health and safety documents.	Bachelor of Science; Business Management; over 18 years of professional experience in occupational safety and industrial hygiene.
Gus Remenicky	FOL	AECOM	Supervises, coordinates, and performs field sampling activities. Ensures that health and safety requirements are implemented during field work. Functions as the on-site communications link between field staff members, the facility POC, and the AECOM PM. Oversees mobilization and demobilization of all field equipment and subcontractors. Ensures proper maintenance of site logbooks, field logbooks, and field recordkeeping. Identifies and resolves problems in the field, resolving difficulties via consultation with the Facility POC and NASA PM, implementing and documenting corrective action procedures, and facilitating communication between the field team and project management.	Bachelor of Science; Environmental Science; over 15 years of professional experience in performing and overseeing site investigation and remediation activities, building deconstruction, and health and safety oversight.

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Gus Remenicky	SSHO	AECOM	Responsible for on-site project-specific health and safety training and monitoring site conditions. Details of health and safety responsibilities are presented in the Health and Safety Plan (HASP).	
Naoum Tavantzis	QAM/ Project Chemist	AECOM	Coordinates analyses with laboratory chemists, ensures that the laboratory scope or work is followed, and that QA has been performed for QA data packages, and communicates with AECOM staff. Ensures that the project meets objectives from the standpoint of laboratory performance. Provides technical advice to the AECOM team on project chemistry matters. Functions as the primary interface with the subcontracted laboratory and the AECOM PM.	Bachelor of Arts; Environmental Science, Master of Business Administration, 14 years of experience in environmental analytical chemistry including 2 years of environmental laboratory experience and 12 years at AECOM as a project chemist.
Naoum Tavantzis	DVM	AECOM	Manages data validation activities within AECOM, including ensuring QA of data validation deliverables, providing technical advice on data usability, and coordinating and maintaining the data validation review schedule.	Bachelor of Arts; Environmental Science, Master of Business Administration, 14 years of experience in environmental analytical chemistry including 2 years of environmental laboratory experience and 12 years at AECOM as a project chemist.
Tara Bhat	Data Manager	AECOM	Coordinates data receipt and upload to AECOM and NASA databases; manages and controls flow of data to ensure data are secure.	Bachelor of Arts in Chemistry (minor in Environmental Science and Policy), 4 years of experience with risk assessment and data management in Microsoft Excel, and 0.5 years of experience with environmental databases.
Kathy Smith	Laboratory PM	Pace Laboratories	Coordinates analyses with laboratory chemists, ensures that the laboratory scope of work is followed, performs QA of data packages, and communicates with AECOM staff.	To be provided upon request

SAP Worksheet #7 Special Personnel Training Requirements Table

(UFP-QAPP Manual Section 2.4.4- Worksheet #8)

Project-specific safety and training requirements are addressed in detail in the HASP.

SAP Worksheet #8 Project Scoping Session Participants Sheet

(UFP-QAPP Manual Section 2.5.1- Worksheet #9)

Scoping sessions that apply to the Operable Unit (OU) 7 - Construction Debris Landfill (CDL) will be held with regulators and stakeholders periodically and on an as-needed basis. These meetings will include project-specific meetings, teleconference calls, and RPM meetings. RPM meeting scoping sessions will be documented in meeting minutes prepared for each meeting or the template below will be used to document the scoping session. Meeting minutes will be reviewed and approved by attendees and captured in the NASA WFF Administrative Record.

Project Name:	WFF CDL Excavation	**Site Name:**	Construction Debris Landfill
Projected Date(s) of Sampling:	TBD	**Site Location:**	WFF
Project Manager:	Jerry Kashatus (AECOM); John Brodt (NASA)		
Date of Session:	TBD		
Scoping Session Purpose:	Project kick-off call		

Name	Title	Affiliation	Phone #	Email Address
John Brodt	NASA PM	NASA	216-433-6028	John.p.brodt@nasa.gov
Susan Dunn	Facility POC and Fieldwork Coordination	Bluestone Environmental Group Inc	757-824-1832	Susan.K.Dunn@nasa.gov
Jerry Kashatus	AECOM Facility Coordinator and PM	AECOM	240-370-3285	Jerry.Kashatus@aecom.com
TBD	TBD	TBD	TBD	TBD

Comments/Decisions: TBD

Action Items: TBD

SAP Worksheet #9 Problem Definition

(UFP-QAPP Manual Section 2.5.2- Worksheet #9)

This SAP focuses on the excavation of waste and affected soil at the CDL and the collection of environmental data that will support the excavation activities. This worksheet presents an overview of information for WFF and the CDL.

9.1 PHYSICAL SETTING

NASA WFF is in Accomack County, Virginia, on the Atlantic Coast of the Delmarva Peninsula (**Figure 9-1**), approximately 5 miles south of the Maryland/Virginia state boundary, and immediately west of Chincoteague Island. WFF is bounded on the north and east by Little Mosquito Creek, Simoneaston Bay to the east, and Wattsville Branch to the west, and it sits just above mean sea level (amsl), at 20 to 30 feet amsl. WFF consists of three separate land areas: Wallops Main Base, Wallops Mainland, and Wallops Island. The Project 15 CDL is located northeast of the runways, on the Main Base (**Figure 9-1)**.

Prior to development of the Site by the U.S. government, the current WFF property was farmland and marshland. The Department of Navy acquired the property in 1942 and established a World War II training facility. The Navy discontinued naval training operations at the facility in 1959, when NASA took custody of the Main Base as well as the northern portion of Wallops Island and the Mainland. NASA excised approximately 397 acres of the Main Base to the U.S. Fish and Wildlife Service in 1975.

9.1.1 Site Physical Characteristics

Site soils have been classified as Molena loamy sand. Molena loamy sand is characterized by 0 to 6 percent slopes, with rapid permeability and is somewhat excessively drained. The background study (Tetra Tech, 2021) found that Molena loamy sand had concentrations of naturally occurring metals that are statistically distinct from concentrations of these metals in the other soil type found on Wallops Main Base, the Bojac soils.

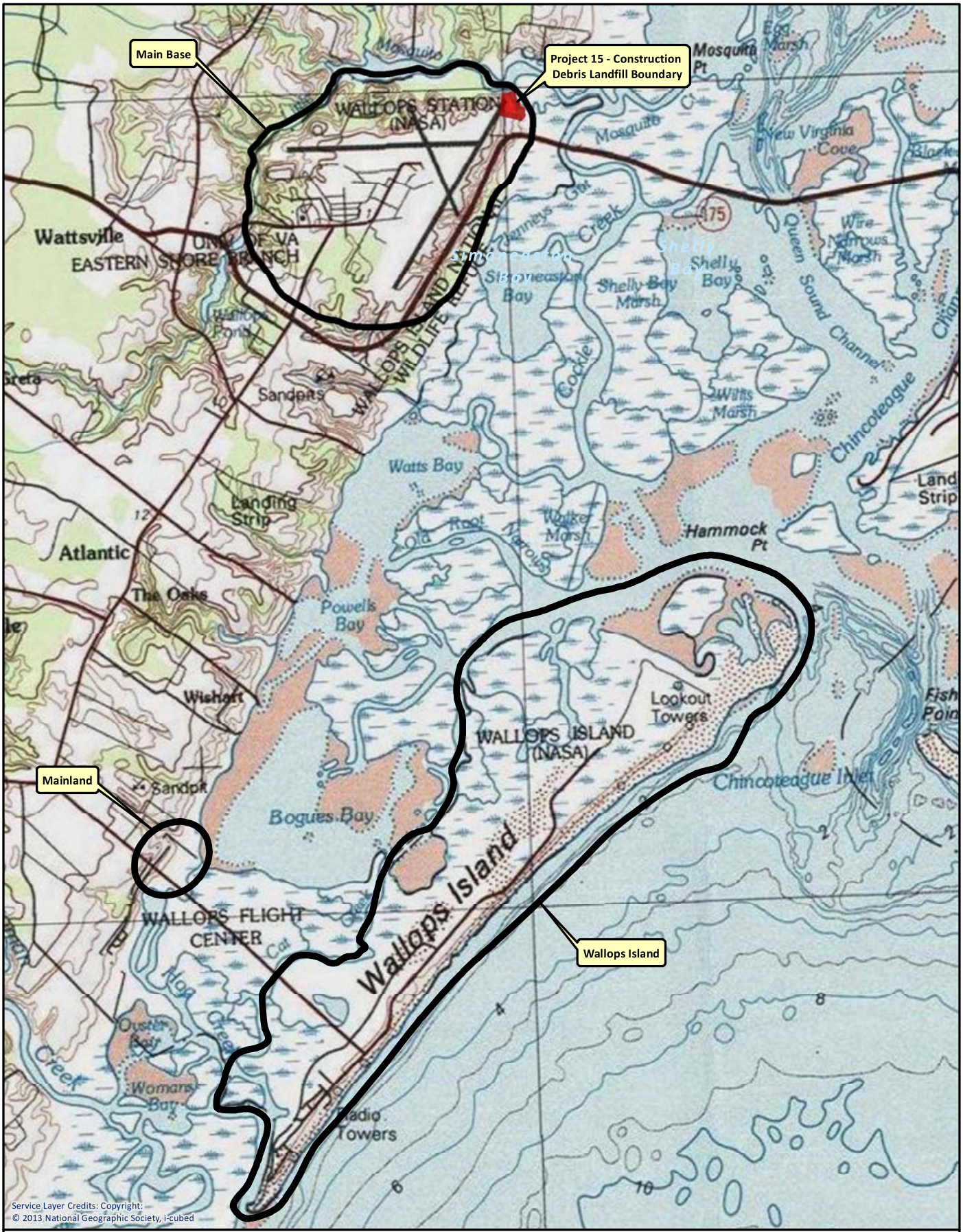
Based on a review of historical aerial photographs (see Section 9.4), four areas of concern (AOCs) were identified (**Figure 9-2**).

9.1.2 Site Topography and Drainage

Elevations on the WFF range from mean seal level to 42 feet amsl, with slopes of 1 to 2% (Versar 2011). In general, the land surface at the CDL slopes from north to south and west to east. Elevations ranged from just over 1-foot amsl on the southeastern edge to just under 21 feet amsl west of the access road. In the northern portion of the CDL, elevations decrease from about 19 feet west of the access road to about 10 feet, near the edge of the marsh. In the southern part of the Site, elevations just west of the access road are about 12 to 13 feet amsl; near the edge of the marsh, elevations are 3 to 4 feet amsl.

The Site is bound on the east by a narrow tidal channel of Mosquito Creek; water flows north to south in the immediate vicinity of the CDL. The tidal channel turns eastward south of the Site and rejoins Mosquito Creek.

Drainage on and adjacent to the CDL is controlled by ditches on the west side of the access road as well as two concrete culverts that connect the drainage ditch to west-to-east drainage features.



N

Legend

CDL Boundary

Location of Project 15, Construction Debris Landfill on Wallops Flight Facility

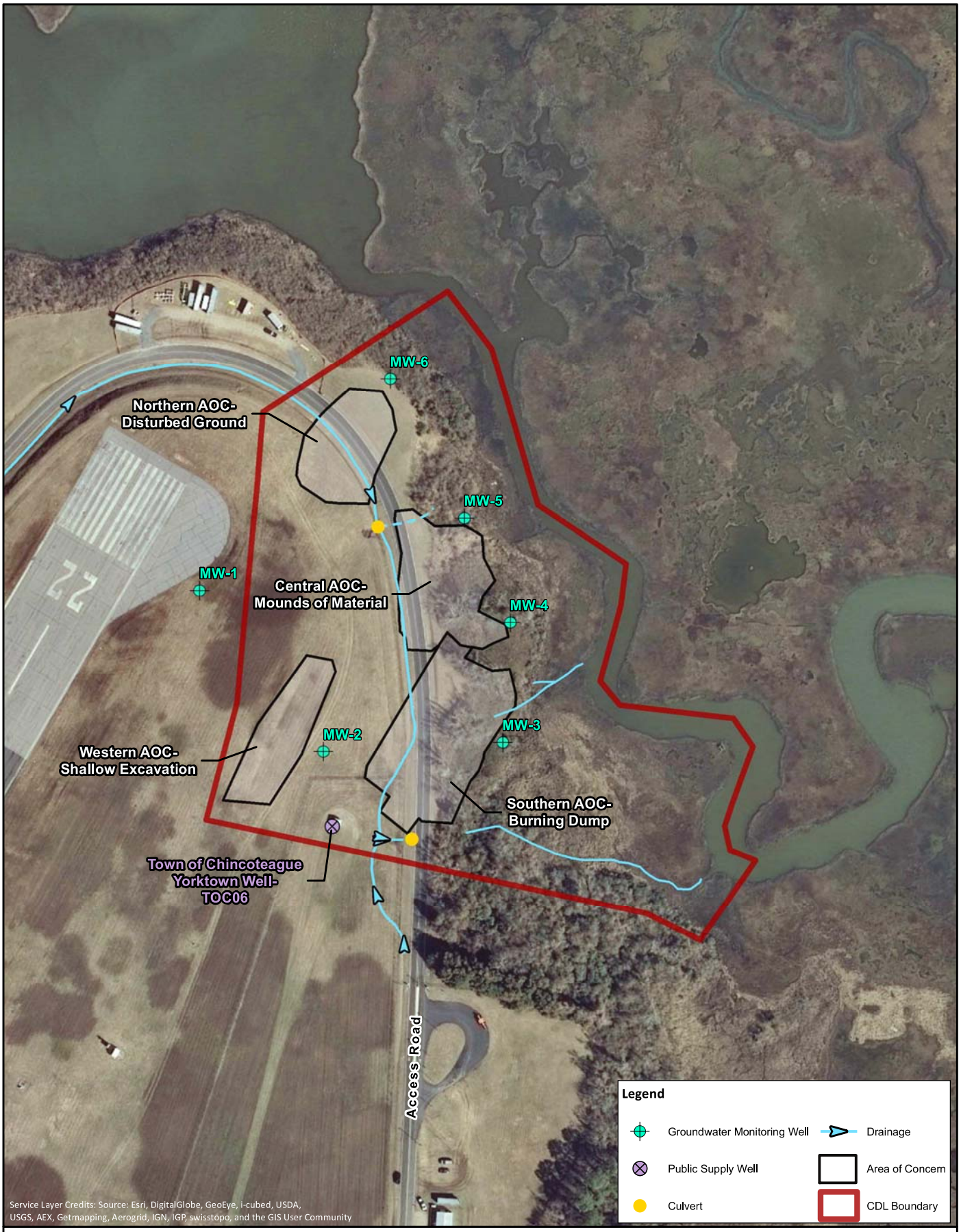
0 0.5 1 2 Miles

Project 15 CDL

WFF FUDS

Figure 9-1

FarmerRA C:_projects\Wallops Island CDL\GIS\MXD\Field_Plan\Figure2-1_Project15_CDL_FP_WFF_Layout.mxd 6/19/2014



N

Site Features

0 100 200 400 Feet

Project 15 CDL

WFF FUDS

Figure 9-2

FarmerRA C:_projects\Wallops Island CDL\GIS\MXD\SupplementalFieldInvestigation\Figure1-2_Project15_CDL_FP_CDL_Site_Features.mxd 7/1/2018

9.2 GEOLOGICAL SETTING

NASA WFF is located on the Eastern Shore of Virginia within the Atlantic Coastal Plain physiographic province. The geology of the Eastern Shore of Virginia can be characterized as a series of layered, unconsolidated, sediments that comprise an eastward-thickening wedge that dips to the northeast towards the Atlantic Ocean. Approximately 7,000 feet of sediment lies atop crystalline basement rock at NASA WFF.

9.2.1 Site Geology

The site geology is defined based on information collected during the Remedial Investigation (RI) (Versar, 2011) as well as the boring logs completed during the Supplemental Field Investigation (SFI). From the surface downward, the materials present at the Site and investigated during the RI and SFI include fill (including waste); Pleistocene-age sands, silts, and clays; and the Yorktown Formation.

Fill and Waste

Fill is present in the Central and Southern AOCs (**Figure 9-2**) and consists of both natural materials (predominantly sand) and man-made materials disposed of in the AOCs (waste). Natural material (sand) appears to have been used to grade the topography as well as to cover disposed waste material. The eastern extent of the waste is the western edge of the marsh in the Central AOC and the Southern AOC.

Within the Central AOC waste is found at the surface but is also buried beneath as much as 11 feet of sand fill. The waste includes empty metal drums, paint, scrap metal, rebar, glass bottles, bricks, cable, springs, tires, and broken concrete. During the SFI, waste was found at depths of up to 12 feet bgs; during the RI the maximum depth of waste in the Central AOC was about 13 feet bgs.

As in the Central AOC, waste in the Southern AOC is found both at the surface and buried beneath a layer of sand fill (up to 4 feet deep). Waste in the Southern AOC consists of cinders and burned rubbish (glass, cans, wires) (Versar, 2011) as well as plastic, plates, scrap metal, bathroom fixtures, railroad ties, and metal pipes. The maximum thickness of cover material (fill) was 4 feet of fine silty sand, but in most locations, only about 1 foot of cover sand was present. The maximum thickness of waste found during the SFI was 5 feet.

Pleistocene Deposits

Deep temporary wells were constructed during the SFI to sample the lower part of the surficial aquifer. The borings for these wells terminated at the top of the Yorktown Formation clay. Based on the logs from these four borings, the Pleistocene deposits are as much as 68 feet thick at the Site. The unit consists predominantly of fine to coarse quartz sands, coarsening downward and containing trace gravel in the lower part of the unit. Sands and minor organic clay layers consistent with the sandy estuarine and beach deposits are found in the upper part of the Pleistocene deposits near the marsh.

In the southern part of the Site, a significant clay unit—the Columbia Clay—is found within the Pleistocene deposits. At the Town of Chincoteague supply well TOC-06, this orange-brown and gray clay is present from 21 to 42 feet bgs. At DTW-4, located 140 feet east-northeast of TOC-06, the olive-grey clay and silt are present from 24.6 to 39.6 feet bgs.

Yorktown Formation

The Yorktown Formation clays and sands underlie the Pleistocene deposits. The top of the Yorktown Formation is a clay, distinguished by a distinctive greenish color, present at a depth of about 60 to 68 feet; the Yorktown clay was found in each of the deep temporary borings drilled in the SFI. The deep borings

completed during SFI did not penetrate more than approximately 5 feet into the Yorktown clay. At TOC-06, the Yorktown clay is 33 feet thick (from 84 to 117 feet bgs).

9.3 FACILITY HISTORY

NASA and its predecessor, the National Advisory Committee for Aeronautics (NACA), have had a presence at WFF since 1945. NACA established a rocket launch site on the southern portion of Wallops Island (Wallops Station) in 1945 under the direction of the Langley Research Center and launched its first rocket that year. NACA constructed launch and radar support and experimental facilities in 1946. Access to Wallops Island at that time was by water vessel only. Operations by NACA at WFF were limited to these test facilities until 1959 (Occu-Health, 1999). NASA absorbed the Langley Research Center and other NACA field centers and facilities when it was created by the U.S. government in 1958. NASA expanded its presence at WFF with the acquisition of the Main Base and Mainland parcels in 1959. The Wallops Station was named Wallops Flight Center in 1974, and the name was changed to WFF in 1981, when it became part of Goddard Space Flight Center (GSFC), Greenbelt, Maryland (Wallace, 1997).

The Navy operated the Chincoteague Naval Auxiliary Air Station at the Main Base from 1942 until the transfer to NASA in 1959. During that time, the Navy constructed runways, buildings, and other support facilities for naval aviation and aviation ordnance testing and training (Occu-Health, 1999). NASA continues to maintain the runways and occupies many of the structures and buildings that were present at the time of the property transfer from the Navy. In addition, NASA has expanded and constructed additional buildings to support their mission and provides support to other tenant organizations. The Navy used the north end of Wallops Island as a training area and maintained a series of ranges used to develop ordnance and ordnance delivery systems, but few permanent structures were built.

The mission of WFF has undergone several changes since it was established in 1959, but the main focus has been and continues to be rocket research, the management of suborbital projects, suborbital and orbital tracking, aeronautical research, and space technology research. Rocket motors are transported to WFF from other facilities.

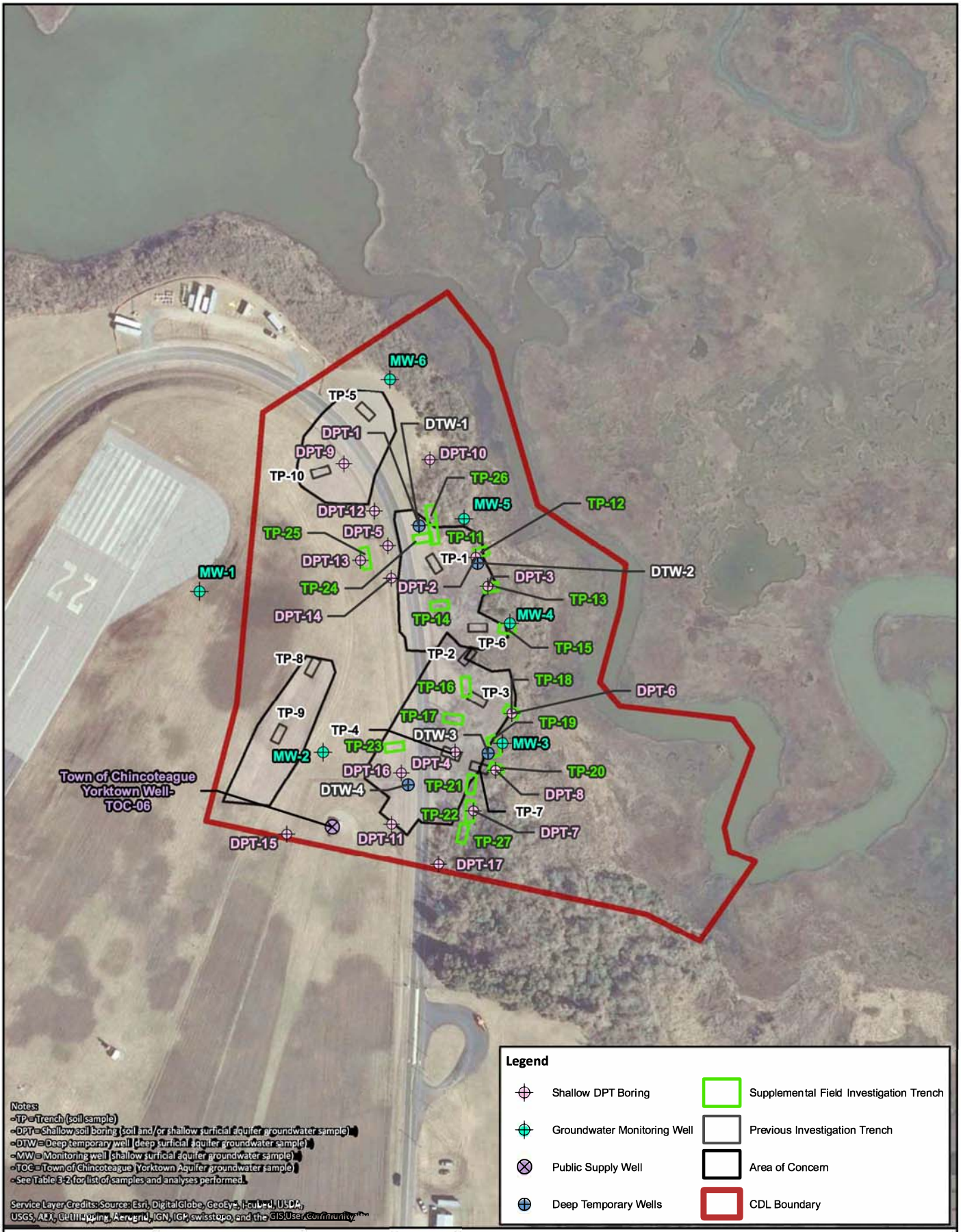
9.4 SITE HISTORY

Analysis of historical aerial photographs, as reported in the RI (Versar, 2011), document a series of ground scars indicative of waste disposal in areas at the northeast corner of WFF that are visible in 1949, 1954, 1959, and 1963 aerial photographs. By 1963, some of these features are no longer visible, and by 1974, none of them remained visible (USATEC, 2000). Investigations, described in the following subsections, confirmed that the area had been used for disposal of construction debris and ash. Based on the historical aerial photographs, four AOCs were identified (**Figure 9-2**).

In 2001, the Department of Defense and USACE assumed responsibility for various sites, including the CDL, under the FUDS program. Since then, the site has been the subject of the following investigations:

2002 Limited Site Investigation (LSI) (Science Applications International Corporation [SAIC], 2003): Samples were collected from three direct push technology (DPT) borings (**Figure 9-3**). Significant findings from the LSI are included in **Table 9-1**.

2004 Forensic Soil Sampling and Analysis (USACE): USACE collected a single soil sample from the contaminated soil layer found at approximately 7.5 feet in boring SB-CDL-01. The sample was analyzed for volatile petroleum hydrocarbons and extractable hydrocarbons using the Massachusetts Department of Environmental Protection (MADEP) method and USEPA Method 8015 gas chromatograph fingerprinting



Supplemental Field Investigation Soil and Groundwater Sample Locations

Project 15 CDL
WFF FUDS

Figure 9-3

FarmerRA C:_projects\Wallops Island CDL\GIS\MXD\SupplementalFieldInvestigation\Figure3-1_Project15_CDL_Soil_GW_SampleLocs_20180410.mxd 4/10/2018

Table 9-1: Observations and Soil Samples Collected During Environmental Investigations of the Construction Debris Landfill

Location	TD	Elevated PID (>25ppm)		Staining Observed		Waste Observed		Type of waste	Soil Sample Depth		Soil Sample Chemical Analysis	Soil Sample ID
	ft	from	to	from	to	from	to		from	to		
MIP-1	33.85											
MIP-2	31.15											
MIP-3	37.95											
MIP-4	27.15											
MIP-5	26.25	10	25			10	13	Paint can				
MIP-6	39.85											
MIP-7	29.80											
MIP-8	30.05											
TP-1/MIP-5	13								0	0.5	VOCs, SVOCs, Pesticides, PCBs, Cyanide, Metals	TP-1A (BURN AREA)
TP-1/MIP-5									0.5	2	VOCs, SVOCs, Pesticides, PCBs, Cyanide, Metals	TP-1B (BURN AREA)
TP-1/MIP-5						8.5/ 11	8.5/ 13	Paint can/ Empty, crushed 55-gallon drums; elevated PID; acrid odor	2	8.5	VOCs, SVOCs, Pesticides, PCBs, Cyanide, Metals	TP-1C (BURN AREA)
TP-2/MIP-7	8								0	0.5	VOCs, SVOCs, Pesticides, PCBs, Cyanide, Metals	TP-2A
TP-2/MIP-7									0.5	2	VOCs, SVOCs, Pesticides, PCBs, Cyanide, Metals	TP-2B
TP-3/MIP-6	11					3.5	5.5	Cinders, burned rubbish (glass, cans, wires, etc.) and ash	0	0.5	VOCs, SVOCs, Pesticides, PCBs, Cyanide, Metals	TP-3A (BURN AREA)
TP-3/MIP-6									0.5	2	VOCs, SVOCs, Pesticides, PCBs, Cyanide, Metals	TP-3B (BURN AREA)
TP-3/MIP-6									4	5	VOCs, SVOCs, Pesticides, PCBs, Cyanide, Metals	TP-3C (BURN AREA)
TP-4/MIP-8	8					1	8	Cinders and other burned debris	0	0.5	VOCs, SVOCs, Pesticides, PCBs, Cyanide, Metals	TP-4A (BURN AREA)
TP-4/MIP-8									0.5	2	VOCs, SVOCs, Pesticides, PCBs, Cyanide, Metals	TP-4B (BURN AREA)
TP-5/MIP-4	8								0	0.5	VOCs, SVOCs, Pesticides, PCBs, Cyanide, Metals	TP-5A
TP-5/MIP-4									0.5	2	VOCs, SVOCs, Pesticides, PCBs, Cyanide, Metals	TP-5B
TP-6	8								0	0.5	VOCs, SVOCs, Pesticides, PCBs, Cyanide, Metals	TP-6A
TP-6									0.5	2	VOCs, SVOCs, Pesticides, PCBs, Cyanide, Metals	TP-6B
TP-7	4					0	2	Cinders and other burning dump debris	0	0.5	VOCs, SVOCs, Pesticides, PCBs, Cyanide, Metals	TP-7A (BURN AREA)
TP-7									0.5	2	VOCs, SVOCs, Pesticides, PCBs, Cyanide, Metals	TP-7B (BURN AREA)
TP-8/MIP-1	7.5-8.0								0	0.5	VOCs, SVOCs, Pesticides, PCBs, Cyanide, Metals	TP-8A
TP-8/MIP-1									0.5	2	VOCs, SVOCs, Pesticides, PCBs, Cyanide, Metals	TP-8B
TP-9/MIP-2	7.5-8.0											
TP-10/MIP-3	7.5-8.0											
TP-11A	12											
TP-11B	12			11.5	12+	6	8	Metal, glass, rebar, concrete	11	12	VOCs, SVOCs, Metals	CDL-TP11B-00-12
TP-11C	12	12	12	11	12+	6	8	Metal, 'waste layer'	11	12	VOCs, SVOCs, Metals	CDL-TP11C-00-12
TP-12A	11					0	9.5	Metal, bricks, glass				

Table 9-1: Observations and Soil Samples Collected During Environmental Investigations of the Construction Debris Landfill

Location	TD	Elevated PID (>25ppm)		Staining Observed		Waste Observed		Type of waste	Soil Sample Depth		Soil Sample Chemical Analysis	Soil Sample ID
	ft	from	to	from	to	from	to		from	to		
TP-12B	8					0	8	Metal, bricks, glass				
TP-12C	5					0	5+	Metal, bricks, glass	4.5	5	VOCs, SVOCs, Metals	CDL-TP12C-00-009
TP-13A	10	6	10+	6	10+							
TP-13B	5	3	5	3	5			FPH/NAPL*	4.5	5	VOCs, SVOCs, Metals	CDL-TP13B-00-010
TP-13C	5	2	4	2	5	1	2	Metal, terra cotta pipe	2.5	3	VOCs, SVOCs, Metals	CDL-TP13C-00-006
TP-14A	12											
TP-14B	12											
TP-14C	12											
TP-15A	5											
TP-15B	9					0	1	Concrete				
TP-15C	8					0	2	Scrap metal, bottle				
TP-16A	10											
TP-16B	11											
TP-16C	11											
TP-17A	10											
TP-17B	12					1	2	Metal				
TP-17C	12					1	2	Metal, plastic				
TP-18A	12					1	6	Metal, glass, debris				
TP-18B	12					1	6	Metal, glass	3.5	4	VOCs, SVOCs, Metals	CDL-TP18B-00-008
TP-18C	4.5					1.5	4.5	Metal, glass, debris				
TP-19A	6					1	6	Metal, glass	4	4.5	VOCs, SVOCs, Metals	CDL-TP19A-00-009
TP-19B	5					1	5	Metal, glass, bricks				
TP-19C	3.5					1	3.5	Metal, ceramics, glass				
TP-20A	4					3.5	4	Metal				
TP-20B	5.5					1.5	5	Blue material, black burn material, charred wood	4	4.5	VOCs, SVOCs, Dioxins/Furans, Metals	CDL-TP20B-00-010
TP-20C	1.5											
TP-21A	4.5					3	4.5	Metal, glass	3	3.5	VOCs, SVOCs, Metals	CDL-TP21A-00-007
TP-21B	4					1.5	4	Blue corroded material, glass			VOCs, SVOCs, Metals	
TP-21C	4.5					4	4.5+	Metal, glass, wood				
TP-22A	4					1	1	Blue corroded material				
TP-22B	3								1.5	2		CDL-TP22B-00-004
TP-22C	2											
TP-23A	10											
TP-23B	10											
TP-23C	10											
TP-24A	12	5.5	12+	6	12	11	12	Paint cans, paste material	5.5	6	VOCs, SVOCs, Metals	CDL-TP24A-00-012
TP-24B	12	6	10	6	12							
~~TP-24C~~												
TP-25A	17											
TP-25B	17											
TP-25C	17								16.5	17	VOCs, SVOCs, Metals	CDL-TP25C-00-034
TP-26A	15					4	8.5	Drum, tractor tire				
TP-26B	14					4	5.5	Drum, tractor tire	4	4.5	VOCs, SVOCs, Metals	CDL-TP26B-00-010
TP-26C	16			15	16	4.5	6	Metal, glass, drums				

Table 9-1: Observations and Soil Samples Collected During Environmental Investigations of the Construction Debris Landfill

Location	TD	Elevated PID (>25ppm)		Staining Observed		Waste Observed		Type of waste	Soil Sample Depth		Soil Sample Chemical Analysis	Soil Sample ID
	ft	from	to	from	to	from	to		from	to		
TP-27A	3					1	3	Metal, glass, springs	1.5	1.5	VOCs, SVOCs, Dioxins/Furans, Metals	CDL-TP27A-00-004
TP-27B	3											
TP-27C	4											
SB/MW-1	33								0	0.5	VOCs, SVOCs, Pesticides, PCBs, Cyanide, Metals	SB-1A
SB/MW-1									0.5	2	VOCs, SVOCs, Pesticides, PCBs, Cyanide, Metals	SB-1B
SB/MW-2	24								0	0.5	VOCs, SVOCs, Pesticides, PCBs, Cyanide, Metals	SB-2A
SB/MW-2									0.5	2	VOCs, SVOCs, Pesticides, PCBs, Cyanide, Metals	SB-2B
SB/MW-3	16					2	7.5	Debris, glass, concrete	0	0.5	VOCs, SVOCs, Pesticides, PCBs, Cyanide, Metals	SB-3A
SB/MW-3									0.5	2	VOCs, SVOCs, Pesticides, PCBs, Cyanide, Metals	SB-3B
SB/MW-4	17	9	14+	9	14				0	0.5	VOCs, SVOCs, Pesticides, PCBs, Cyanide, Metals	SB-4A
SB/MW-4									0.5	2	VOCs, SVOCs, Pesticides, PCBs, Cyanide, Metals	SB-4B
SB/MW-4									9	10	VOCs, SVOCs, Pesticides, PCBs, Cyanide, Metals	SB-4C
SB/MW-5	17					5.5	8	Debris, glass, concrete	0	0.5	VOCs, SVOCs, Pesticides, PCBs, Cyanide, Metals	SB-5A
SB/MW-5									0.5	2	VOCs, SVOCs, Pesticides, PCBs, Cyanide, Metals	SB-5B
SB/MW-6	24								0	0.5	VOCs, SVOCs, Pesticides, PCBs, Cyanide, Metals	SB-6A
SB/MW-6									0.5	2	VOCs, SVOCs, Pesticides, PCBs, Cyanide, Metals	SB-6B
DPT-1	20	6	16	15	17	5	5.8	Little debris	13	13.5	VOCs, SVOCs, Metals	CDL-DPS01-00-027
DPT-2	15					8	9.4	glass	9.5	10	VOCs, SVOCs, Metals	CDL-DPS02-00-020
DPT-3	10	7	8.5	7	10				4.5	5	VOCs, SVOCs, Metals	CDL-DPS03-00-010
DPT-4	15					3	7.9	Blue corroded material, metal, glass	7	7.5	VOCs, SVOCs, Metals	CDL-DPS04-00-15
DPT-5	20			14	15	14.5	16.5		12.5	13	VOCs, SVOCs, Metals	CDL-DPS05-00-026
DPT-6	15					0	1.3	fill/waste	6.5	7	VOCs, SVOCs, Metals	CDL-DPS06-00-014
DPT-7	15					0	1	fill/waste	0.5	1	VOCs, SVOCs, Metals	CDL-DPS07-00-002
DPT-8	15					3.3	7	fill*/burnt cinders	4.5	5	VOCs, SVOCs, Metals	CDL-DPS08-00-010
DPT-9	25								17	17.5	VOCs, SVOCs, Metals	CDL-DPS09-00-035
DPT-10	15								7.5	8	VOCs, SVOCs, Metals	CDL-DPS10-00-016
DPT-11	15					2.2	3.2		9	9.5	VOCs, SVOCs, Metals	CDL-DPS11-00-019
DPT-12	20											
DPT-13	25	18	18.5	17	19							
DPT-14	20	14	20+	13.2	20							
DPT-15	20											
DPT-16	15											

Table 9-1: Observations and Soil Samples Collected During Environmental Investigations of the Construction Debris Landfill

Location	TD	Elevated PID (>25ppm)		Staining Observed		Waste Observed		Type of waste	Soil Sample Depth		Soil Sample Chemical Analysis	Soil Sample ID
	ft	from	to	from	to	from	to		from	to		
DPT-17	12											
DTW-1	68											
DTW-2	60											
DTW-3	55											
DTW-4	60											

analysis. The chromatograph produced by the analysis appeared to be weathered JP-5 aviation fuel with a significant number of the lower boiling point hydrocarbons missing. The results of the MADEP method also suggest that the contamination is a distillate fuel.

2004 Soil Gas Investigation (USACE): Soil gas samples were collected across the Site and measured elevated levels of benzene, toluene, ethylbenzene, and xylene (BTEX) as well as chlorinated solvents (defined in the survey as PCE, TCE and DCE). BTEX compounds were found in heaviest concentrations on the eastern edge of the Site. Chlorinated solvents were found in heaviest concentrations in soil gas in the eastern and southeastern portions of the Site.

2007 Remedial Investigation (Versar, 2011): Remedial Investigation (RI) activities included: electromagnetic and ground penetrating radar geophysical surveys; membrane interface probe (MIP) survey; excavating, logging, and soil sampling of 10 test pits; installing and sampling six shallow monitoring wells and collecting two rounds of groundwater samples; collecting 10 sediment samples and 10 surface water samples; and measuring water levels, hydraulic gradients, and hydraulic conductivity of the shallow surficial aquifer. The geophysical investigation identified five anomalies located in either the Central or Southern AOCs. Elevated detector values were found in one of the eight MIP borings. No waste was encountered in the test pits located in the Northern or Western AOCs. Surface soil from the Northern and Western AOCs did not contain any organic compounds above background. Sample locations are shown on **Figure 9-3**. Significant findings from the RI are included in **Table 9-1**.

Draft Feasibility Study (Versar 2012). Additional information and analytical data were needed to allow a refinement of the remedial alternatives presented in the draft Feasibility Study (FS), so an SFI was completed. Data from the RI also informed the planning of the SFI field activities and are used with the SFI data to refine the Conceptual Site Model (CSM).

2022 Supplemental Field Investigation (SFI) (AECOM, 2022): Field activities included trenching, collecting soil samples from trenches and from DPT borings, sampling shallow and deep temporary wells, collecting two rounds of surficial aquifer groundwater samples from monitoring wells, collecting a Yorktown Aquifer groundwater sample from water supply well TOC-06, and collecting pore water and sediment samples from Little Mosquito Creek. A 3-D model was used to calculate the volume of waste debris (7,450 cubic yards) and the volume of stained sand (8,065 cubic yards). Sample locations are shown on **Figure 9-3**. Significant findings from the SFI are included in **Table 9-1**.

2022 Human Health Risk Assessment and Ecological Risk Assessment as part of SFI: The human health risk assessment (HHRA), completed for the SFI report, indicated that the Reasonable Maximum Exposure (RME) and Central Tendency Exposure (CTE) results for the Upgradient Drainage and Downgradient/Estuarine exposure areas have cumulative Excess Lifetime Cancer Risk (ELCR) and Hazard Index (HI) results below the USEPA target levels and are eliminated from further evaluation. The carcinogenic risk and/or noncarcinogenic hazard results for some receptors at the Central/ Southern (C/S) and Northern/Western (N/W) exposure areas either equaled or were above EPA target cumulative levels for exposure to soil and/or vapor intrusion (VI) from groundwater. There are no soil-related risks at the N/W exposure area. Chemicals of Concern for ingestion or dermal contact with soil or inhalation of vapors from soil are 1,1,2-trichloroethane, 2-hexanone, benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, dibenz(a,h)anthracene, indeno(1,2,3-cd)pyrene, 2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD) toxicity equivalence (TEQ), aluminum, arsenic, cadmium (diet), chromium (total), cobalt, copper, iron, manganese, thallium, and vanadium. The Integrated Exposure Uptake Biokinetic (IEUBK) lead results were above the 5% probability thresholds for soil.

The Screening Level Ecological Risk Assessment (SLERA) and Baseline Ecological Risk Assessment (BERA) Step 3a evaluation indicates a potential for adverse ecological effects for Central AOC soil macroinvertebrates (nickel) and terrestrial wildlife (nickel); and Southern AOC soil macroinvertebrates

(barium, chromium, copper, manganese, mercury, and zinc) and terrestrial wildlife (total DDD/DDE/DDT and total TEQ [mammal]). The contaminants of Potential Ecological Concern (COPEC) in soil are barium, chromium, copper, manganese, mercury, nickel, and zinc. The COPECs in sediment are bis(2-ethylhexyl) phthalate, di-n-octyl phthalate, total DDD/DDE/DDT, total TEQ (dioxin/furan congener concentration), aluminum, and sodium.

2021 Site Background Soil and Groundwater Investigation for The Main Base (Tetra Tech, 2021): This study provided data to represent background conditions at WFF. Data from the two types of soils found at the Main Base—Molena and Bojac Series—were evaluated. The Molena soils are found on the Project 15 CDL. The constituent concentrations in surface and subsurface soil within each series were found to be statistically distinct. Groundwater samples were collected from only the shallowest water-bearing zone, the surficial aquifer. The Background Study also found that groundwater samples collected via low-flow sampling techniques provide a better representation of groundwater conditions. The Background Study identified Background Threshold Values (BTVs) for soil.

2022 Engineering Evaluation / Cost Analysis (AECOM, 2022b): An Engineering Evaluation / Cost Analysis (EE/CA) was completed because “Unacceptable risks associated with the Central and Southern [Areas of Concern] are present at the site. Solid waste is clearly evident at the surface and persists to a depth of at least 13 feet in some areas” (United States [U.S.] Army Corps of Engineers [USACE], 2011). Action is necessary to address the potential for direct contact with exposed landfill wastes by human and ecological receptors. NASA is the lead Agency for WFF, under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), and has determined that a Non-Time-Critical Removal Action (NTCRA) is necessary because waste is exposed at or near the surface of the CDL.

The following removal action objective (RAO) was developed for the Project 15 CDL site: reduce or eliminate direct exposures to waste and Preliminary Constituents of Concern in soil by human and ecological receptors as well as mitigate transport of these Chemicals of Concern to surface water or sediment receptors. Based on a comparison of effectiveness, implementability, and cost, the recommended alternative for the NTCRA for the CDL is the excavation and off-site disposal of waste and affected soil.

Preliminary remediation goals (PRGs) and Ecological screening values (ESVs) were presented in the EE/CA (AECOM, 2022b). **Table 9-2** presents the Residential PRGs, Industrial PRGs, and ESVs for soil. **Figure 9-4** shows the locations of Industrial PRG exceedances in soil. **Table 9-3** presents all soil samples collected at the CDL and summarizes if there was any compound in the sample that exceeded an industrial or residential PRG for soil. Although remediation will be to industrial PRG standards, residential PRGs are included in **Table 9-3** in order to present a complete picture of current environmental conditions at the CDL.

Table 9-2: Summary of PRGs and ESVs for Soil

Compound (values are in mg/kg)	Residential PRG Surface Soil	Residential PRG Subsurface Soil	Residential PRG for Total Soil	Industrial Soil PRGs	ESV Surface Soil	Value for subsurface soil assessment
1,1,2-Trichloroethane	0.12	0.12	0.12	2.1		2.1
2,3,7,8-TCDD TEQ	2.60E-05	2.60E-05	2.60E-05			
2-Hexanone	29	29	29	433.3		433.3
Benzo(a)pyrene	1	1	1	52.5		52.5
Aluminum	13900	22400	13900			
Antimony	2.4	2.4	2.4			
Arsenic	13.72	5.3	5.3	75		5.3
Barium					330	
Cadmium	24	24	24			
Chromium	18.4	21	18.4	157.5	57	157.5
Cobalt	7.3	8.8	7.3		13	
Copper	1033.3333	1033.3333	1033		80	
Iron	18333	24200	18333			
Lead						
Manganese	781	257	257	8667	450	8667
Mercury					0.5	
Nickel	125	125	125		280	
Thallium	0.76	0.26	0.26			
Vanadium	32.5	42	33			
Zinc					153	
Total DDD/DDE/DDT					0.044	
Total HMW PAHs					18	

Legend:
All values in mg/kg
mg/kg = milligrams per kilogram
ESV = Ecological Screening Value
PRG = Preliminary Remediation Goal from Table 3-1 of the EE/CA (AECOM, 2022)
Total DDD/DDE/DDT is the summation of BTVs for the individual compounds

Table 9-3 : Previous Soil Samples and PRG Exceedances

Location	Soil Sample ID	Soil Sample Depth		Soil Sample Chemical Analysis	PRG or ESV exceedence
		from	to		
TP-1/MIP-5	TP-1A (BURN AREA)	0	0.5	V, S, Pe, P, C, M	
TP-1/MIP-5	TP-1B (BURN AREA)	0.5	2	V, S, Pe, P, C, M	
TP-1/MIP-5	TP-1C (BURN AREA)	2	8.5	V, S, Pe, P, C, M	
TP-2/MIP-7	TP-2A	0	0.5	V, S, Pe, P, C, M	
TP-2/MIP-7	TP-2B	0.5	2	V, S, Pe, P, C, M	
TP-3/MIP-6	TP-3A (BURN AREA)	0	0.5	V, S, Pe, P, C, M	residential PRG exceedances of metals and ESV exceedances of metals and pesticides
TP-3/MIP-6	TP-3B (BURN AREA)	0.5	2	V, S, Pe, P, C, M	residential PRG exceedances of metals and ESV exceedances of metals and pesticides
TP-3/MIP-6	TP-3C (BURN AREA)	4	5	V, S, Pe, P, C, M	residential PRG exceedances of metals
TP-4/MIP-8	TP-4A (BURN AREA)	0	0.5	V, S, Pe, P, C, M	residential PRG exceedances of metals, ESV exceedances of metals and pesticides
TP-4/MIP-8	TP-4B (BURN AREA)	0.5	2	V, S, Pe, P, C, M	residential PRG exceedances of metals
TP-5/MIP-4	TP-5A	0	0.5	V, S, Pe, P, C, M	
TP-5/MIP-4	TP-5B	0.5	2	V, S, Pe, P, C, M	
TP-6	TP-6A	0	0.5	V, S, Pe, P, C, M	
TP-6	TP-6B	0.5	2	V, S, Pe, P, C, M	residential PRG exceedances of metals
TP-7	TP-7A (BURN AREA)	0	0.5	V, S, Pe, P, C, M	residential PRG exceedances of metals, ESV exceedances of metals and pesticides.
TP-7	TP-7B (BURN AREA)	0.5	2	V, S, Pe, P, C, M	Industrial and residential PRG exceedances of metals, ESV exceedances of metals and pesticides.
TP-8/MIP-1	TP-8A	0	0.5	V, S, Pe, P, C, M	
TP-8/MIP-1	TP-8B	0.5	2	V, S, Pe, P, C, M	
TP-11B	CDL-TP11B-00-12	11	12	V, S, M	
TP-11C	CDL-TP11C-00-12	11	12	V, S, M	
TP-12C	CDL-TP12C-00-009	4.5	5	V, S, M	residential PRG exceedances of metals and SVOCs.
TP-13B	CDL-TP13B-00-010	4.5	5	V, S, M	Industrial and residential PRG exceedances of VOCs.
TP-13C	CDL-TP13C-00-006	2.5	3	V, S, M	Industrial and residential PRG exceedances of VOCs.
TP-18B	CDL-TP18B-00-008	3.5	4	V, S, M	residential PRG exceedances of metals.
TP-19A	CDL-TP19A-00-009	4	4.5	V, S, M	residential PRG exceedances of metals.
TP-20B	CDL-TP20B-00-010	4	4.5	V, S, D, M	residential PRG exceedances of metals.
TP-21A	CDL-TP21A-00-007	3	3.5	V, S, M	
TP-22B	CDL-TP22B-00-004	1.5	2	V, S, M	
TP-24A	CDL-TP24A-00-012	5.5	6	V, S, M	residential PRG exceedances of metals
TP-25C	CDL-TP25C-00-034	16.5	17	V, S, M	
TP-26B	CDL-TP26B-00-010	4	4.5	V, S, M	residential PRG exceedances of metals and SVOCs.
TP-27A	CDL-TP27A-00-004	1.5	1.5	V, S, D, M	residential metals and dioxin PRG exceedances and ESV exceedances of metals
SB/MW-1	SB-1A	0	0.5	V, S, Pe, P, C, M	
SB/MW-1	SB-1B	0.5	2	V, S, Pe, P, C, M	
SB/MW-2	SB-2A	0	0.5	V, S, Pe, P, C, M	
SB/MW-2	SB-2B	0.5	2	V, S, Pe, P, C, M	
SB/MW-3	SB-3A	0	0.5	V, S, Pe, P, C, M	
SB/MW-3	SB-3B	0.5	2	V, S, Pe, P, C, M	
SB/MW-4	SB-4A	0	0.5	V, S, Pe, P, C, M	residential PRG exceedances of SVOCs and metals, and ESV exceedances of PAHs and metals.
SB/MW-4	SB-4B	0.5	2	V, S, Pe, P, C, M	
SB/MW-4	SB-4C	9	10	V, S, Pe, P, C, M	
SB/MW-5	SB-5A	0	0.5	V, S, Pe, P, C, M	
SB/MW-5	SB-5B	0.5	2	V, S, Pe, P, C, M	
SB/MW-6	SB-6A	0	0.5	V, S, Pe, P, C, M	
SB/MW-6	SB-6B	0.5	2	V, S, Pe, P, C, M	residential PRG exceedances of metals.
DPT-1	CDL-DPS01-00-027	13	13.5	V, S, M	
DPT-2	CDL-DPS02-00-020	9.5	10	V, S, M	residential PRG exceedances of metals.
DPT-3	CDL-DPS03-00-010	4.5	5	V, S, M	
DPT-4	CDL-DPS04-00-15	7	7.5	V, S, M	residential PRG exceedances of metals, ESV exceedences of metals and pesticides.
DPT-5	CDL-DPS05-00-026	12.5	13	V, S, M	
DPT-6	CDL-DPS06-00-014	6.5	7	V, S, M	
DPT-7	CDL-DPS07-00-002	0.5	1	V, S, M	residential PRG exceedances of metals and ESV exceedance of metals.
DPT-8	CDL-DPS08-00-010	4.5	5	V, S, M	residential PRG exceedances of metals.
DPT-9	CDL-DPS09-00-035	17	17.5	V, S, M	
DPT-10	CDL-DPS10-00-016	7.5	8	V, S, M	
DPT-11	CDL-DPS11-00-019	9	9.5	V, S, M	

*PRG - Preliminary Remediation Goals

Legend

Below residential PRGs

Above residential PRGs but below industrial PRGs

Above industrial PRGs

0.5 1 Surface soil sample

V VOCs

S SVOCs

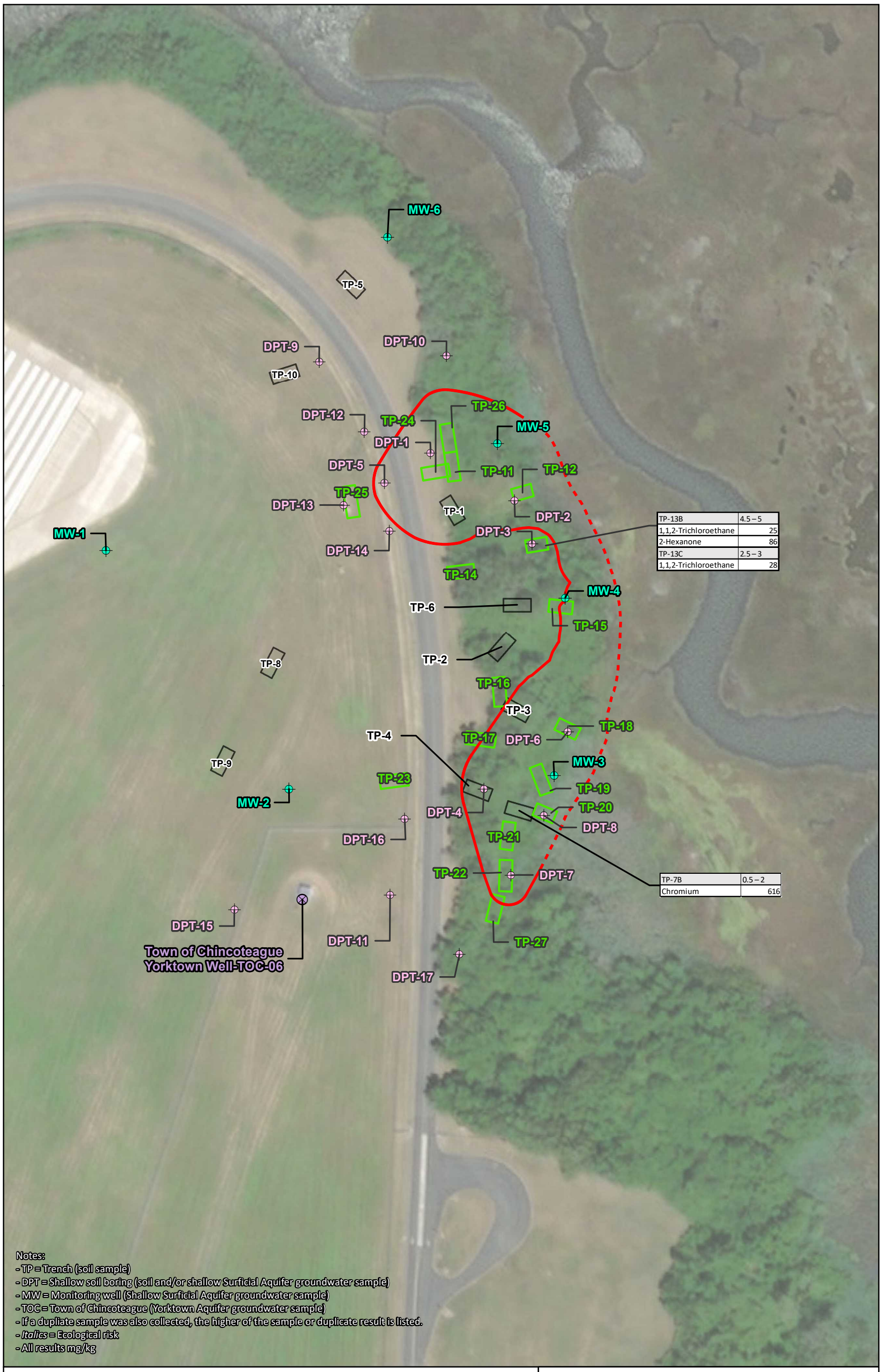
M Metals

Pe Pesticides

P PCBs

C Cyanide

D Dioxins/Furans



Industrial PRG Exceedances

CLIENT	NASA WFF CDL SFI & NTCRA			
PROJECT	Supplemental Field Investigation			
REVISED	2/22/2023	GIS BY	MS	2/22/2023
SCALE	1:1,200	CHK BY	JK	2/22/2023
Base Map: Source: Esri, Maxar, Earthstar Geographics, and the GIS User Community		PM	JK	2/22/2023

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12420 Milestone Center Drive
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Figure 9-4

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SAP Worksheet #10 Project Quality Objectives/Systematic Planning Process Statements

(UFP-QAPP Manual Section 2.6.1- Worksheet #10)

10.1 PROBLEM STATEMENT

Debris and affected soil are present at the CDL. NASA will excavate both and dispose of them offsite. Additional sampling is required pre-excavation to characterize the waste and soil for disposal; post-excavation sampling will not be conducted, sufficient pre-excavation soil samples will be collected and analyzed to assess the limits of excavation and to assess the native soil that will remain.

10.2 INFORMATION INPUTS

Based on field observations made during the RI and SFI, debris is generally buried under clean fill. There are locations where waste is observed near the surface (**Figure 10-1**).

Within the Central AOC, waste is found at the surface but is also buried beneath as much as 11 feet of sand fill. The waste includes empty metal drums, paint, scrap metal, rebar, glass bottles, bricks, cable, springs, tires, and broken concrete. The maximum depth of waste in the Central AOC was about 13 feet bgs. Using the ESVs and industrial PRGS, overburden soil in the Central AOC can be reused as backfill.

Waste in the Southern AOC is found both at the surface and buried beneath a layer of sand fill up to 4 feet deep. Waste in the Southern AOC consists of cinders and burned rubbish (glass, cans, wires) (Versar, 2011), as well as plastic, plates, scrap metal, bathroom fixtures, railroad ties, and metal pipes. The maximum thickness of waste found during the SFI was 5 feet. Based on the PRGS and ESVs, overburden soil in the Southern AOC will be properly disposed offsite.

10.3 STUDY BOUNDARIES

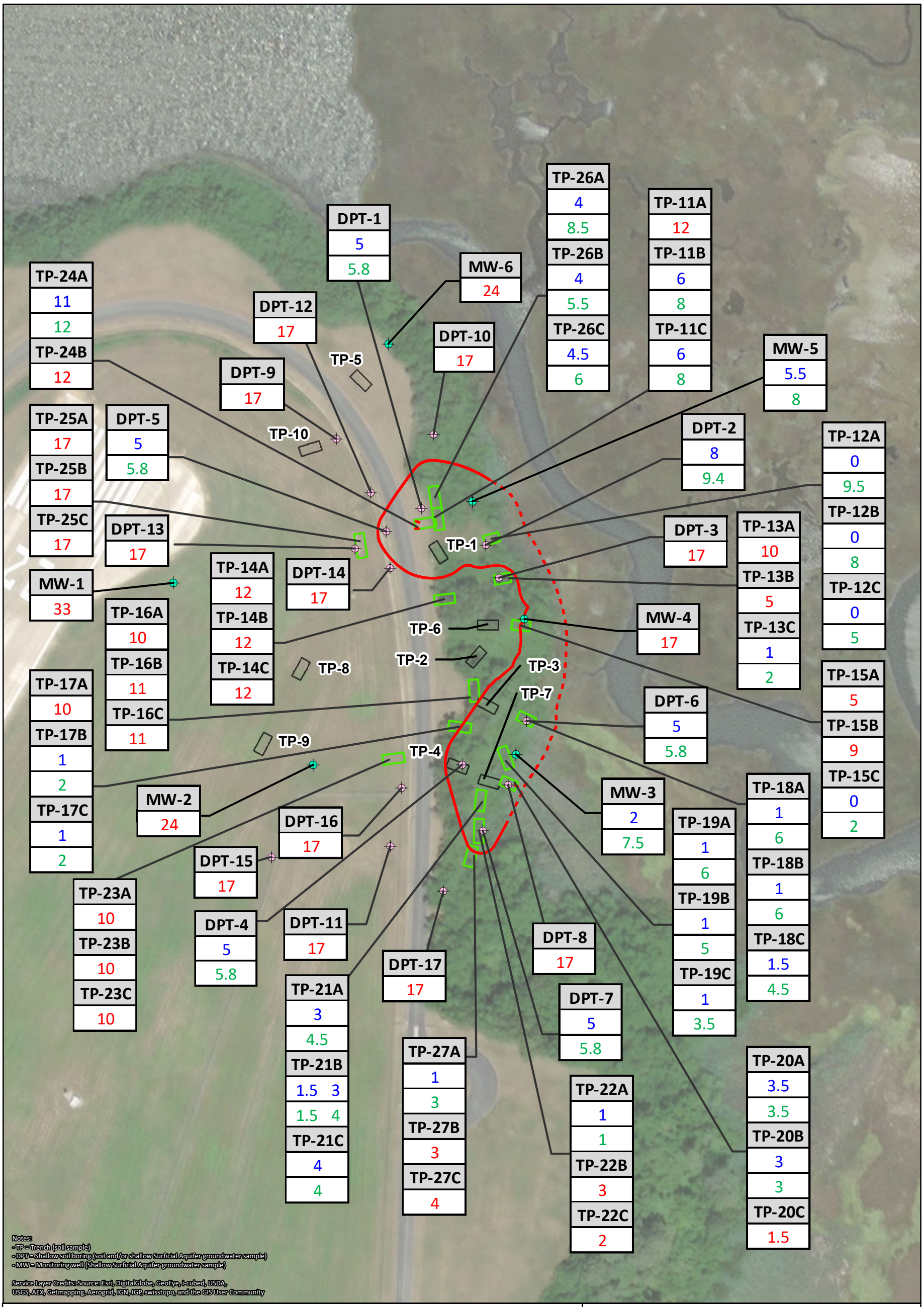
The boundary of the CDL based on field observations is depicted on **Figure 10-1**. The extent of the excavation will include all accessible waste and overburden soil and will not extend under the roadway. The proposed extent of the excavation is depicted on **Figure 10-2**.

10.4 ANALYTIC APPROACH

The analytical approach consists of pre-excavation sampling.

10.4.1 Pre-Excavation Characterization Sampling

Prior to excavation activities, AECOM personnel will mobilize to the site to collect samples of the soil/waste for characterization for disposal and to verify that the waste is non-hazardous. Based on landfill analytical requirements, TCLP samples will be collected at the rate of one sample per 1,000 tons (approximately 1,400 cubic yards [yds3]) and total sulfur samples will be collected for material that contains ash (the southern portion of the excavation). No fly ash was observed on the northern half of the site. Each characterization sample will be a composite of several samples identified in **Table 10-1**. The characterization samples will be collected from the middle of the soil core at each location, i.e., if the soil core is ten feet, the characterization sample will be collected from a depth of 4-6 feet. This ensures that the characterization sample will be composed of waste and material that will be shipped to the landfill and will not impede the pre-characterization sampling at depth, which is meant to assess the soil that will remain.



Legend

- Groundwater Monitoring Well
- Shallow DPT Boring
- Supplemental Field Investigation Trench
- Previous Investigation Trench
- Extent of Waste (dashed where estimated)

Location ID
Top of Waste (ft)
Bottom of Waste (ft)
Total Depth (ft)

Note: All previous investigation trenches (TP-1 - TP-10) do not have soil boring logs

0 100 200 400 Feet

N

Source: SFI Appendix A: Trench and Direct Push Technology Boring Logs, 2015-2016

Extent of Waste Based on Field Observations

CLIENT	NASA WFF CDL SFI & NTCRA			
PROJECT	Supplimental Field Investigation			
REVISED	2/16/2022	GIS BY	EG	2/16/2022
SCALE	1:1,560	CHK BY	MS	2/16/2022
Base Map: Source: Esri, Maxar, GeoEye, Earthstar Geographics, CNES/Airbus DS, USDA, USGS, AeroGRID, IGN,		PM	JK	2/16/2022

AECOM 12420 Milestone Center Drive Germantown, MD 20876

Figure 10-1

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Legend

Extent of Waste

Wetland

N

0 30 60 120

Feet

Proposed Excavation Area, Construction Debris Landfill

CLIENT	NASA WFF CDL SFI & NTCRA			
PROJECT	Supplemental Field Investigation			
REVISED	10/5/2022	GIS BY	MS	10/5/2022
SCALE	1:720	CHK BY	JK	10/5/2022
Base Map: Source: Esri, Maxar, Earthstar Geographics, and the GIS User Community		PM	JK	10/5/2022

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Figure 10-2

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Table 10-1: Proposed Pre-Excavation Soil Sample Locations

Sample location	Depth of Direct Push core	Sample number	Depth of sample (ft bgs)	Sample number	Depth of sample (ft bgs)	Sample number	Depth of sample (ft bgs)	Metals	VOC	SVOC	Dioxins	Composite middle of core for analysis
Bottom of Excavation Samples												
B01	15	B01A	6	B01B	8	B01C	10	X				C1 for TCLP and Total Sulfur
B02	15	B02A	6	B02B	8	B02C	10	X				
B03	15	B03A	6	B03B	8	B03C	10	X				
B04	15	B04A	6	B04B	8	B04C	10	X				
B05	15	B05A	6	B05B	8	B05C	10	X				C2 for TCLP and Total Sulfur
B06	15	B06A	8	B06B	10	B06C	12	X				
B07	15	B07A	6	B07B	8	B07C	10	X				
B08	10	B08A	6	B08B	8	B08C	10	X				
B09	10	B09A	8	B09B	10	B09C	12	X				
B10	10	B10A	5	B10B	7	B10C	9	X				C3 for TCLP and Total Sulfur
B11	10	B11A	5	B11B	7	B11C	9	X				
B12	10	B12A	5	B12B	7	B12C	9	X				
B13	10	B13A	5	B13B	7	B13C	9	X				
B14	10	B14A	5	B14B	7	B14C	9	X				
B15	10	B15A	2	B15B	4	B15C	6	X				C4 for TCLP and Total Sulfur
B16	10	B16A	2	B16B	4	B16C	6	X				
B17	10	B17A	2	B17B	4	B17C	6	X				
B18	10	B18A	2	B18B	4	B18C	6	X		X		
B19	10	B19A	2	B19B	4	B19C	6	X		X		
B20	10	B20A	2	B20B	4	B20C	6	X				
B21	15	B21A	10	B21B	12	B21C	14	X				C5 for TCLP
B22	15	B22A	10	B22B	12	B22C	14	X				
B23	15	B23A	10	B23B	12	B23C	14	X				
B24	15	B24A	10	B24B	12	B24C	14	X				C6 for TCLP
B25	15	B25A	10	B25B	12	B25C	14	X				
B26	15	B26A	6	B26B	8	B26C	10	X		X		
B27	20	B27A	13	B27B	15	B27C	17	X				C7 for TCLP
B28	20	B28A	12	B28B	14	B28C	16	X				
B29	15	B29A	8	B29B	10	B29C	12	X				
B30	15	B30A	8	B30B	10	B30C	12	X				
B31	15	B31A	6	B31B	8	B31C	10	X				C8 for TCLP
B32	15	B32A	6	B32B	8	B32C	10	X				
B33	15	B33A	6	B33B	8	B33C	10	X				
B34	15	B34A	6	B34B	8	B34C	10	X				
Sidewall Samples												
S01	5	S01A	3	S01B	3	S01C	3	X			X	
S02	5	S02A	3	S02B	3	S02C	3	X				
S03	5	S03A	2	S03B	2	S03C	2	X				
S04	5	S04A	1	S04B	1	S04C	1	X				
S05	5	S05A	1	S05B	1	S05C	1	X	X			
S06	5	S06A	5	S06B	5	S06C	5	X				
S07	5	S07A	5	S07B	5	S07C	5	X				
S08	5	S08A	5	S08B	5	S08C	5	X				
S09	5	S09A	5	S09B	5	S09C	5	X				
S10	5	S10A	4	S10B	4	S10C	4	X				
								44	1	3	1	

Number of samples = 132 8

Previous Sample Location	Total Depth (Ft.)	Waste Observed		Soil Sample Chemical Analysis	Soil Sample Depth (ft bgs)	Date drilled or excavated	Industrial PRG Exceedances	Residential PRG Exceedances	ESV Exceedances
	ft	from	to						
DPT-7	15	0	1	V, S, M	0.5-1.0	7/27/2015		Al, Sb, Cr, Cu	Cu, Zn
TP-21	4	1.5	4	V, S, M	3-305	6/9/2015			
DPT-4	15	3	7.9	V, S, M	7.0-7.5	7/23/2015		Sb, As, Cd, Cr, Co, Fe, Mg	
TP-7	4	0	2	V, S, Pe, P, C, M	0-2.0	5/29/2015	Cr	Al, Sb, As, Cr, Cu, Ni	Total DDD/DDE/DDT, Cr, Cu, Zn
DPT-8	15	3.3	7	V, S, M	4.5-5.0	7/23/2015		Al, Sb, As, Cd, Cr, Co, Cu, Fe, Mg	
DPT-4	15	3	7.9	V, S, M	7.0-7.5	7/23/2015		Sb, As, Cd, Cr, Co, Fe, Mg	
TP-19A	6	1	6	V, S, M	4.0-4.5	6/9/2015		Sb, As, Cr, Co, Fe, Mg	
TP-3/MIP-6	11	3.5	5.5	V, S, Pe, P, C, M	4.0-5.0	4/9/2007		Al, Sb, As, Cd, Cr, Co, Cu, Fe, Mg, V	Total DDD/DDE/DDT, Ba, Cu, Mg, Hg, Zn
TP-18B	12	1	6	V, S, M	3.5-4.0	6/9/2015		Sb, As, Cr, Co, Cu, Fe, Mg	
SB/MW-4	17			V, S, Pe, P, C, M	9.0-10.0	4/18/2007		BaP, Cr, Co, Fe, Ni	Cr, Co, Ni, Total HMW PAHs
SB/MW-4	17			V, S, Pe, P, C, M	9.0-10.0	4/18/2007		BaP, Cr, Co, Fe, Ni	Cr, Co, Ni, Total HMW PAHs
TP-1/MIP-5	13	8.5	8.5	V, S, Pe, P, C, M	2.0-8.5	5/29/2015			
DPT-2	15	8	9.4	V, S, M	9.5-10.0	7/22/2015		Sb, Cr	
TP-1/MIP-5	13	8.5	8.5	V, S, Pe, P, C, M	2.0-8.5	5/29/2015			
TP-12	5	0	5+	V, S, M	4.5-5.0	6/2/2015		Sb, As, BaP	
DPT-1	20	5	5.8	V, S, M	13.0-13.5	7/21/2015			
TP-11	12	6	8	V, S, M	12	5/28/2015			
SB/MW-5	17	5.5	8	V, S, Pe, P, C, M	0.5-2.0	4/17/2007			
SB/MW-5	17	5.5	8	V, S, Pe, P, C, M	0.5-2.0	4/17/2007			
TP-26	14	4	5.5	V, S, M	4.0-4.5	5/11/2016		As, BaP, Cr, Co, Fe	
SB/MW-5	17	5.5	8	V, S, Pe, P, C, M	0.5-2.0	4/17/2007			
TP-27	3	1	3	V, S, D, M	1.5-1.5	5/10/2016		TCDD, Sb, As, Cr, Co, Fe, Mg	Ba, Cr, Co, Cu, Mg, Zn
TP-4/MIP-8	8	1	8	V, S, Pe, P, C, M	0.5-2.0	4/9/2007		Sb, Cr	Total DDD/DDE/DDT, Cu, Zn
TP-3/MIP-6	11	3.5	5.5	V, S, Pe, P, C, M	4.0-5.0	4/9/2007		Al, Sb, As, Cd, Cr, Co, Cu, Fe, Mg, V	Total DDD/DDE/DDT, Ba, Cu, Mg, Hg, Zn
SB/MW-4	17			V, S, Pe, P, C, M	9.0-10.0	4/18/2007			
TP-13	5			V, S, M	4.5-5.0	6/4/2015	1,1,2-TCA, MBK	1,1,2-TCA, MBK	
TP-24	12	11	12	V, S, M	5.5-6.0	6/10/2015		As	

V - VOCs
S - SVOCs
M - Metals
Pe - Pesticides
P - PCBs
C - Cyanide
D - Dioxins/Furans

Al - Aluminum
Sb - Antimony
As - Arsenic
Ba - Barium
BaP - Benzo(a)pyrene
Cd - Cadmium
Cr - Chromium
Co - Cobalt
Cu - Copper
MBK - 2-Hexanone
Fe - Iron

Mn - Manganese
Hg - Mercury
Ni - Nickel
TCDD - 2,3,7,8-TCDD TEQ
1,1,2-TCA - 1,1,2-Trichloroethane
V - Vanadium
Zn - Zinc

Several locations will be composited, i.e., locations B1, B2, B3, and B4 will be composited as sample C1, which will be analyzed for TCLP VOCs, SVOCs, and metals, and reactivity, ignitability, and corrosivity.

Some of the landfill waste in the southern portion of the site may be below the water table, making the waste wet and requiring dewatering prior to transportation and disposal. Any water from dewatering would be considered IDW and will be containerized, sampled, and disposed offsite. The sampling will adhere to the requirements of the disposal facility.

In the northern portion of the excavation, the overburden varies from zero feet to 14.5 feet thick and the volume is approximately 2,800 yds3. The volume of waste is approximately 5,600 yds3, therefore, four composite samples of the waste in the northern portion will be collected and analyzed for TCLP VOCs, SVOCs, and metals, and reactivity, ignitability, and corrosivity. **Table 10-1** presents the proposed pre-excavation sample numbers.

Besides sampling for TCLP, the pre-excavation sampling will include the collection and analysis of samples from beneath the waste and along the sidewalls of the excavation. Sufficient pre-excavation soil samples will be collected and analyzed to assess the limits of excavation, thereby, eliminating the need for post-excavation sampling. Although remediation will be to the industrial PRGs, there are only two previous sample locations where industrial PRGs were exceeded. Therefore, residential PRG exceedances will be relied upon to determine pre-excavation sample analysis. Since metals exceed a residential PRG at every location that has a residential PRG exceedance (see **Figure 9-4**), every pre-excavation sample will be analyzed for metals, in order to assess the soil that will remain in place. PRG exceedances of VOCs, and/or SVOCs, and/or dioxins occur at a subset of locations that have a PRG exceedance; pre-excavation sampling at those locations will include analysis for VOCs, SVOCs, and/or dioxins, as appropriate (**Table 10-1**). Analytical results will be compared to the industrial PRGs and a decision will be made regarding the extent of the excavation. The ESVs will not be used as a comparison since the ESVs are applicable to surface soils and the pre-excavation soil will be sampled at depth. This pre-excavation sampling approach also allows accurate calculations of the volume of soil to be disposed and the volume of clean soil required prior to excavation. These pre-excavation samples will be collected at a rate of one sample per 1,000 square feet (ft2) of area (bottom and sidewalls). **Table 10-1** and **Figure 10-3** presents the proposed pre-excavation soil sampling locations. **Figure 10-4** combines the locations of all past soil samples and the proposed pre-excavation sample locations on one figure to show the overlap between the sampling events. Note that location TP-13, which reported compounds exceeding the industrial PRGs, is outside the outline of the buried debris on multiple figures, but pre-excavation sample location S05 is located west of TP-13. Therefore, the excavation will extend to at least S05 and include sample location TP-13 and surrounding soils.

Bottom samples will be collected at the estimated depth of waste, based on prior test pits and soil borings. Because it is unknow if any sample will exceed a PRG, an additional sample will be collected 5 feet deeper than the initial sample at each bottom location. Each soil core will be assessed, and if waste appears to be deeper than the targeted depth at a location, the depth of soil sample will be adjusted to the bottom of observed waste (sample A in **Table 10-1**) and 5 feet below that (sample B in **Table 10-1**). The square foot area of sidewalls varies, since the depth of waste and thus, the depth of excavation varies throughout the site. Along the western sidewall, going from south to north, the depth of excavation varies from less than 6 feet to 2 feet to 12 feet to 8 feet. In order to collect one sample per 1,000 ft2 of area of sidewall, the distance between samples can vary from every 500 linear feet to every 83 linear feet (see **Table 10-1**). Since the topography slopes to the east, to the marsh, and there is no waste in or under the marsh, the excavation will stop at, or before the marsh and there will be no sidewall on the eastern edge of the excavation; therefore, there will be no pre-excavation sampling along the eastern edge of the excavation, except for any bottom samples that might end up near the edge of the marsh. Similar to the bottom samples, it is

Legend

- Bottom Sample
- Sidewall Sample
- 32ft x 32ft Grid
- Extent of Waste
- Wetland

N

0 30 60 120 Feet

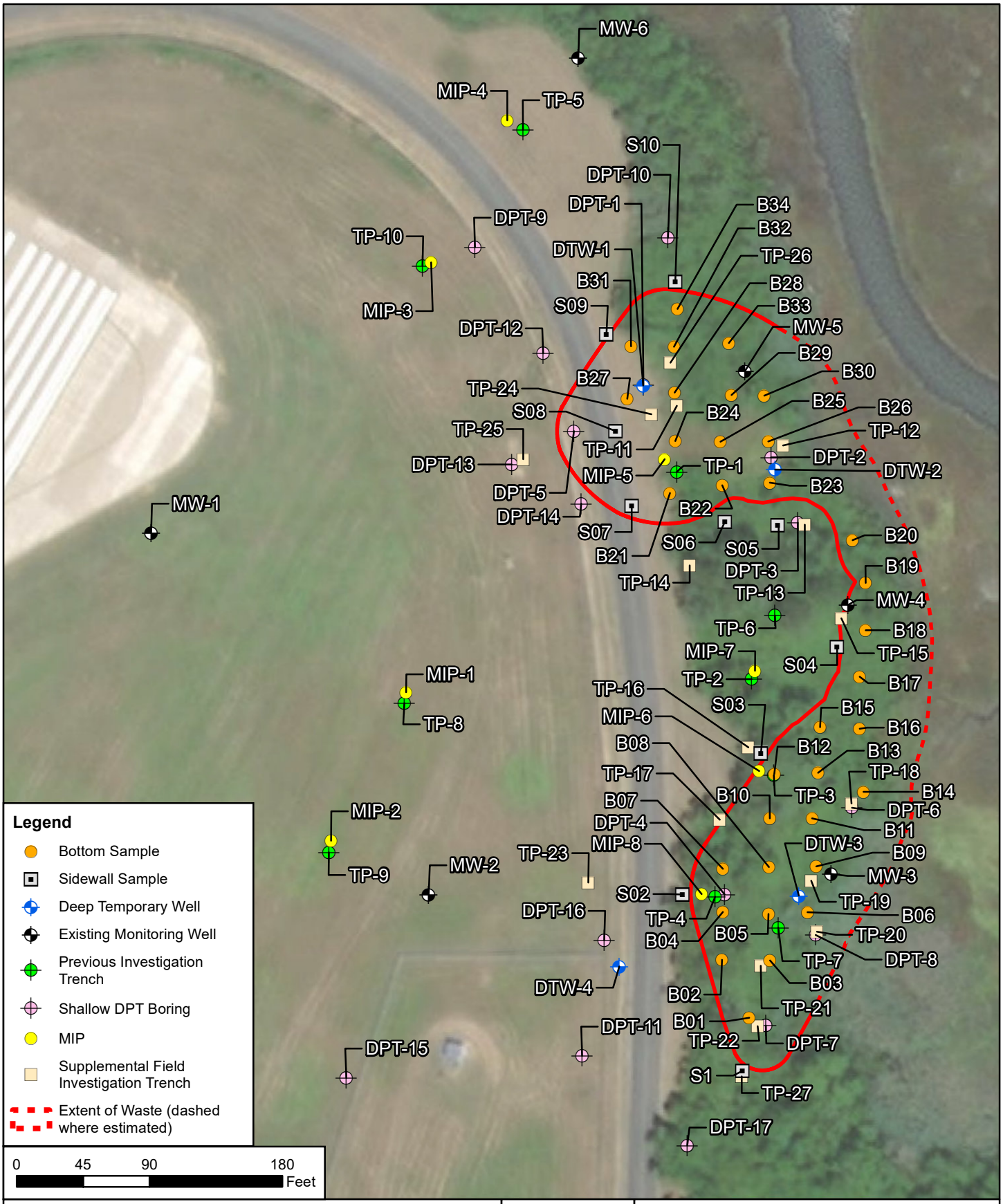
Proposed Pre-Excavation Sample Locations

CLIENT	NASA WFF CDL SFI & NTCRA			
PROJECT	Supplemental Field Investigation			
REVISED	2/28/2023	GIS BY	MS	2/28/2023
SCALE	1:720	CHK BY	JK	2/28/2023
Base Map: Source: Esri, Maxar, Earthstar Geographics, and the GIS User Community		PM	JK	2/28/2023

AECOM 12420 Milestone Center Drive Germantown, MD 20876

Figure 10-3

L:\DCS\Projects\ENV\GEARS\GEO\NASA\WFF CDL SFI\GIS\MXD\AECOM_Figures\Supplemental_Field_Investigation\Fig_10-3_Proposed_Pre-Excavation_Sample_Locations.mxd



CLIENT	NASA WFF CDL SFI & NTCRA			
NOTES	Supplemental Field Investigation			
REVISED	2/28/2023	GIS BY	MS	2/28/2023
SCALE	1:1,080	CHK BY	JK	2/28/2023
Base Map: Source: Esri, Maxar, Earthstar Geographics, and the GIS User Community		PM	JK	2/28/2023

Previous Soil Samples and Proposed Pre-Excavation Sample Locations

AECOM
12420 Milestone Center Drive
Germantown, MD 20876

Figure 10-4

L:\DCS\Projects\ENV\GEARS\GEO\NASA\WFF CDL SFI\GIS\MXD\AECOM_Figures\Supplemental_Field_Investigation\Fig_10-4_Wallops_Island_Previous_Sample_Field_Location.mxd

unknown if any sample will exceed a PRG, therefore, an additional sample (sample B in **Table 10-1**) will be collected 5 feet further away from the initial sample at each sidewall location (sample A in **Table 10-1**).

Based on the sample results, an additional mobilization may be required to collect pre-excavation samples to address data gaps and fully delineate the extent of soil to be removed under this removal action.

10.4.2 Clean Backfill Confirmation Sampling

Following completion of the excavation, AECOM will backfill the excavation with onsite overburden and clean fill obtained from offsite. Fill and topsoil will be sampled for Total Petroleum Hydrocarbons (TPH-DRO and TPH-GRO), Benzene, Toluene, Ethylbenzene, Xylenes (BTEX), VOCs, SVOCs, metals, PFAS, and TCLP, including ignitability, corrosivity, and reactivity to demonstrate that it is free of contamination prior to being brought on site. The analytical results will be compared to the EPA Region 3 BTAG Ecologically Protective Backfill Values (EPA, 2019) and Industrial Soil Regional Screening Levels based on HQ of 1 (EPA, 2022).

10.5 PERFORMANCE CRITERIA

The Project Team will use the results of the pre-excavation sampling to confirm that the waste and soil are non-hazardous and to verify that risk to human health and the environment from soil that remains is acceptable.

Sensitivity criteria for laboratory-generated data are the analysis specific laboratory method detection limits (MDL) for target analytes listed in **Worksheet #16**.

The data quality will be reviewed to ensure that performance criteria have been met and that the data are sufficient for decision-making purposes. If all data are collected as planned and no data points are missing or rejected for quality reasons, then the investigation completeness will be satisfactory. If any data gaps are identified, including missing or rejected data, the Project Team will assess whether a claim of having achieved project objectives is reasonable based on the quantity and types of data gaps. Project Team members will be involved in rendering the conclusion by consensus regarding adequacy of the data.

To limit uncertainty in the field and laboratory data, performance criteria for field collection and laboratory analysis will be measured. Performance criteria are described in **Worksheets #11, 16, and 18.** In addition, standardized procedures for sampling and analysis will be used. Use of these standardized protocols and adherence to this SAP are designed to minimize uncertainties in decision making.

10.6 SAMPLING DESIGN

The general data collection plan is included in **Worksheet #13** and is detailed in this SAP. The proposed pre-excavation soil sampling locations (**Figure 10-3**) were based on the available chemical and physical data.

SAP Worksheet #11 Field Quality Control Samples

(UFP-QAPP Manual Section 2.6.2-Worksheet #12)

Quality Control (QC) Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria (MPC)
Field Duplicates (collected for total analyses e.g. TAL VOCs/SVOC/Pesticides/TPH/Dioxin/Metals but not TCLP/waste characterization samples)		1 per 10 field samples	Precision	If both the original and duplicate results are ≥ 2x reporting limit, Relative Percentage Difference (RPD) must be ≤ 30% If either the original or duplicate result is < 2x reporting limit, use professional judgment.
Cooler Temperature Indicator		1 per cooler	Representativeness	Temperature must be between 0 and 6 degrees Celsius (°C), but samples must not be frozen.
Matrix Spike (MS) (1) (collected for total analyses e.g. TAL VOCs/SVOC/Pesticides/TPH/Dioxin/Metals but not TCLP/waste characterization samples)		1 per 20 discrete field samples collected	Accuracy/Bias	See **Worksheet #18**.
Matrix Spike Duplicate (MSD) (1) (collected for total analyses e.g. TAL VOCs/SVOC/Pesticides/TPH/Dioxin/Metals but not TCLP/waste characterization samples)		1 per 20 discrete field samples collected	Accuracy/Bias and precision	See **Worksheet #18.**

SAP Worksheet #12 Summary of Project Tasks & Schedule

(UFP-QAPP Manual Section 2.8.1-Worksheets #14 & #16)

12.1 PRE-EXCAVATION SAMPLING

Pre-excavation activities include securing approvals of this SAP, enlisting subcontractors, securing the excavation permit, clearing each sample location for utilities, survey the site as a requirement for the erosion and sediment control plan and the post-excavation design of the land surface, notifications to National Oceanic and Atmospheric Administration (NOAA) the U.S. Environmental Protection Agency (USEPA), and Virginia Department of Environmental Quality (VDEQ) of the planned field work, mobilization, sampling of soils for disposal parameters and to complement the characterization of the waste and surrounding soil, IDW management, site cleanup and demobilizing from the site, laboratory subcontractor chemical analysis of the samples, evaluation of the results, presentation of the results to NASA, EPA, and VDEQ, submittal of the chemical results to the landfill, and the landfill's preparation of manifests and bills of laden. A short description of each task is provided under **Section 12.2.**

The following is a list of additional project-related analytical and reporting tasks that will be completed as part of this investigation. A short description of each task is provided under **Section 12.3**.

- Analytical Tasks
- Data Handling and Management
- Data Tracking
- Data Storage, Archiving, and Retrieval
- Data Security
- Electronic Data
- Data Review and Validation
- Project Reports

AECOM Standard Operating Procedures (SOPs) and field forms for field tasks referenced in this section are identified by title in **SAP Worksheet #14**, and copies of applicable SOPs are provided in **Appendix A**. Field activities will be conducted in accordance with AECOM SOPs. Nondedicated field sampling equipment used at each sample location should be cleaned between uses.

12.2 EXCAVATION

Following completion of the required plans and permits, AECOM will mobilize the project team to the site. Specific activities include mobilizing equipment and materials to execute the work, verifying utility locations, surveying the extent of the area to be excavated, establishing material lay-down and stockpile areas, and site clearing and grubbing.

Erosion control measures will be implemented in accordance with the Erosion and Sedimentation Control (E&SC) Plan (ESCP), which will be submitted under separate cover. WFF has an Annual Standards and Specifications for E&SC and Stormwater Management approved by VDEQ and administered by the WFF Facilities Management Branch (FMB). The ESCP will be approved by FMB prior to mobilization. The ESCP will include clean soil (overburden) and contaminated soil staging areas; clearing and grubbing for perimeter E&SC; installation of perimeter E&SC; clearing and grubbing site for excavation; excavation; backfill with

clean fill; final grading; vegetative stabilization; stabilization and removal of E&SC; stabilization of disturbed areas from removal of E&SC.

Overburden soil will be excavated to access the buried waste. Overburden soil with concentrations above PRGs will be transported offsite for disposal. AECOM will import general fill and topsoil to bring excavation areas back to their original grades. Grass seed and straw will be placed over the surface and the land surface will be surveyed.

12.3 FIELD TASKS

A summary of each field task is provided in the following sections.

12.3.1 Mobilization/Demobilization and Safety Training

Mobilization will consist of the delivery of equipment, materials, and supplies to the site, the complete assembly in satisfactory working order of such equipment at the site, and the satisfactory storage at the site of such materials and supplies. The necessary equipment and supplies include, but are not limited to, the following:

- Documents, forms, logbooks, log sheets, labels, custody seals, air bills, and other paperwork required by the SAP and HASP.
- Vehicles for personnel, equipment, and sample transport.
- Personnel, supplies, and equipment (e.g., bottle ware and personal protective equipment [PPE]) required by the SAP and HASP.
- Required sample containers.
- Equipment and supplies for sample custody, preservation, and packaging.
- Other miscellaneous office and field supplies.
- Mobilization/demobilization for the excavation portion also includes machinery for excavation of soil and transport offsite

Project-specific health and safety training for all AECOM field staff and subcontractors, as applicable, will be provided as part of site mobilization and included in the HASP. During the required training and orientation, field team members will review the SAP and will be given any project-specific health and safety training based on the HASP. Field personnel must review and sign off on the HASP. The SSHO will be responsible for reviewing the HASP with field team members. It will also be necessary to provide orientation and health and safety training for any additional or replacement field team members assigned after mobilization.

The AECOM FOL or designee will also coordinate with the Facility POC, Ms. Susan Dunn, verbally or via email at least 2 weeks prior to commencement of field work to arrange for access to the site and to identify appropriate locations for the temporary storage of equipment and supplies.

Demobilization will consist of the prompt and timely removal of all equipment, materials, and supplies from the site following completion of the work. Demobilization includes the cleanup and removal of IDW generated during the investigation. Upon demobilization, field investigation paperwork will be filed and docketed in the project file.

12.3.2 Equipment Calibration

Field equipment will be calibrated at the beginning of each day, unless otherwise stated by the equipment manufacturer or if problems are noted in the field. These procedures are described in **Worksheet #15**.

12.3.3 Soil Sample Collection

Pre-excavation soil sample collection will be accomplished via direct push methods as per AECOM SOP 3-17 (**Appendix A**). Post-excavation soil samples will not be collected; sufficient pre-excavation soil samples will be collected and analyzed to assess the limits of excavation.

12.3.4 Field Decontamination Procedures

Decontamination will be required for all reusable sampling/measuring equipment to prevent transferring potential contaminants to other locations or samples. Reusable sampling/measuring equipment will be decontaminated with a non-phosphate detergent scrub followed by a water rinse. The water used to decontaminate the tooling and equipment will be NASA potable water. All fluids generated during decontamination will be discharged to the ground near the sample location (**Section 12.2.8**). Decontamination procedures are detailed in AECOM SOP 3-06 (**Appendix A**).

12.3.5 Investigation Derived Waste Management

IDW will consist of excess soil from pre-excavation soil sampling and water from dewatering activities. IDW will be containerized in 55-gallon drums, properly labeled, and characterized. The soil IDW will be transported and disposed of with the excavated waste. The water IDW will be characterized and properly disposed offsite. The specific IDW analytical requirements for waste characterization will be dependent on the actual disposal facility requirements and NASA policies.

Used PPE (i.e., nitrile gloves), tubing, and general refuse will be bagged and disposed of as general trash.

12.3.6 Documentation

Sample log sheets will be maintained for each sample collected. In addition, sample collection information will be recorded in bound field notebooks or on specific field forms. Samples will be packaged and shipped according to AECOM SOPs 3-03 and 3-04 (**Appendix A**).

A summary of field activities will be properly recorded in a bound logbook with consecutively numbered pages. Logbooks will be assigned to field personnel and stored in a secured area when not in use. Logbooks will be maintained in accordance with AECOM SOP 3-02 (**Appendix A**).

Entries will be written in ink. No erasures will be made. If an incorrect entry is made, striking a single line through the incorrect information will mark the correction. The person making the correction will then initial, date, and provide the corrected entry. Sampling forms and other field forms will also be used to document field activities.

12.4 ANALYTICAL AND REPORTING TASKS

The following is a list of analytical and data management/reporting tasks.

12.4.1 Analytical Tasks

Chemical analyses will be performed by Pace South Carolina located in West Columbia, SC, sub-contracting analyses to Pace Minnesota in Minneapolis, MN and Pace National in Joliet, TN. These labs have been accredited to conduct the analyses required by this SAP. A copy of Pace's accreditation is provided in **Appendix B**. Analyses will be performed in accordance with the analytical methods specified in **Worksheet #13**. The laboratory will perform chemical analysis following laboratory-specific SOPs (**Worksheet #17** and **Appendix C**) based on the analytical methods listed in **Worksheet #13**. Laboratory data will be delivered to AECOM in the form of an Electronic Data Deliverable (EDD) and portable document format (PDF) data package. This information will be captured in the project database that will be uploaded upon completion of the report.

12.4.2 Data Handling and Management

After each sampling event is completed, the field sampling log sheets will be organized by date and medium and filed in the project files. The field logbooks for this project will be used only for this facility and will also be categorized and maintained in the project files after completion of the field program. When possible, logbooks will be segregated by sampling activity. The field logbooks will be titled based on date and activity. Prior to placement in the project files, the logbook pages will be scanned electronically. The data handling procedures to be followed by the laboratory will meet the requirements of the laboratory technical specification. The electronic data will be automatically downloaded into the AECOM database in accordance with proprietary AECOM processes.

12.4.3 Data Tracking

Data will be tracked from generation to archiving in the project-specific files. The AECOM QAM/Project Chemist (or designee) is responsible for tracking the samples collected and shipped to the contracted analytical laboratory. Upon receipt of the data packages from the analytical laboratory, the project chemist will coordinate the data validation effort, which includes verifying that the data packages are complete and that results for all samples have been delivered by the analytical laboratory.

12.4.4 Data Storage, Archiving, and Retrieval

After the data are validated, the data packages are entered into the AECOM file system and archived in secure files. The field records including field logbooks, sample log sheets, and chain-of-custody records will be submitted by the AECOM FOL to be entered into the AECOM file system prior to archiving in secure project files. As documents are finalized, all relevant data and records are uploaded electronically to the project database and retained there indefinitely.

12.4.5 Data Security

The AECOM project files are restricted to designated personnel only. Records may only be borrowed temporarily from the project file using a sign-out system. The AECOM Data Manager maintains the electronic data files. Access to the data files is restricted to qualified personnel only. File and data backup procedures are routinely performed.

12.4.6 Electronic Data

One hundred percent (100%) of the laboratory data (electronic and PDF report) will be validated in accordance with specifications in **Worksheet #19**, and qualifiers will be manually added to the database. Data will then be compiled and loaded into the project database. This process includes a QA review of the data to ensure that the content and format of the data satisfy AECOM requirements.

12.4.7 Data Review and Validation

Data verification, validation, and usability assessment processes are described on **Worksheet #19**.

12.4.8 Project Reporting

After completion of the data review and resolution of any anomalies (should they occur), project-specific reports will be prepared to present the results of the investigations and satisfy data quality objectives, as identified in **Worksheet #10**. The draft reports will be submitted to the NASA PM for initial review, and NASA comments will be addressed. After NASA approves the edits, the reports will be submitted to the EPA and VDEQ for review and comment. If the EPA or VDEQ provide comments, a response-to-comments table and a redline version of the text will detail the proposed changes. Once these changes are approved, a final report will be generated and sent to the EPA and VDEQ.

12.5 PROJECT SCHEDULE

Activity	Responsible party	Planned start date	Planned completion date	Deliverable(s)	Deliverable due date
UFP-SAP	AECOM	8/4/22	1/10/23	Final UFP-SAP	1/20/23
Mobilization	AECOM	1/19/23	1/29/23	N/A	
Soil sample collection	AECOM	2/9/23	2/20/23	Field notes	2/21/23
Analysis	Pace	2/21/22	3/6/23	Report of analysis/data package	3/6/23
Data Validation	AECOM	3/9/23	3/19/23	Validation summary report	3/19/23
Summarize data	AECOM	3/9/23	3/11/23	Data memorandum	3/19/23
Usability assessment	AECOM	3/20/23	3/23/23	Data memorandum	3/23/23
Mobilization to excavate waste	AECOM	May 2023	October 2023	Closure Report	November 2023

SAP Worksheet #13 Sample Details Table

(UFP-QAPP Manual Section 2.8.1-Worksheets #18,19, 20 and 30)

WFF CDL Wallops Flight Facility, Wallops Island, Virginia (Tentative Sampling Dates: Pre-Excavation – January 2023)	**Analysis Group**	VOCs, SVOCs, PAHs, Pesticides, Metals, Dioxin/Furan TCLP Parameters: TCLP VOC/SVOC/Herbicides/Pesticides/RCRA 8 Metals + Total Sulfur Waste Characterization Parameters: Total PCB, Ignitability, Corrosivity, Reactivity
	Analytical Method	Solid Matrix: 8260D VOCs, 8270E SVOCs/PAHs, 8081B Pesticides, 6010D Metals, 7471A Mercury, 8290 Dioxin/Furan Waste Matrix: 1311+ 8260D/8270E/8081B/8151A/6010D/7471A TCLP Parameters, 8082A/1010A/9045D/SW-846 Section 7.3 Waste Characterization Parameters
	Analytical Laboratory/ Analytical SOP Reference	Solid Matrix: ME0012X-22 VOCs, ME0014Q-17 SVOC/PAH, ME0019A-10 Pesticides, ME001FJ-08 Metals, ME0017R-09 Mercury, ENV-SOP-MIN4-0026 Dioxin/Furan Waste Matrix: TCLP - ME0019C-08+ ME0012X-22/ME0014Q-17/ME0019A-10/ME00157-09/ME001FJ-08/ME0017R-09 + ENV-SOP-MTJL-0215 Waste Characterization - ME0019A-10/ME0017K-11/ME0014S-09/ME001GA-06
	Data Package Turnaround Time	28 Work Days
	Container Type/ Volume Required (if different than container volume)	Total/TCLP VOC: Terracore Kit (1x 40 mL VOA unpreserved, 1x 40ml VOA with 5mL Methanol, 2x 40 mL VOA with 5 mL DI water and stir bar) Total SVOC/PAH: 2x 4 oz Glass Jar Total Pesticides: 1x 4 oz Glass Jar Total Metals/Mercury : 1x 4 oz Glass Jar Total Dioxin/Furan: 1x 4 oz Glass Jar TCLP SVOC/Herbicides/Pesticides/RCRA 8 Metals + Total Sulfur: 1x 16 oz Jar or gallon bag Waste PCB: 1x 4 oz Glass Jar Ignitability: 1x 2 oz Glass Jar Corrosivity: 1x 250 mL Plastic Bottle Reactivity: 1x 4 oz Glass Jar
	Preservative	All methods: Cool to ≤6°C VOCs: As per Terracore Kit
Pace South Carolina (primary), Minnesota (sub), and Pace National (sub) Project Manager: Kathy Smith Direct Dial: 803-791-9700 Email:Kathy.Smith@pacelabs.com	**Holding Time (Preparation/ Analysis)**	Total/TCLP VOC: 14 Days to Analysis Total SVOC/PAH: 14 Days to Extraction/40 Days to Analysis Total Pesticides: 14 Days to Extraction/40 Days to Analysis Total Metals/Mercury: 28 Days to Extraction/6 Months to Analysis Total Dioxin/Furan: 30 Days to Extraction, 45 Days to Analysis TCLP SVOC/Herbicides/Pesticides/RCRA 8 Metals + Total Sulfur: 14 days from collection to start of leach (7 days from end of TCLP to analysis for SVOC), 28 days to leach to analysis Waste PCB: 14 Days to Extraction/40 Days to Analysis Ignitability: 14 Days to Extraction/14 Days to Analysis Corrosivity: Immediate (Samples will ship day of collection and lab will make effort to perform analysis ASAP) Reactivity: 28 days to Analysis

Site	Matrix	Sample Location[1]	Sample ID[2]	Analysis
Proposed Pre-Excavation Soil Samples				
CDL	Soil	B1	B1A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B1	B1B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B2	B2A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B2	B2B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B3	B3A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B3	B3B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B4	B4A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B4	B4B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B5	B5A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B5	B5B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B6	B6A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B6	B6B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B7	B7A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B7	B7B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B8	B8A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B8	B8B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B9	B9A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B9	B9B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B10	B10A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B10	B10B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B11	B11A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B11	B11B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B12	B12A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B12	B12B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B13	B13A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B13	B13B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B14	B14A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B14	B14B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B15	B15A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B15	B15B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B16	B16A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B16	B16B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B17	B17A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B17	B17B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B18	B18A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B18	B18B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B19	B19A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B19	B19B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B20	B20A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B20	B20B	VOC, SVOCs, metals, and pesticides

Site	Matrix	Sample Location[1]	Sample ID[2]	Analysis
CDL	Soil	B21	B21A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B21	B21B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B22	B22A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B22	B22B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B23	B23A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B23	B23B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B24	B24A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B24	B24B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B25	B25A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B25	B25B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B26	B26A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B26	B26B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B27	B27A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B27	B27B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B28	B28A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B28	B28B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B29	B29A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B29	B29B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B30	B30A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B30	B30B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B31	B31A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B31	B31B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B32	B32A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B32	B32B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B33	B33A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B33	B33B	VOC, SVOCs, metals, and pesticides
CDL	Soil	B34	B34A	VOC, SVOCs, metals, and pesticides
CDL	Soil	B34	B34B	VOC, SVOCs, metals, and pesticides
CDL	Soil	S1	S1A	VOC, SVOCs, metals, and pesticides
CDL	Soil	S1	S1B	VOC, SVOCs, metals, and pesticides
CDL	Soil	S2	S2A	VOC, SVOCs, metals, and pesticides
CDL	Soil	S2	S2B	VOC, SVOCs, metals, and pesticides
CDL	Soil	S3	S3A	VOC, SVOCs, metals, and pesticides
CDL	Soil	S3	S3B	VOC, SVOCs, metals, and pesticides
CDL	Soil	S4	S4A	VOC, SVOCs, metals, and pesticides
CDL	Soil	S4	S4B	VOC, SVOCs, metals, and pesticides
CDL	Soil	S5	S5A	VOC, SVOCs, metals, and pesticides
CDL	Soil	S5	S5B	VOC, SVOCs, metals, and pesticides
CDL	Soil	S6	S6A	VOC, SVOCs, metals, and pesticides
CDL	Soil	S6	S6B	VOC, SVOCs, metals, and pesticides
CDL	Soil	S7	S7A	VOC, SVOCs, metals, and pesticides

Site	Matrix	Sample Location[1]	Sample ID[2]	Analysis
CDL	Soil	S7	S7B	VOC, SVOCs, metals, and pesticides
CDL	Soil	S8	S8A	VOC, SVOCs, metals, and pesticides
CDL	Soil	S8	S8B	VOC, SVOCs, metals, and pesticides
CDL	Soil	S9	S9A	VOC, SVOCs, metals, and pesticides
CDL	Soil	S9	S9B	VOC, SVOCs, metals, and pesticides
CDL	Soil	S10	S10A	VOC, SVOCs, metals, and pesticides
CDL	Soil	S10	S10B	VOC, SVOCs, metals, and pesticides
CDL	Soil	Composite from middle of core at B1, B2, B3, B4	C1	TCLP VOCs, SVOCs, metals, reactivity, ignitability, corrosivity, and total sulfur
CDL	Soil	Composite from middle of core at B5, B6, B7, B8, B9	C2	TCLP VOCs, SVOCs, metals, reactivity, ignitability, corrosivity, and total sulfur
CDL	Soil	Composite from middle of core at B10, B11, B12, B13, B14	C3	TCLP VOCs, SVOCs, metals, reactivity, ignitability, corrosivity, and total sulfur
CDL	Soil	Composite from middle of core at B15, B16, B17, B18, B19, B20	C4	TCLP VOCs, SVOCs, metals, reactivity, ignitability, corrosivity, and total sulfur
CDL	Soil	Composite from middle of core at B21, B22, B23	C5	TCLP VOCs, SVOCs, metals, reactivity, ignitability, and corrosivity
CDL	Soil	Composite from middle of core at B24, B25, B26	C6	TCLP VOCs, SVOCs, metals, reactivity, ignitability, and corrosivity
CDL	Soil	Composite from middle of core at B27, B28, B29, B30	C7	TCLP VOCs, SVOCs, metals, reactivity, ignitability, and corrosivity
CDL	Soil	Composite from middle of core at B31, B32, B33, B34	C8	TCLP VOCs, SVOCs, metals, reactivity, ignitability, and corrosivity

Offsite in the vicinity of Wallops Island, Virginia (Tentative Sampling Date: January 2023) **Pace South Carolina (primary), Minnesota (sub), and Pace National (sub)** Project Manager: Kathy Smith Direct Dial: 803-791-9700 Email:Kathy.Smith@pacelabs.com	**Analysis Group**	PFAS, BTEX, VOCs, SVOCs, TPH-GRO, TPH-GRO, metals, TCLP, ignitability, corrosivity, and reactivity
	Analytical Method	QSM Table B-15, 8260D VOCs, 8270E SVOCs, 6010D Metals, 1311+ 8260D/8270E/8081B/8151A/6010D/7471A TCLP Parameters, 8082A/1010A/9045D/SW-846 Section 7.3 Waste Characterization Parameters
	Analytical Laboratory/ Analytical SOP Reference	ME003NI-04 PFAS, ME0012X-22 VOCs, ME0014Q-17 SVOC, ME001FJ-08 Metals, TCLP - ME0019C-08+ ME0012X-22/ME0014Q-17/ME0019A-10/ME00157-09/ME001FJ-08/ME0017R-09 + ENV-SOP-MTJL-0215 Waste Characterization - ME0019A-10/ME0017K-11/ME0014S-09/ME001GA-06
	Data Package Turnaround Time	28 Work Days
	Container Type/ Volume Required (if different than container volume)	PFAS: 1x 4 oz HDPE bottle Total/TCLP VOC+BTEX: Terracore Kit (1x 40 mL VOA unpreserved, 1x 40ml VOA with 5mL Methanol, 2x 40 mL VOA with 5 mL DI water and stir bar) Total SVOC: 2x 4 oz Glass Jar Total GRO: 1x 2 oz Glass Jar Total DRO: 1x 4 oz Glass Jar Total Metals/Mercury : 1x 4 oz Glass Jar Waste PCB: 1x 4 oz Glass Jar TCLP SVOC/Herbicides/Pesticides/RCRA 8 Metals Waste: 1x 16 oz Jar or gallon bag Ignitability: 1x 2 oz Glass Jar Corrosivity: 1x 250 mL Plastic Bottle Reactivity: 1x 4 oz Glass Jar
	Preservative	All methods: Cool to ≤6°C VOCs: As per Terracore Kit
	Holding Time (Preparation/Analysis)	PFAS: 28 Days to Extraction, 28 Days to Analysis Total/TCLP VOC+BTEX: 14 Days to Analysis Total SVOC: 14 Days to Extraction/40 Days to Analysis Total GRO: 14 days to Analysis Total DRO: 14 Days to Extraction/40 Days to Analysis Total Metals: 6 Months to Extraction/6 Months to Analysis TCLP SVOC/Herbicides/Pesticides/RCRA 8 Metals: 14 days from collection to start of leach (7 days from end of TCLP to analysis for SVOC), 28 days to leach to analysis Waste PCB: 14 Days to Extraction/40 Days to Analysis Ignitability: 14 Days to Extraction/14 Days to Analysis Corrosivity: Immediate (Samples will ship day of collection and lab will make effort to perform analysis ASAP) Reactivity: 28 days to Analysis

Site	Matrix	Sample Location[1]	Sample ID[2]	QC
Proposed Soil Samples of Backfill				
Clean fill	Soil	CF-01	CF-01	BTEX, VOCs, TPH-DRO, TPH-GRO, SVOCs, metals, PFAS, TCLP, ignitability, corrosivity, and reactivity
Clean fill	Soil	CF-02	CF-02	BTEX, VOCs, TPH-DRO, TPH-GRO, SVOCs, metals, PFAS, TCLP, ignitability, corrosivity, and reactivity
Clean fill	Soil	CF-03	CF-03	BTEX, VOCs, TPH-DRO, TPH-GRO, SVOCs, metals, PFAS, TCLP, ignitability, corrosivity, and reactivity
Clean fill	Soil	CF-04	CF-04	BTEX, VOCs, TPH-DRO, TPH-GRO, SVOCs, metals, PFAS, TCLP, ignitability, corrosivity, and reactivity

Offsite in the vicinity of Wallops Island, Virginia (Tentative Sampling Date: January 2023) **Pace South Carolina (primary), Minnesota (sub), and Pace National (sub)** Project Manager: Kathy Smith Direct Dial: 803-791-9700 Email:Kathy.Smith@pacelabs.com	**Analysis Group**	PFAS, BTEX, VOCs, SVOCs, TPH-GRO, TPH-GRO, metals, TCLP, ignitability, corrosivity, and reactivity
	Analytical Method	QSM Table B-15, 8260D VOCs, 8270E SVOCs, 6010D Metals, 1311+ 8260D/8270E/8081B/8151A/6010D/7471A TCLP Parameters, 8082A/1010A/9045D/SW-846 Section 7.3 Waste Characterization Parameters
	Analytical Laboratory/ Analytical SOP Reference	ME003NI-04 PFAS, ME0012X-22 VOCs, ME0014Q-17 SVOC, ME001FJ-08 Metals, TCLP - ME0019C-08+ ME0012X-22/ME0014Q-17/ME0019A-10/ME00157-09/ME001FJ-08/ME0017R-09 + ENV-SOP-MTJL-0215 Waste Characterization - ME0019A-10/ME0017K-11/ME0014S-09/ME001GA-06
	Data Package Turnaround Time	28 Work Days
	Container Type/ Volume Required (if different than container volume)	PFAS: 1x 4 oz HDPE bottle Total/TCLP VOC+BTEX: Terracore Kit (1x 40 mL VOA unpreserved, 1x 40ml VOA with 5mL Methanol, 2x 40 mL VOA with 5 mL DI water and stir bar) Total SVOC: 2x 4 oz Glass Jar Total GRO: 1x 2 oz Glass Jar Total DRO: 1x 4 oz Glass Jar Total Metals/Mercury : 1x 4 oz Glass Jar Waste PCB: 1x 4 oz Glass Jar TCLP SVOC/Herbicides/Pesticides/RCRA 8 Metals Waste: 1x 16 oz Jar or gallon bag Ignitability: 1x 2 oz Glass Jar Corrosivity: 1x 250 mL Plastic Bottle Reactivity: 1x 4 oz Glass Jar
	Preservative	All methods: Cool to ≤6°C VOCs: As per Terracore Kit
	Holding Time (Preparation/Analysis)	PFAS: 28 Days to Extraction, 28 Days to Analysis Total/TCLP VOC+BTEX: 14 Days to Analysis Total SVOC: 14 Days to Extraction/40 Days to Analysis Total GRO: 14 days to Analysis Total DRO: 14 Days to Extraction/40 Days to Analysis Total Metals: 6 Months to Extraction/6 Months to Analysis TCLP SVOC/Herbicides/Pesticides/RCRA 8 Metals: 14 days from collection to start of leach (7 days from end of TCLP to analysis for SVOC), 28 days to leach to analysis Waste PCB: 14 Days to Extraction/40 Days to Analysis Ignitability: 14 Days to Extraction/14 Days to Analysis Corrosivity: Immediate (Samples will ship day of collection and lab will make effort to perform analysis ASAP) Reactivity: 28 days to Analysis

Site	Matrix	Sample Location[1]	Sample ID[2]	QC
Proposed Soil Samples of Backfill				
Dewater	Aqueous	DW-01	DW-02	BTEX, VOCs, TPH-DRO, TPH-GRO, SVOCs, metals, PFAS, TCLP, ignitability, corrosivity, and reactivity
Dewater	Aqueous	DW-02	DW-02	BTEX, VOCs, TPH-DRO, TPH-GRO, SVOCs, metals, PFAS, TCLP, ignitability, corrosivity, and reactivity

Field QC Samples			
QC	Field duplicate	CDL-DUP01-YYYYMMDD	Tentative sampling locations will be selected by the FOL in the field based on field screening results and physical observations of potential contamination.
QC	MS	These are noted on the sample and chain of custody form as “MS/MSD”.	
QC	MSD		
QC	Trip Blank	TB-XX	For VOCs only, 1 per cooler that is submitted with VOC samples included

Frequency of QA/QC sample collection:

Duplicates – 1 per 10 samples,

MS/MSD – 1 per 20 samples,

Trip Blank – 1 per cooler containing VOC samples

1 Pre-excavation soil sample locations are presented on **Figure 10-2**.

2 YYYYMMDD stands for the year, month, and date the sample is collected. For example, if a sample is collected from DPT-22-1 on September 25, 2022, the sample ID would be DPT22-1-20220925.

3 Metals of interest: Aluminum, Antimony, Arsenic, Cadmium, Chromium, Cobalt, Copper, Iron, Manganese, Nickel, and Vanadium

SAP Worksheet #14 **Project Sampling SOP References Table**

(UFP-QAPP Manual Section 3.1.2-Worksheet #21)

Reference Number	Title, Revision Date and / or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
3-01	*Utility Clearance*	AECOM		N	Contained in Appendix A
3-02	*Logbooks*	AECOM	Field logbook, field sample forms, boring logs	N	Contained in Appendix A
3-03	*Recordkeeping, Sample Labeling and Chain of Custody*	AECOM		N	Contained in Appendix A
3-04	*Sample Handling, Storage, and Shipping*	AECOM	Sample bottle ware, packaging material, shipping materials	N	Contained in Appendix A
3-05	*Investigation-Derived Waste Management*	AECOM		N	Contained in Appendix A
3-06	*Equipment Decontamination*	AECOM	Pumps, reusable sample scoops, drill rods, and screens	N	Contained in Appendix A
3-07	*Land Surveying*	AECOM		N	Contained in Appendix A
3-16	*Soil and Rock Classification*	AECOM		N	Contained in Appendix A
3-17	*Direct Push Sampling Techniques*	AECOM		N	Contained in Appendix A
3-19	*Headspace Screening for Total VOCs*	AECOM		N	Contained in Appendix A
3-20	*Operation and Calibration of Photoionization Detector*	AECOM		N	Contained in Appendix A
3-21	*Surface and Subsurface Soil Sampling Procedures*	AECOM		N	Contained in Appendix A

SAP Worksheet #15 Field Equipment Calibration, Maintenance, Testing, and Inspection Table

(UFP-QAPP Manual Section 3.1.2.4- Worksheet #22)

Field Equipment	Calibration Activity	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
Depth to water meter	Calibration in accordance with manufacturer's instructions.	N/A	N/A	N/A	Daily before use	Calibration in accordance with manufacturer's instructions.	Replace	FOL or Designee	Manufacturer's Guidance.
Photoionization Detector (PID) – MiniRAE 3000 (or equivalent)	Calibration in accordance with manufacturer's instructions.	N/A	N/A	N/A	Daily before use	Calibration in accordance with manufacturer's instructions.	Replace	FOL or Designee	Manufacturer's Guidance.

Note:

Use the Default Calibration Criteria for Multimeters and Turbidity.

SAP Worksheet #16 Reference Limits and Evaluation Tables

(UFP-QAPP Manual Section 2.8.1 – Worksheet #15)

Matrix: Soil

Analytical Group: Volatile/Semivolatile Organic Compounds (VOCs/SVOCs), Metals, Pesticides, PAH, and Dioxins

Analyte	Chemical Abstracts Service Registry Number (CASRN)	PAL	PAL Reference [1]	Project Quantitation Limit Goal (PQLG) [2]	Laboratory Reference Limits	
					Reporting Limit	MDL
VOCs (mg/kg)						
1,1,2-Trichloroethane	79-00-5	0.12	Residential soil PRG	0.0050	0.0050	0.0020
2-Hexanone	591-78-6	29	Residential soil PRG	0.0010	0.0010	0.0040
SVOCs (mg/kg)						
Benzo(a)pyrene	50-32-8	1.0	Residential soil PRG	0.0027	0.0027	0.00066
Metals (mg/kg)						
Aluminum	7429-90-5	13900	Residential soil PRG	20	20	7.5
Antimony	7440-36-0	2.4	Residential soil PRG	1.0	1.0	0.25
Arsenic	7440-38-2	5.3	Residential soil PRG	0.75	0.75	0.25
Barium	7440-39-3	330	ESV	1.3	1.3	0.33
Cadmium	7440-43-9	24	Residential soil PRG	0.25	0.25	0.063
Chromium	7440-47-3	18.4	Residential soil PRG	0.50	0.50	0.13
Cobalt	7440-48-4	7.3	Residential soil PRG	1.3	1.3	0.33
Copper	7440-50-8	1033	Residential soil PRG	0.50	0.50	0.23
Iron	7439-89-6	18333	Residential soil PRG	5.0	5.0	2.3
Manganese	7439-96-5	257	Residential soil PRG	0.75	0.75	0.30
Mercury	7439-97-6	0.5	ESV	0.083	0.083	0.020
Nickel	7440-02-0	125	Residential soil PRG	2.0	2.0	0.53
Thallium	7440-28-0	0.26	Residential soil PRG	2.5	2.5[a]	0.63
Vanadium	7440-62-2	33	Residential soil PRG	2.5	2.5	0.63
Zinc	7440-66-6	153	ESV	2.5	2.5	0.63
Dioxins (mg/kg)						

Analyte	Chemical Abstracts Service Registry Number (CASRN)	PAL	PAL Reference [1]	Project Quantitation Limit Goal (PQLG) [2]	Laboratory Reference Limits	
					Reporting Limit	MDL
2,3,7,8-TCDD TEQ	1746-01-6	2.60E-05	Residential soil PRG	1.0E-06	1.0E-06	2.0E-07
Pesticides (mg/kg)						
Total DDD/DDE/DDT	72-54-8/72-55-9/ 50-29-3	0.044	ESV	0.0010	0.0010	0.00018
Total HMW PAHs	N/A	18	ESV	0.0027	0.0027	0.0010

1 mg/kg = milligrams per kilogram
2 PRG = Preliminary Remediation Goal
3 ESV = Ecological Screening Value
4 Total DDD/DDE/DDT is the summation of BTVs for the individual compounds
5 PALs determined from the PRGs and ESVs.
6 The PQLG is approximately one-third of the PAL.
a PAL not achievable by laboratory method

SAP Worksheet #17 Analytical SOP References Table

(UFP-QAPP Manual Section 3.2.1 – Worksheet #23)

Pace South Carolina (Pace Minnesota sub for Dioxin & PAH)

Project Manager: Kathy Smith

Direct Dial: 803-791-9700

Email: Kathy.Smith@pacelabs.com

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)
ME003NI-04	Determination of Per- and Polyfluoroalkyl Substances (PFAS) by LC/MS/MS (Isotope Dilution) QSM 5.1 Table B-15 03/30/2021	Definitive	PFAS – Solid	LC-MS-MS	Pace Analytical Services - South Carolina	N
ME0012X-22	GC/MS Volatiles Analysis EPA SW-846 8260B/C/D, EPA 624.1, and SM6200 B-2011 Effective Date: 09/23/21	Definitive	GC/MS Volatiles - Solid	GC/MS	Pace Analytical Services - South Carolina	N
ME0014Q-17	Semivolatiles by GC/MS Analysis EPA Methods 8270D / 625.1; Prepared by EPA Methods 3520C, 3546, 3550C, 3510C, 3540C and 3580A Effective Date: 09/27/21	Definitive	GC/MS Semi-volatiles – Solid	GC/MS	Pace Analytical Services - South Carolina	N
ME0014S-09	pH by Electrometric Measurement / pH Paper Method SM4500-H B-2011 / 9040C / 150.1 / 9041A / 9045D Effective Date: 01/21/22	Definitive	Inorganic Non-Metals Solid	Meter	Pace Analytical Services - South Carolina	N
ME00157-09	Herbicides by Gas Chromatographic Analysis EPA SW-846 Method 8151A Effective Date: 09/23/21	Definitive	GC Semi-volatiles - Solid	GC/ECD	Pace Analytical Services - South Carolina	N
ME0017K-11	Flash Point by Pensky-Marten Closed Cup Tester Method 1010A/1010B Effective Date: 02/10/22	Definitive	Inorganic Non-Metals - Solid	NA	Pace Analytical	N

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
					Services - South Carolina	
ME0017R-09	Mercury Analysis by Cold-Vapor Atomic Absorption Methods 245.1 / 7470A and 7471B Effective Date: 12/09/21	Definitive	Metals - Solid	CVAA	Pace Analytical Services - South Carolina	N
ME0019A-10	Pesticides and PCBs by Gas Chromatographic Analysis EPA 608.3; SW-846 Methods 8081B and 8082A Effective Date: 09/28/21	Definitive	GC Semi-volatiles - Solid	GC/ECD	Pace Analytical Services - South Carolina	N
ME0019C-08	Toxicity Characteristic Leaching Procedure EPA Method 1311 / ISM02.4 / SOM02.4 Effective Date: 05/14/21	Preparation	Waste Preparation	NA	Pace Analytical Services - South Carolina	N
ME001FJ-08	Inductively Coupled Plasma - Atomic Emission Spectroscopy Method 6010D Effective Date: 10/14/21	Definitive	Metals – Solid	ICP-AES	Pace Analytical Services - South Carolina	N
ME001GA-06	Reactivity - Reactive Cyanide and Reactive Sulfide SW-846 Method Guidance Section 7.3 Effective Date: 10/29/21	Definitive	Inorganic Non-Metals - Solid	NA	Pace Analytical Services - South Carolina	N
ME001IM-07	Extraction of Chlorinated Herbicides Method 8151A Effective Date: 02/10/22	Preparation	Semi-volatile Extractables - Solid	NA	Pace Analytical Services - South Carolina	N
ENV-SOP-MIN4-0026	Analysis of Dioxin and Furans by 8290, 8290A, and 1613B Effective Date: 08/16/21	Definitive	Dioxin+Furan – Solid	HRGC/MS	Pace Analytical Services - Minnesota	N
ENV-SOP-MTJL-0215	Determination Metals and Trace Elements in Various Matrices by ICP-AES (EPA Methods 6010B, 6010C, 6010D [ICP-OES], and 200.7)	Definitive	Total Sulfur – Solid	ICP-AES	Pace National	N

Laboratory SOPs are available directly from the laboratory upon request.

SAP Worksheet #18 Analytical Instrument and Equipment Maintenance, Testing, and Inspection

(UFP-QAPP Manual Section 3.4 – Worksheet #25)

Laboratory: Pace

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
LC-MS/MS	Check column pressure and mobile phase levels/expirations daily. Perform the following as needed: prepare aqueous mobile phase, clean/replace injection needle, replace guard cartridge, backflush/replace column, replace injector seat, clean curtain/orifice plate, retune MS	PFAS	Inspect all tubing connections at time of maintenance to assure no leaks present. Monitor instrument performance via calibrations, CCVs, and blanks.	Initially, after major maintenance, CCV not meeting 2X	Same as initial calibration and continuing calibration verification	Same as initial calibration and continuing calibration verification	Analyst/Supervisor	ME003NI
GC/MS	Check for leaks, replace gas line filters, recondition or replace trap, replace column, clean injection port/liner.	Volatiles	Monitor instrument performance via Continuing Calibration Verification	As needed	No maintenance is required as long as instrument QC meets DOD criteria	Replace connections, clean source, replace gas line filters, replace trap, replace GC column, clip column, replace injection port liner, clean injection port, replace Electron Multiplier	Analyst, Supervisor, QA Manager	ME0012X

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GC/MS	Check pressure and gas supply daily. Bake trap/column, manual tune if BFB/DFTPP not in criteria. Perform the following as needed: change septa, cut/replace column, change trap, clean source, clean injection port/liner.	Volatiles/ Semi-volatiles	ion source, injector liner, column, column flow. Monitor instrument performance via calibrations and blanks.	Initially, after major maintenance, CCV not meeting 2X	Same as Initial Calibration and Continuing Calibration Verification	Same as Initial Calibration and Continuing Calibration Verification	Analyst/Supervisor	ME0014Q
GC/ECD	Check pressure and gas supply daily. Bake column, change septa as needed, cut column as needed	Pesticides /PCBs/Herbicides	Injector liner, septa, column, column flow. Monitor instrument performance via calibrations and blanks	Initially, after major maintenance, CCV not meeting 2X	Same as Initial Calibration and Continuing Calibration Verification	Same as Initial Calibration and Continuing Calibration Verification	Analyst/Supervisor	ME0019A
GC/ECD	Check pressure and gas supply daily. Bake column, change septa as needed, cut column as needed	Pesticides / PCBs/ Herbicides	Injector liner, septa, column, column flow. Monitor instrument performance via calibrations and blanks	As needed	Same as Initial Calibration and Continuing Calibration Verification	Same as Initial Calibration and Continuing Calibration Verification	Analyst/ Supervisor	ME00157
HRGC/MS	Per Laboratory SOP Section 9.1	Dioxin/Furan	Per Laboratory SOP Section 9.1	Per Laboratory SOP Section 9.1	Per Laboratory SOP Section 9.1	Per Laboratory SOP Section 9.1	Analyst/ Supervisor	ENV-SOP-MIN4-0026
ICP-AES	Check pressure and gas supply daily. Replace pump tubing as needed. Clean or replace nebulizer, spray chamber and torch as needed.	metals	Evaluate occurrences of high blanks, instrument drift, erratic readings, flickering torch and/or high RSDs	Daily to semi-annually depending on the maintenance activity performed.	Per SOP	Per SOP	Analyst/ supervisor	ME001FJ
Hg	Check pressure and gas supply daily. Replace pump tubing, clean/replace optical cell, replace lamp as needed	Hg	Tubing, sample probe, optical cell.	Initially, daily, or as needed	Monitor instrument performance via calibration and QC acceptance criteria.	Varies	Analyst/Supervisor	ME0017R

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
pH Meter	Check electrode	pH	Flush and refill electrode; clean electrode with methanol	As needed	No maintenance is required as long as instrument QC meets criteria	Clean or replace as necessary	Analyst, Supervisor QA Manager	ME0014S
Pensky-Martens Closed Cup Tester	Test cup and cover are cleaned with acetone and dried.	Cleaning	Visual	Prior to use – sample cup is cleaned in between each analysis	Cleaned and dried	Clean and dry	Analyst, Supervisor QA Manager	ME0017K-07

Notes:
ESI = electrospray ionization
LC/MS = liquid chromatography/mass spectrometry
QA = quality assurance
QC = quality control
SOP = standard operating procedure

SAP Worksheet #19 Sample Handling, Custody, and Disposal

(UFP-QAPP Manual Section 3.4 – Worksheet #26 & #27)

Sampling Organization: AECOM
Laboratory: Pace
Method of sample delivery (shipper/carrier): FedEx
Number of days from reporting until sample disposal: 60 Days

Activity	Organization and title or position of person responsible for the activity	SOP reference
Sample labeling	AECOM	SOP 3-03 *Recordkeeping, Sample Labeling, and Chain of Custody*
CoC form completion	AECOM	
Packaging	AECOM	SOP 3-04 *Sample Handling, Storage, and Shipping*
Shipping coordination	AECOM	
Sample receipt, inspection, and log-in	Pace	ME0013H-17 *Sample Receiving*
Sample custody and storage	Pace	
Sample disposal	Pace	ME0012A-09 *Hazardous and Non-Hazardous Laboratory Waste Management Plan*

Notes:
AECOM = AECOM Technical Services, Inc.
Pace = Pace South Carolina
CoC = chain of custody
SOP = Standard operating procedure

SAP Worksheet #20 Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4 – Worksheet #28)

Matrix	Soil
Analytical Group	LC-MS-MS (PFAS)
Analytical Method/ SOP Reference	QSM Table B-15 / ME003NI

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQIs	MPCs
Soil and Sediment Sample Preparation	Each sample and associated batch QC samples.	Entire sample received by the laboratory must be homogenized prior to subsampling.	NA	Analyst, Supervisor	Representative-ness	Same as Method/SOP QC Acceptance Limits.
Sample Cleanup Procedure using ENVI-Carb or equivalent	Each sample and associated batch QC samples. Not applicable to AFFF and AFFF Mixture samples.	ENVI-Carb™ or equivalent must be used on each sample and batch QC sample.	NA	Analyst, Supervisor	Representative-ness	Same as Method/SOP QC Acceptance Limits.
Sample PFAS Identification	All analytes detected in a sample	The chemical derivation of the ion transitions must be documented. A minimum of two ion transitions (Precursor→quant ion and precursor→ confirmation ion) and the ion transitions ratio per analyte are required for confirmation. Exception is made for analytes where two transitions do not exist (PFBA, PFPeA, 9Cl-PF3ONS, 11Cl-PF3OUDS, PFOSA, MeFOSE, EtFOSE).	NA	Analyst, Supervisor	Comparability	Same as Method/SOP QC Acceptance Limits.

		Documentation of the primary and confirmation transitions and the ion ratios is required. In-house acceptance criteria for evaluation of ion ratios must be used and must not exceed 50-150%. Signal to Noise Ratio (S/N) must be ≥10 for all ions used for quantification and must be ≥3 for all ions used for confirmation. Quant ion and confirmation ion must be present and must maximize simultaneously (±2 seconds).				
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA	Analyst, Supervisor	Comparability	Same as Method/SOP QC Acceptance Limits.
Retention Time (RT) window width	Every field sample standard, blank, and QC sample.	RT of each analyte and EIS analyte must fall within 0.4 minutes of the predicted retention times from a daily calibration verification or, on days when ICAL is performed, from the midpoint standard of the ICAL. Analytes must elute within 0.1 minutes of the associated EIS. This criterion applies only to	Correct problem and reanalyze samples.	Analyst, Supervisor	Comparability	Same as Method/SOP QC Acceptance Limits.

		analyte and labeled analog pairs.				
Extracted Internal Standard Analytes	Every field sample, standard, blank, and QC sample.	Added to solid sample prior to extraction. Extracted Internal Standard Analyte recoveries must be within 50% to150% of ICAL midpoint standard area or area measured in the initial CCV on days when an ICAL is not performed	Correct problem. If required, re-extract and reanalyze associated field and QC samples. If recoveries are acceptable for QC samples, but not field samples, the field samples must be re-extracted and analyzed (greater dilution may be needed). Samples may be re-extracted and analyzed outside of hold times, as necessary for corrective action associated with QC failure.	Analyst, Supervisor	Accuracy	Same as Method/SOP QC Acceptance Limits.
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	Correct problem. If required, re-extract and reanalyze MB and all QC samples and field samples processed with the contaminated blank. Samples may be re-extracted and analyzed outside of hold times, as necessary for corrective action associated with QC failure.	Analyst, Supervisor	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
Laboratory Control Sample (LCS)	One per preparatory batch.	Blank spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. A laboratory must use the DoD/DOE QSM Appendix C Limits or laboratory SOP limits if not present	Correct problem, then re-extract and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available. Samples may be re-extracted and analyzed outside of hold times, as necessary for corrective action associated with QC failure.	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Matrix Spike (MS)	One per preparatory batch.	Sample spiked with all analytes at a concentration ≥ LOQ and	For matrix evaluation only. If MS results are outside the limits, the data shall be	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC

		≤ the mid-level calibration concentration. A laboratory must use the DoD/DOE QSM Appendix C Limits or laboratory SOP limits if not present	evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error).			Acceptance Limits.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	For MSD: Sample spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. A laboratory must use the DoD/DOE QSM Appendix C Limits or laboratory SOP limits if not present. RPD ≤ 30% (between MS and MSD or sample and MD).	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error).	Analyst, Supervisor	Accuracy/Bias/ Precision	Same as Method/SOP QC Acceptance Limits.

Matrix	Soil
Analytical Group	GC/MS (VOC + BTEX)
Analytical Method/ SOP Reference	8260D/ ME0012X

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQIs	MPCs
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, re-prepare and reanalyze MB and all samples processed with the contaminated blank.	Analyst, Supervisor	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.

Matrix	Soil
Analytical Group	GC/MS (VOC + BTEX)
Analytical Method/ SOP Reference	8260D/ ME0012X

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQIs	MPCs
Laboratory Control Sample (LCS)	One per preparatory batch.	Sample spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. Use in-house limits	Correct problem, then re-prepare and reanalyze LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Internal standards (IS)	Every field sample, standard and QC sample.	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV can be used.	Inspect mass spectrometer and GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Matrix Spike (MS)	One per preparatory batch.	Sample spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. Use in-house LCS limits	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Soil
Analytical Group	GC/MS (VOC + BTEX)
Analytical Method/ SOP Reference	8260D/ ME0012X

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQIs	MPCs
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	For MSD: One per preparatory batch.	For MSD: Sample spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. Use in-house LCS limits RPD ≤ 30% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Surrogate Spike	All field and QC samples.	Use in-house limits	Correct problem, then re-prepare and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Soil
Analytical Group	GC/MS (SVOC)
Analytical Method/ SOP Reference	8270E /ME0014Q 8270D SIM/ME0014Q

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQIs	MPCs
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, re-prepare and reanalyze MB and all samples processed with the contaminated blank.	Analyst, Supervisor	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
Laboratory Control Sample (LCS)	One per preparatory batch.	Sample spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. Use in-house limits	Correct problem, then re-prepare and reanalyze LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Soil
Analytical Group	GC/MS (SVOC)
Analytical Method/ SOP Reference	8270E /ME0014Q 8270D SIM/ME0014Q

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQIs	MPCs
Internal standards (IS)	Every field sample, standard and QC sample.	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV can be used.	Inspect mass spectrometer and GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Matrix Spike (MS)	One per preparatory batch.	Sample spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. Use in-house LCS limits	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	For MSD: One per preparatory batch.	For MSD: Sample spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. Use in-house LCS limits RPD ≤ 30% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Soil
Analytical Group	GC/MS (SVOC)
Analytical Method/ SOP Reference	8270E /ME0014Q 8270D SIM/ME0014Q

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQIs	MPCs
Surrogate Spike	All field and QC samples.	Use in-house limits	Correct problem, then re-prepare and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Soil
Analytical Group	TPH- GRO
Analytical Method/ SOP Reference	8015C & ME00138

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQIs	MPCs
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, re-prepare and reanalyze MB and all samples processed with the contaminated blank.	Analyst, Supervisor	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.

Matrix	Soil
Analytical Group	TPH- GRO
Analytical Method/ SOP Reference	8015C & ME00138

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQIs	MPCs
Laboratory Control Sample (LCS)	One per preparatory batch.	Sample spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. Use in-house limits	Correct problem, then re-prepare and reanalyze LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Matrix Spike (MS)	One per preparatory batch.	Sample spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. Use in-house LCS limits	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	For MSD: One per preparatory batch.	For MSD: Sample spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. Use in-house LCS limits RPD ≤ 30% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Soil
Analytical Group	TPH- GRO
Analytical Method/ SOP Reference	8015C & ME00138

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQIs	MPCs
Surrogate Spike	All field and QC samples.	Use in-house limits	Correct problem, then re-prepare and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Soil
Analytical Group	TPH-DRO
Analytical Method/ SOP Reference	8015C/ME00137

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQIs	MPCs
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, re-prepare and reanalyze MB and all samples processed with the contaminated blank.	Analyst, Supervisor	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.

Matrix	Soil
Analytical Group	TPH-DRO
Analytical Method/ SOP Reference	8015C/ME00137

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQIs	MPCs
Laboratory Control Sample (LCS)	One per preparatory batch.	Sample spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. Use in-house limits	Correct problem, then re-prepare and reanalyze LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Matrix Spike (MS)	One per preparatory batch.	Sample spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. Use in-house LCS limits	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	For MSD: One per preparatory batch.	For MSD: Sample spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. Use in-house LCS limits RPD ≤ 30% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Soil
Analytical Group	TPH-DRO
Analytical Method/ SOP Reference	8015C/ME00137

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQIs	MPCs
Surrogate Spike	All field and QC samples.	Use in-house limits	Correct problem, then re-prepare and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Soil
Analytical Group	GC (Pesticides)
Analytical Method/ SOP Reference	8081B/ME0019A

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQIs	MPCs
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, re-prepare and reanalyze MB and all samples processed with the contaminated blank.	Analyst, Supervisor	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
Laboratory Control Sample (LCS)	One per preparatory batch.	Sample spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. Use in-house limits	Correct problem, then re-prepare and reanalyze LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Matrix Spike (MS)	One per preparatory batch.	Sample spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. Use in-house LCS limits	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Soil
Analytical Group	GC (Pesticides)
Analytical Method/ SOP Reference	8081B/ME0019A

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQIs	MPCs
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	For MSD: One per preparatory batch.	For MSD: Sample spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. Use in-house LCS limits RPD ≤ 30% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Surrogate Spike	All field and QC samples.	Use in-house limits	Correct problem, then re-prepare and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Soil
Analytical Group	GC (Pesticides)
Analytical Method/ SOP Reference	8081B/ME0019A

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQIs	MPCs
Confirmation of positive results (second column)	All positive results must be confirmed	Calibration and QC criteria for second column are the same as for initial or primary column analysis. Results between primary and secondary column RPD ≤ 40%.	NA.	NA.	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Soil
Analytical Group	HR GC/MS
Analytical Method/ SOP Reference	8290/ENV-SOP-MIN4-0026

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQIs	MPCs
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, re-prepare and reanalyze MB and all samples processed with the contaminated blank.	Analyst, Supervisor	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
Laboratory Control Sample (LCS)	One per preparatory batch.	Sample spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. Use in-house limits	Correct problem, then re-prepare and reanalyze LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Matrix Spike (MS)	One per preparatory batch.	Sample spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. Use in-house LCS limits	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Soil
Analytical Group	HR GC/MS
Analytical Method/ SOP Reference	8290/ENV-SOP-MIN4-0026

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQIs	MPCs
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	For MSD: One per preparatory batch.	For MSD: Sample spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. Use in-house LCS limits RPD ≤ 30% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Sample Estimated Maximum Possible Concentration (EMPC)	Every sample with a response S/N ≥ 2.5 for both quantitation ions.	Identification criteria per method must be met, and the S/N of response for both quantitation ions must be ≥ 2.5.	NA.	Analyst, Supervisor	Comparability	Same as Method/SOP QC Acceptance Limits.

Matrix	Soil
Analytical Group	HR GC/MS
Analytical Method/ SOP Reference	8290/ENV-SOP-MIN4-0026

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQIs	MPCs
Sample 2,3,7,8TCDD toxicity equivalents (TEQ) concentration	All positive detections.	Per method.	NA.	NA.	Comparability	Same as Method/SOP QC Acceptance Limits.

Matrix	Soil
Analytical Group	Metals
Analytical Method/ SOP Reference	6010D/ ME001FJ-08 & ENV-SOP-MTJL-0215

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQIs	MPCs
Method Blank (MB)	One per preparatory batch.	The absolute values of all analytes must be < ½ LOQ or < 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Analyst, Supervisor	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
Laboratory Control Sample (LCS)	One per preparatory batch.	Use in-house LCS limits	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Soil
Analytical Group	Metals
Analytical Method/ SOP Reference	6010D/ ME001FJ-08 & ENV-SOP-MTJL-0215

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQIs	MPCs
MS	One per preparatory batch.	Use in-house limits.	Examine the project- specific requirements. Contact the client as to additional measures to be taken. For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error).	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Soil
Analytical Group	Metals
Analytical Method/ SOP Reference	6010D/ ME001FJ-08 & ENV-SOP-MTJL-0215

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQIs	MPCs
MSD or MD	One per preparatory batch.	Use in-house limits. MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and MD). For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the reporting limit.	Examine the project- specific requirements. Contact the client as to additional measures to be taken. For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative. The data shall be evaluated to determine the source of difference.	Analyst, Supervisor	Precision/ Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Dilution Test	One per preparatory batch if MS or MSD fails. Only applicable for samples with concentrations > 50 X reporting limit (prior to dilution). Use along with MS/MSD or post digestion spike (PDS) data to confirm matrix effects.	Five-fold dilution must agree within ± 10% of the original measurement.	No specific corrective action unless required by the project. For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Soil
Analytical Group	Metals
Analytical Method/ SOP Reference	6010D/ ME001FJ-08 & ENV-SOP-MTJL-0215

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQIs	MPCs
PDS Addition	One per preparatory batch if MS or MSD fails (using the same sample as used for the MS/MSD if possible). Criteria apply for samples with concentrations < 50 X reporting limit prior to dilution.	Recovery within 80-120%.	No specific corrective action unless required by the project. For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Method of Standard Additions (MSA)	When dilution and post digestion spike fails. Document use of MSA in the Case Narrative.	NA.	NA.	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

SAP Worksheet #21 Data Verification and Validation (Steps I and IIa/IIb) Process Table

(UFP-QAPP Manual Section 5.2.1, UFP-QAPP Manual Section 5.2.2, Figure 37 UFP-QAPP Manual, Table 9 UFP-QAPP Manual – Worksheets #34, 35, 36)

Data Review Input	Description	Responsible for Verification (name, organization)	Step I/IIa/IIb [1]	Internal/ External
Sample log sheets, chain of custody forms, SAP, and laboratory sample login documentation	The FOL will verify that samples were correctly identified, chain of custody records are legible, data will be traceable to the corresponding samples, the samples listed in **Worksheets #11 and #13** were collected from intended locations, and the correct sampling and analytical methods/SOPs were assigned to samples listed on the chain of custody record. The PM will verify that the sampling plan was implemented and carried out as written and will make sure that any significant deviations are documented in the project report.	FOL and PM, AECOM	I	Internal
Chain-of-custody forms	Verify that the chain-of-custody form is complete and accurate; and was signed and dated by the sampler relinquishing the samples and by the laboratory receiving the samples. Resolve discrepancies, if possible. Alert the AECOM PM verbally or via email if discrepancies are unresolvable.	FOL, AECOM	I	Internal
Chain-of-custody forms	Verify sample shipment completeness against the chain-of-custody record, verify proper sample preservation/integrity, sign to indicate receipt, note any discrepancies, and correct them as necessary. Notify the AECOM FOL or PM of any deviations from sample shipping requirements such as damaged sample containers, or inappropriate temperature or pH. Note uncorrectable discrepancies in the data package case narrative.	Laboratory sample custodian, SGS	I	External

Data Review Input	Description	Responsible for Verification (name, organization)	Step I/IIa/IIb [1]	Internal/ External
Analytical calibration standards	Verify that standards are traceable and meet contract, method, and procedural requirements, as applicable, and include certificates of analysis in the laboratory data package to document the traceability. If discrepancies in traceability are found, bring the discrepancies to the laboratory PM attention for correction.	Laboratory analyst, SGS	I	Internal
SAP, analytical SOPs, and analytical data packages	Verify that the correct analytical methods/SOPs were applied. Establish that method QC samples were analyzed and in control as listed in the analytical SOPs. If method QC is not in control, the Laboratory PM will contact the AECOM Project Chemist or PM verbally or via email for guidance prior to laboratory data package preparation.	Laboratory PM, SGS	I	Internal
Laboratory analytical data package	Verify the analytical data package for completeness and accuracy, including certificates of analysis for calibration and check standards. The laboratory QAM will sign the case narrative for each data package.	Laboratory QAM, SGS	I	Internal
Laboratory analytical data package	Review chain-of-custody records to ensure that the required analytical samples were collected, appropriate sample identifications were used, and correct analytical methods were applied to each sample. Verify the analytical data package for completeness and accuracy, including certificates of analysis for calibration and check standards. Obtain missing data package elements from the laboratory. Document unrecoverable elements, if any, in the data validation report submitted to the AECOM PM and alert the project chemist or PM.	Data validator, AECOM	I/IIa	External

Data Review Input	Description	Responsible for Verification (name, organization)	Step I/IIa/IIb [1]	Internal/ External
EDDs/Analytical data packages	Verify 100% of EDD results for accuracy and completeness against hard copy data package and chain of custody records at the start of validation. If required elements are missing, obtain missing elements form the laboratory before completing the validation. If any element cannot be obtained, document the omission in the DV report and identify the missing elements to the AECOM project chemist or PM as early as possible.	Data validator, AECOM	I/IIa	External
Sample shipment and storage conditions; and holding times for representativeness	Verify that sample shipping and storage conditions satisfy **Worksheet #13** requirements. Document deviations from requirements in the DV report and notify the AECOM project chemist or PM if deviations from the SAP requirements are serious enough to warrant data rejection. Document findings in the DV report.	Data validator, AECOM	I/IIa	External
QC samples/MPC compliance	Ensure that the scheduled laboratory and field QC samples were submitted for analysis and that the MPCs listed in SAP **Worksheets #11, #16, and #18** were met for all field samples and QC samples. Document findings in the DV report. Evaluate sample results for laboratory contamination and qualify false detections using the laboratory method/preparation blank summaries. Qualify analyte concentrations between the MDL and the reporting limit as estimated ("*J*" qualifier). Replace laboratory flags with validation qualifiers on validated data in accordance with the laboratory data validation process described below and document findings in the data validation report. Retain laboratory flags in the database and provide them in the DV reports to document data as received from the laboratory.	Data validator, AECOM	IIa/IIb	External

Data Review Input	Description	Responsible for Verification (name, organization)	Step I/IIa/IIb [1]	Internal/ External
Field and laboratory duplicate analyses for precision	Verify field sampling precision by checking RPDs for field duplicate samples. Verify laboratory precision by checking RPDs or %D values from calibrations, laboratory duplicates, MS/MSDs, and LCS/laboratory control sample duplicates (LCSDs). Ensure compliance with MPC accuracy and precision goals listed in **Worksheets #11 and #18**. Document findings in the DV report.	Data validator, AECOM	I/IIb	External
SAP/Laboratory data packages/ EDDs	Conduct EPA Stage 2A data validation on 100% of the definitive laboratory data generated by the selected methods using QC criteria listed in this SAP. This does not apply to TCLP and Waste Characterization Parameters Apply validation qualifiers in accordance with logic provided in the National Functional Guidelines (NFG) for Inorganic Superfund (EPA, 2020a) or Organic Superfund (EPA, 2020b) data review (including EPA Region-specific requirements, if applicable). Document findings in the DV report.	Data validator, AECOM	IIa/IIb	External

Usability Assessment

(UFP-QAPP Manual Section 2.6.2 and 5.2.3 Worksheet #37)

After data validation, the data and data quality will be reviewed to determine whether sufficient data of acceptable quality are available for decision making. The AECOM PM and designees will be responsible for conducting this data usability assessment. Summary statistics for target analytes such as sampling completeness (Cs), analytical data completeness (Ca), maximum concentration, minimum concentration, number of samples with non-detected results, number of samples with detected results, and the proportion of samples with detected and non-detected results will be compiled at the discretion of the AECOM PM. The project team will consider whether any missing or rejected data have reduced the Cs and Ca to less than the associated goals and will consider factors that affect sample integrity such as holding times, preservation, and storage conditions. The assessment findings will be presented to the NASA PM, EPA, and VDEQ.

The goal for Cs is 90% and for Ca it is 90%. These values will be calculated as follows:

$$\%Cs = \frac{No.\,of\ Valid\ Samples}{No.\,of\ Planned\ Samples} x100\%$$

$$\%Ca = \frac{No.\,of\ Valid\ Analytical\ Results}{\text{No. of Planned Analytical Results}} x100\%$$

Field and laboratory precision will be evaluated for each matrix, analytical fraction, and concentration level (as applicable), with the expectation that laboratory duplicate results which will be no less precise than field duplicate results.

Precision will be computed in terms of RPD as follows:

$$RPD = \frac{200 * |\text{Result}_A - Result_B|}{(\text{Result}_A + Result_B)}$$

Accuracy will be computed in terms of percent recovery (%R) as follows for MS/MSD samples:

$$\%R = \frac{Amount\ in\ Spiked\ Sample - Amount\ in\ Sample}{Known\ Amount\ Added} \text{ X } 100\%$$

The %R calculation for laboratory control samples and surrogate spikes will be as follows:

$$\%R = \frac{Experimental\ Concentration}{Certified\ or\ Known\ Concentration} \text{ x } 100$$

Data comparability and representativeness will be evaluated by reviewing sample collection processes and associated documentation; and by comparing overall precision and bias among data sets for each matrix and analytical fraction. This will not require quantitative comparisons unless professional judgment of the AECOM project chemist indicates that such quantitative analysis is required. If comparability or

representativeness deficiencies are identified, limitations on the data will be described in the project report. If data gaps exist, they will be identified and, if appropriate, the project team will take them into consideration if additional work is required to meet project objectives.

Data validators apply "*X*" qualifiers to results that are seriously compromised and potentially unusable from a quality perspective. The project team will consider whether any missing or "*X*"-qualified data have compromised the ability to make decisions or to make decisions with the desired level of confidence. In these cases, "X"-qualified data will be qualified with an "*R*" qualifier if rejected. The data will be evaluated to determine whether available data can compensate for missing or rejected data.

The project report will identify and describe the data usability limitations and suggest resampling or other corrective actions, if necessary. If applicable, this will include a description of unacceptable levels of bias/contamination, non-representativeness of data, or poor data comparability that could affect the accuracy and usability of reported results. These discussions may be specific to a matrix, analytical fraction, or other logical grouping of results

SAP Worksheet #22 References

AECOM, 2022. Final Supplemental Field Investigation Data Report Revision 2, Human Health Risk Assessment update, and Ecological Risk Assessment update, Project 15 Construction Debris Landfill, Wallops Flight Facility Formerly Used Defense Site, Wallops Island, Virginia. June 2022.

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Richardson, D.L. 1994. Hydrogeology and Analysis of the Ground-Water Flow System of the Eastern Shore, Virginia. U.S. Geological Survey Water-Supply Paper 2401.SAIC. 2003. Limited Site Investigation Report (Final), NASA Wallops Flight Facility, Accomack County, Virginia. May 2003.

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USATEC. 2000. *Wallops Flight Facility Main Base Ground Features: GIS Based Historical Photographic Analysis. 1938-1980.* November 2000.

Versar, Inc. 2012. *Draft Feasibility Study Report, Construction Debris Landfill, NASA Wallops Flight Facility, Accomack County, Virginia*. 2012.

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Wallace, Harold D., 1997. Wallops Station and the Creation of An American Space Program. The NASA History Series. NASA History Office, Office of Policy and Plans, Washington, D.C. Full text available at http://www.archive.org/stream/wallopsstationcr00wallrich.

APPENDIX A

FIELD STANDARD OPERATING PROCEDURES

Utility Clearance

Procedure 3-01

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes the process for determining the presence of subsurface utilities and other cultural features at locations where planned site activities involve the physical disturbance of subsurface materials.

1.2 This procedure is the Program-approved professional guidance for work performed by AECOM under the client contract.

1.3 The procedure applies to the following activities: soil gas surveying, excavating, trenching, drilling of borings and installation of monitoring and extraction wells, use of soil recovery or slide-hammer hand augers, and all other intrusive sampling activities.

1.4 The primary purpose of the procedure is to minimize the potential for damage to underground utilities and other subsurface features, which could result in physical injury, disruption of utility service, or disturbance of other subsurface cultural features.

1.5 If there are procedures, whether it be from AECOM, state, and/or federal, that are not addressed in this SOP and are applicable to utility clearance, those procedures should be added as an appendix to the project specific Quality Assurance Project Plan (QAPP).

1.6 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

2.1 Field and subcontractor personnel shall adhere to a site-specific health and safety plan (HASP).

3.0 Terms and Definitions

3.1 Utility

For the proposes of this SOP, a utility is defined as a manmade underground line or conduit, cable, pipe, vault or tank that is, or was, used for the transmission of material or energy (e.g., gas, electrical, telephone, steam, water or sewage, product transfer lines, or underground storage tanks).

3.2 As-Built Plans

As-built plans are plans or blueprints depicting the locations of structures and associated utilities on a property.

3.3 One-Call

The Utility Notification Center is the one-call agency for nationwide call before you dig. The Utility Notification Center is open 24 hours a day and accepts calls from anyone planning to dig. The phone number 811 is the designated call before you dig phone number that directly connects you to your local one-call center. Additional information can be found at www.call811.com.

Calling before you dig ensures that any publicly owned underground lines will be marked so that you can dig around them safely. Having the utility lines marked not only prevents accidental damage to the lines but prevents property damage and personal injuries that could result in breaking a line.

The following information will need to be provided when a call is placed to One-Call:

- Your name, phone number, company name (if applicable), and mailing address.
- What type or work is being done.
- Who the work is being done for.
- The county and city the work is taking place in.
- The address or the street where the work is taking place.
- Marking instructions, (specific instructions as to where the work is taking place).

Under normal circumstances it takes between 2 to 5 days from the time you call (not counting weekends or holidays) to have the underground lines marked. Because these laws vary from state to state, exactly how long it will take depends on where your worksite is located. You will be given an exact start time and date when your locate request is completed, which will comply with the laws in your area.

In the event of an emergency (any situation causing damage to life or property, or a service outage), lines can be marked sooner than the original given time if requested.

3.4 Toning

Toning is the process of surveying an area utilizing one or more surface geophysical methods to determine the presence or absence of underground utilities. Typically, toning is conducted after identifying the general location of utilities and carefully examining all available site utility plans. Each location is marked according to the type of utility being identified. In addition, areas cleared by toning are flagged or staked to indicate that all identified utilities in a given area have been toned.

4.0 Training and Qualifications

4.1 The **Task Order (TO) Manager** is responsible for verifying that these utility locating procedures are performed prior to the initiation of active subsurface exploration.

4.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

4.3 The **Field Manager** is responsible for ensuring that all utility locating activities are performed in accordance with this procedure.

4.4 All **Field Personnel** are responsible for the implementation of this procedure.

5.0 Equipment and Supplies

5.1 Equipment and supplies necessary for locating subsurface utilities will be provided by the subcontractor; however, the project **Field Manager/Field Personnel** will provide any additional equipment and supplies as needed as well as maintain information regarding the utility clearance activities in the field logbook.

6.0 Procedure

Proceed with the following steps where subsurface exploration will include excavations, drilling, or any other subsurface investigative method that could damage utilities at a site. In addition to the steps outlined below, always exercise caution while conducting subsurface exploratory work.

6.1 Prepare Preliminary Site Plan

- Prepare a preliminary, scaled site plan depicting the proposed exploratory locations as part of the project specific QAPP. Include as many of the cultural and natural features as practical in this plan.

6.2 Review Background Information

- Search existing plan files to review the as-built plans to identify the known location of utilities at the site. Plot the locations of utilities identified onto a preliminary, scaled site plan. Inform the TO Manager if utilities lie within close proximity to a proposed exploration or excavation location. The TO Manager will determine if it is necessary to relocate proposed sampling or excavation locations.
- Include the utility location information gathered during previous investigations (e.g., remedial investigation or remedial site evaluation) in the project design documents for removal or remedial actions. In this manner, information regarding utility locations collected during implementation of a TO can be shared with the subcontractor during implementation of a particular task order. In many instances, this will help to reduce the amount of additional geophysical surveying work the subcontractor may have to perform.
- Conduct interviews with onsite and facility personnel familiar with the site to obtain additional information regarding the known and suspected locations of underground utilities. In addition, if appropriate, contact shall be made with local utility companies to request their help in locating underground lines. Pencil in the dimensions, orientation, and depth of utilities, other than those identified on the as-built plans, at their approximate locations on the preliminary plans. Enter the type of utility, the personnel who provided the information, and the date the information was provided into the field log.
- During the pre-field work interviewing process, the interviewer will determine which site personnel should be notified in the event of an incident involving damage to existing utilities. Record this information in the field logbook with the corresponding telephone numbers and addresses.

6.3 Site Visit/Locate Utilities/Toning

- Prior to the initiation of field activities, the Field Task Manager or similarly qualified field personnel shall visit the site and note existing structures and evidence of associated utilities, such as fire hydrants, irrigation systems, manhole and vault box covers, standpipes, telephone switch boxes, free-standing light poles, gas or electric meters, pavement cuts, and linear depression. Compare notes of the actual site configuration to the preliminary site plan. Note deviations in the field logbook and on the preliminary site plan. Accurately locate or survey and clearly mark with stakes, pins, flags, paint, or other suitable devices all areas where subsurface exploration is proposed. These areas shall correspond with the locations drawn on the preliminary site plan.
- Following the initial site visit by the Field Task Manager, a trained utility locating subcontractor will locate, identify, and tone all utilities depicted on the preliminary site plan. The Field Task Manager or similarly qualified field personnel shall visit the site and identify the areas of subsurface disturbance with white spray paint, chalk, white pin flags or some other easily identifiable marking. The utility locator should utilize appropriate sensing equipment to attempt to locate utilities that might not have appeared on the as-built plans. At a minimum, the utility subcontractor should utilize a metal detector and/or magnetometer; however, it is important to consider the possibility that non-metallic utilities or tanks might be present at the site. Use other appropriate surface geophysical methods such as Ground Penetrating Radar, Radio detection, etc. as appropriate. Clear proposed exploration areas of all utilities in the immediate area where subsurface exploration is proposed. Clearly tone all anomalous areas. Clearly identify all toned areas on the preliminary site plan. All utilities near the area of subsurface disturbance should also be marked out by the utility subcontractor using the universal colors for subsurface utilities (i.e., red – electric; blue – water; green – sewer; yellow – gas; etc.). After toning the site and plotting all known or suspected buried utilities on the preliminary site plan, the utility locator shall provide the Field Task Manager with a copy of the completed preliminary site plan. Alternatively, the Field Task Manager or designee shall document the results of the survey on the preliminary site plan.
- Report to the Field Task Manager anomalous areas detected and toned that are in close proximity to the exploration or excavation areas. The Field Task Manager shall determine the safe distance to maintain from the known or suspected utility. It may be necessary to relocate the proposed

exploration or excavation areas. If this is required, the Field Task Manager or designee shall relocate them and clearly mark them using the methods described above. Completely remove the markings at the prior location. Plot the new locations on the site plan and delete the prior locations from the plan. In some instances, such as in areas extremely congested with subsurface utilities, it may be necessary to dig by hand or use techniques such as air knife to determine the location of the utilities.

6.4 Prepare Site Plan

- Prior to the initiation of field activities, draft a final site plan that indicates the location of subsurface exploration areas and all known or suspected utilities present at the site. Provide copies of this site plan to the client, the TO Manager, and the subcontractor who is to conduct the subsurface exploration/excavation work. Review the site plan with the client to verify its accuracy prior to initiating subsurface sampling activities.

7.0 Quality Control and Assurance

7.1 Utility locating must incorporate quality control measures to ensure conformance to these and the project requirements.

8.0 Records, Data Analysis, Calculations

8.1 A bound field logbook will be kept detailing all activities conducted during the utility locating procedure.

8.2 The logbook will describe any changes and modifications made to the original exploration plan. The trained utility locator shall prepare a report and keep it in the project file. Also, a copy of the final site plan will be kept in the project file.

9.0 Attachments or References

Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual.* Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

Author	Reviewer	Revisions (Technical or Editorial)
Caryn DeJesus Senior Scientist	Bob Shoemaker Senior Scientist	Rev 0 – Initial Issue (June 2012)
Ken O'Donnell, PG Geologist	Claire Mitchell, PE, PMP Senior Engineer	Rev 1 – PFAS sampling update (July 2019)

Logbooks

Procedure 3-02

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes the activities and responsibilities pertaining to the identification, use, and control of logbooks and associated field data records.

1.2 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

2.1 In order to keep the logbook clean, store it in a clean location and use it only when outer gloves used for PPE have been removed.

3.0 Terms and Definitions

3.1 Logbook

A logbook is a bound field notebook with consecutively numbered, non-water-repellent binding or pages that is clearly identified with the name of the relevant activity, the person assigned responsibility for maintenance of the logbook, and the beginning and ending dates of the entries.

3.2 Data Form

A data form is a predetermined format utilized for recording field data that may become, by reference, a part of the logbook (e.g., soil boring logs, trenching logs, surface soil sampling logs, groundwater sample logs, and well construction logs are data forms).

4.0 Training and Qualifications

4.1 The **Task Order (TO) Manager** or **designee** is responsible for determining which team members shall record information in field logbooks and for obtaining and maintaining control of the required logbooks. The **TO Manager** shall review the field logbook on at least a monthly basis. The **TO Manager** or **designee** is responsible for reviewing logbook entries to determine compliance with this procedure and to ensure that the entries meet the project requirements.

4.2 A knowledgeable individual such as the **Field Manager**, **TO Manager**, or **Program Quality Manager** shall perform a technical review of each logbook at a frequency commensurate with the level of activity (weekly is suggested, or, at a minimum, monthly). Document these reviews by the dated signature of the reviewer on the last page or page immediately following the material reviewed.

4.3 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

4.4 The **Field Manager** is responsible for ensuring that all **field personnel** follow these procedures and that the logbook is completed properly and daily. The **Field Manager** is also responsible for submitting copies to the **TO Manager**, who is responsible for filing them and submitting a copy (if required by the TO Statement of Work).

4.5 The **logbook user** is responsible for recording pertinent data into the logbook to satisfy project requirements and for attesting to the accuracy of the entries by dated signature. The **logbook user** is also responsible for safeguarding the logbook while having custody of it.

4.6 All **field personnel** are responsible for the implementation of this procedure.

5.0 Equipment and Supplies

5.1 Field logbooks shall be bound field notebooks with non-water-repellent binding or pages.

5.2 Ballpoint pens shall have indelible black ink.

6.0 Procedure

6.1 The field logbook serves as the primary record of field activities. Make entries chronologically and in sufficient detail to allow the writer or a knowledgeable reviewer to reconstruct the applicable events. Store the logbook in a clean location and use it only when outer gloves used for personal protective equipment (PPE) have been removed.

6.2 Individual data forms may be generated to provide systematic data collection documentation. Entries on these forms shall meet the same requirements as entries in the logbook and shall be referenced in the applicable logbook entry. Individual data forms shall reference the applicable logbook and page number. At a minimum, include names of all samples collected in the logbook even if they are recorded elsewhere.

6.3 Enter field descriptions and observations into the logbook, as described in Attachment 1, using indelible black ink.

6.4 Typical information to be entered includes the following:

- Dates (month/day/year) and times (military) of all on-site activities and entries made in logbooks/forms;
- Site name and description;
- Site location by longitude and latitude, if known;
- Weather conditions, including temperature and relative humidity;
- Fieldwork documentation, including site entry and exit times;
- Descriptions of, and rationale for, approved deviations from the Quality Assurance Project Plan (QAPP) or field sampling plan;
- Field instrumentation readings;
- Names, job functions, and organizational affiliations of on-site personnel;
- Photograph references;
- Site sketches and diagrams made on site;
- Identification and description of sample morphology, collection locations, and sample numbers;
- Sample collection information, including dates (month/day/year) and times (military) of sample collections, sample collection methods and devices, station location numbers, sample collection depths/heights, sample preservation information, sample pH (if applicable), analysis requested (analytical groups), etc., as well as chain of custody (CoC) information such as sample identification numbers cross-referenced to COC sample numbers;
- Sample naming convention;
- Field quality control (QC) sample information;
- Site observations, field descriptions, equipment used, and field activities accomplished to reconstruct field operations;

- Meeting information;
- Important times and dates of telephone conversations, correspondence, or deliverables;
- Field calculations;
- PPE level;
- Calibration records;
- Contractor and subcontractor information (address, names of personnel, job functions, organizational affiliations, contract number, contract name, and work assignment number);
- Equipment decontamination procedures and effectiveness;
- Laboratories receiving samples and shipping information, such as carrier, shipment time, number of sample containers shipped, and analyses requested; and
- User signatures.

6.5 The logbook shall reference data maintained in other logs, forms, etc. Correct entry errors by drawing a single line through the incorrect entry, then initialing and dating this change. Enter an explanation for the correction if the correction is more than for a mistake.

6.6 At least at the end of each day, the person making the entry shall sign or initial each entry or group of entries.

6.7 Enter logbook page numbers on each page to facilitate identification of photocopies.

6.8 If a person's initials are used for identification, or if uncommon acronyms are used, identify these on a page at the beginning of the logbook.

6.9 At least weekly and preferably daily, the **preparer** shall photocopy and retain the pages completed during that session for backup. This will prevent loss of a large amount of information if the logbook is lost.

7.0 Quality Control and Assurance

7.1 Review per Section 4.2 shall be recorded.

8.0 Records, Data Analysis, Calculations

8.1 Retain the field logbook as a permanent project record. If a particular TO requires submittal of photocopies of logbooks, perform this as required.

8.2 Deviations from this procedure shall be documented in field records. Significant changes shall be approved by the **Program Quality Manager**.

9.0 Attachments or References

9.1 Attachment 1 – Description of Logbook Entries

9.2 Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

Author	Reviewer	Revisions (Technical or Editorial)
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue
Ken O'Donnell, PG Geologist	Claire Mitchell, PE, PMP Senior Engineer	Rev 1 – PFAS sampling update (July 2019)

Attachment 1
Description of Logbook Entries

Logbook entries shall be consistent with Section A.1.4 *Field Documentation SOPs* of the UFP-QAPP Manual (DoD 2005) and contain the following information, as applicable, for each activity recorded. Some of these details may be entered on data forms, as described previously.

Name of Activity	For example, Asbestos Bulk Sampling, Charcoal Canister Sampling, Aquifer Testing.
Task Team Members and Equipment	Name all members on the field team involved in the specified activity. List equipment used by serial number or other unique identification, including calibration information.
Activity Location	Indicate location of sampling area as indicated in the field sampling plan.
Weather	Indicate general weather and precipitation conditions.
Level of PPE	Record the level of PPE (e.g., Level D).
Methods	Indicate method or procedure number employed for the activity.
Sample Numbers	Indicate the unique numbers associated with the physical samples. Identify QC samples.
Sample Type and Volume	Indicate the medium, container type, preservative, and the volume for each sample.
Time and Date	Record the time and date when the activity was performed (e.g., 0830/08/OCT/89). Use the 24-hour clock for recording the time and two digits for recording the day of the month and the year.
Analyses	Indicate the appropriate code for analyses to be performed on each sample, as specified in the WP.
Field Measurements	Indicate measurements and field instrument readings taken during the activity.
CoC and Distribution	Indicate CoC for each sample collected and indicate to whom the samples are transferred and the destination.
References	If appropriate, indicate references to other logs or forms, drawings, or photographs employed in the activity.
Narrative (including time and location)	Create a factual, chronological record of the team's activities throughout the day including the time and location of each activity. Include descriptions of general problems encountered and their resolution. Provide the names and affiliations of non-field team personnel who visit the site, request changes in activity, impact the work schedule, request information, or observe team activities. Record any visual or other observations relevant to the activity, the contamination source, or the sample itself. It should be emphasized that logbook entries are for recording data and chronologies of events. The logbook author must include observations and descriptive notations, taking care to be objective and recording no opinions or subjective comments unless appropriate.
Recorded by	Include the signature of the individual responsible for the entries contained in the logbook and referenced forms.
Checked by	Include the signature of the individual who performs the review of the completed entries.

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Recordkeeping, Sample Labeling, and Chain of Custody

Procedure 3-03

1.0 Purpose and Scope

1.1 The purpose of this standard operating procedure is to establish standard protocols for all field personnel for use in maintaining field and sampling activity records, writing sample logs, labeling samples, ensuring that proper sample custody procedures are utilized, and completing chain of custody (CoC) /analytical request forms.

1.2 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

Not applicable.

3.0 Terms and Definitions

3.1 Logbook

A logbook is a bound field notebook with consecutively numbered, non-water-repellent binding or pages that is clearly identified with the name of the relevant activity, the person responsible for maintenance of the logbook, and the beginning and ending dates of the entries.

3.2 Chain of Custody

A CoC is documentation of the process of custody control. Custody control includes possession of a sample from the time of its collection in the field to its receipt by the analytical laboratory, and through analysis and storage prior to disposal.

4.0 Training and Qualifications

4.1 The **Task Order (TO) Manager** is responsible for determining which team members shall record information in the field logbook and for checking sample logbooks and CoC forms to ensure compliance with these procedures. The **TO Manager** shall review CoC forms on a monthly basis at a minimum.

4.2 The **TO Manager** and **Program Quality Manager** are responsible for evaluating project compliance with the Project Procedures Manual.

4.3 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

4.4 The **Laboratory Project Manager** or **Sample Control Department Manager** is responsible for reporting any sample documentation or CoC problems to the **TO Manager** or **TO Laboratory Coordinator** within 24 hours of sample receipt.

4.5 The **Field Manager** is responsible for ensuring that all **field personnel** follow these procedures. The **TO Laboratory Coordinator** is responsible for verifying that the CoC/analytical request forms have been completed properly and match the sampling and analysis plan. The **TO Manager** or **TO Laboratory Coordinator** is responsible for notifying the **laboratory**, **data managers**, and **data validators** in writing if analytical request changes are required as a corrective action. These small changes are different from change orders, which involve changes to the scope of the subcontract with the laboratory and must be made in accordance with a respective contract (e.g., client remedial action contract).

4.6 All **field personnel** are responsible for following these procedures while conducting sampling activities. **Field personnel** are responsible for recording pertinent data into the logbook to satisfy project requirements and for attesting to the accuracy of the entries by dated signature.

5.0 Procedure

This procedure provides standards for documenting field activities, labeling the samples, documenting sample custody, and completing CoC/analytical request forms. The standards presented in this section shall be followed to ensure that samples collected are maintained for their intended purpose and that the conditions encountered during field activities are documented.

5.1 Recordkeeping

The field logbook serves as the primary record of field activities. Make entries chronologically and in sufficient detail to allow the writer or a knowledgeable reviewer to reconstruct each day's events. Field logs such as soil boring logs and ground-water sampling logs will also be used. These procedures are described in Procedure 3-02, *Logbooks*.

5.2 Sample Labeling

Affix a sample label with adhesive backing to each individual sample container. Place clear tape over each label (preferably prior to sampling) to prevent the labels from tearing off, falling off, being smeared, and to prevent loss of information on the label. Record the following information with a ballpoint pen or pre-printed text on each label:

- Project name or number (optional);
- CoC sample number;
- Date and time of collection;
- Sampler's initials;
- Matrix (optional);
- Sample preservatives (if applicable); and
- Analysis to be performed on sample (this shall be identified by the method number or name identified in the subcontract with the laboratory).

These labels may be obtained from the analytical laboratory or printed from a computer file onto adhesive labels.

5.3 Custody Procedures

For samples intended for chemical analysis, sample custody procedures shall be followed through collection, transfer, analysis, and disposal to ensure that the integrity of the samples is maintained. Maintain custody of samples in accordance with the U.S. Environmental Protection Agency (EPA) CoC guidelines prescribed in EPA *NEIC Policies and Procedures*, National Enforcement Investigations Center, Denver, Colorado, revised May 1986; EPA *RCRA Ground Water Monitoring Technical Enforcement Guidance Document* (TEGD); *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA* (EPA OSWER Directive 9355 3-01); Appendix 2 of the *Technical Guidance Manual for Solid Waste Water Quality Assessment Test (SWAT) Proposals and Reports*; and *Test Methods for Evaluating Solid Waste* (EPA SW-846)

A description of sample custody procedures is provided below.

5.3.1 Sample Collection Custody Procedures

According to the U.S. EPA guidelines, a sample is considered to be in custody if one of the following conditions is met:

- It is in one's actual physical possession or view;
- It is in one's physical possession and has not been tampered with (i.e., it is under lock or official seal);
- It is retained in a secured area with restricted access; and
- It is placed in a container and secured with an official seal such that the sample cannot be reached without breaking the seal.

Place custody seals on sample containers immediately after sample collection and on shipping coolers if the cooler is to be removed from the sampler's custody. Place custody seals in such a manner that they must be broken to open the containers or coolers. Label the custody seals with the following information:

- Sampler's name or initials; and
- Date and time that the sample/cooler was sealed.

These seals are designed to enable detection of sample tampering. An example of a custody seal is shown in Attachment 1.

Field personnel shall also log individual samples onto CoC forms (carbon copy or computer generated) when a sample is collected. These forms may also serve as the request for analyses. Procedures for completing these forms are discussed in Section 5.4, indicating sample identification number, matrix, date and time of collection, number of containers, analytical methods to be performed on the sample, and preservatives added (if any). The **samplers** will also sign the CoC form signifying that they were the personnel who collected the samples. The CoC form shall accompany the samples from the field to the laboratory. When a cooler is ready for shipment to the analytical laboratory, the **person delivering the samples for transport** will sign and indicate the date and time on the accompanying CoC form. One copy of the CoC form will be retained by the **sampler** and the remaining copies of the CoC form shall be placed inside a self-sealing bag and taped to the inside of the cooler. Each cooler must be associated with a unique CoC form. Whenever a transfer of custody takes place, **both parties** shall sign and date the accompanying carbon copy CoC forms, and the **individual relinquishing the samples** shall retain a copy of each form. One exception is when the samples are shipped; the **delivery service personnel** will not sign or receive a copy because they do not open the coolers. The **laboratory** shall attach copies of the completed CoC forms to the reports containing the results of the analytical tests. An example CoC form is provided in Attachment 2.

5.3.2 Laboratory Custody Procedures

The following custody procedures are to be followed by an **independent laboratory** receiving samples for chemical analysis; the procedures in their Naval Facilities Engineering Service Center-evaluated Laboratory Quality Assurance Plan must follow these same procedures. A **designated sample custodian** shall take custody of all samples upon their arrival at the analytical laboratory. The **custodian** shall inspect all sample labels and CoC forms to ensure that the information is consistent, and that each is properly completed. The **custodian** will also measure the temperature of the temperature blank in the coolers upon arrival using either a National Institute for Standards and Technology calibrated thermometer or an infra-red temperature gun. The **custodian** shall note the condition of the samples including:

- If the samples show signs of damage or tampering;
- If the containers are broken or leaking;
- If headspace is present in sample vials;
- If proper preservation of samples has occurred (made by pH measurement, except volatile organic compounds [VOCs] and purgeable total petroleum hydrocarbons [TPH] and temperature). The pH of VOC and purgeable TPH samples will be checked by the **laboratory analyst** after the sample aliquot has been removed from the vial for analysis; and
- If any sample holding times have been exceeded.

All of the above information shall be documented on a sample receipt sheet by the **custodian**.

Discrepancies or improper preservation shall be noted by the **laboratory** as an out-of-control event and shall be documented on an out-of-control form with corrective action taken. The out-of-control form shall be signed and dated by the **sample control custodian** and **any other persons** responsible for corrective action. An example of an out-of-control form is included as Attachment 4.

The **custodian** shall then assign a unique laboratory number to each sample and distribute the samples to secured storage areas maintained at 4 degrees Celsius (soil samples for VOC analysis are to be stored in a frozen state until analysis). The unique laboratory number for each sample, CoC sample number, client name, date and time received, analysis due date, and storage shall also be manually logged onto a sample receipt record and later entered into the laboratory's computerized data management system. The **custodian** shall sign the shipping bill and maintain a copy.

Laboratory personnel shall be responsible for the care and custody of samples from the time of their receipt at the laboratory through their exhaustion or disposal. Samples should be logged in and out on internal laboratory CoC forms each time they are removed from storage for extraction or analysis.

5.4 Completing CoC/Analytical Request Forms

CoC form/analytical request form completion procedures are crucial in properly transferring the custody and responsibility of samples from field personnel to the laboratory. This form is important for accurately and concisely requesting analyses for each sample; it is essentially a release order from the analysis subcontract.

Attachment 2 is an example of a generic CoC/analytical request form that may be used by **field personnel**. Multiple copies may be tailored to each project so that much of the information described below need not be handwritten each time. Attachment 3 is an example of a completed site-specific CoC/analytical request form, with box numbers identified and discussed in text below.

CoC forms tailored to each TO can be drafted and printed onto multi-ply forms. This eliminates the need to rewrite the analytical methods column headers each time. It also eliminates the need to write the project manager, name, and number; QC Level; TAT; and the same general comments each time.

Complete one CoC form per cooler. Whenever possible, place all VOC analyte vials into one cooler in order to reduce the number of trip blanks. Complete all sections and be sure to sign and date the CoC form. One copy of the CoC form must remain with the field personnel.

Box 2 **Bill To:** List the name and address of the person/company to bill only if it is not in the subcontract with the laboratory.

Box 3 **Sample Disposal Instructions:** These instructions will be stated in the Master Service Agreement or each TO statement of work with each laboratory.

Shipment Method: State the method of shipment (e.g., hand carry or air courier via FedEx or DHL).

Comments: This area shall be used by the field team to communicate observations, potential hazards, or limitations that may have occurred in the field or additional information regarding analysis (e.g., a specific metals list, samples expected to contain high analyte concentrations).

Box 4 **Cooler No.:** This will be written on the inside or outside of the cooler and shall be included on the CoC. Some laboratories attach this number to the trip blank identification, which helps track samples for VOC analysis. If a number is not on the cooler, field personnel shall assign a number, write it on the cooler, and write it on the CoC.

QC Level: Enter the reporting quality control (QC) requirements (e.g., Full Data Package, Summary Data Package).

Turnaround time (TAT): TAT will be determined by a sample delivery group (SDG), which may be formed over a 14-day period, not to exceed 20 samples. Once the SDG has been completed, standard TAT is 21 calendar days from receipt of the last sample in the SDG. Entering NORMAL or STANDARD in this field will be acceptable. If quicker TAT is required, it shall be in the subcontract with the laboratory and reiterated on each CoC to remind the laboratory.

Box 5 **Type of Containers:** Write the type of container used (e.g., 1-liter glass amber, for a given parameter in that column).

Preservatives: Field personnel must indicate on the CoC the correct preservative used for the analysis requested. Indicate the pH of the sample (if tested) in case there are buffering conditions found in the sample matrix.

Box 6 **Sample Identification (ID) Number:** This is typically a five-character alphanumeric identifier used by the contractor to identify samples. The use of this identifier is important since the laboratories are restricted to the number of characters they are able to use. Sample numbering shall be in accordance with the project-specific sampling and analysis plan.

Description (Sample ID): This name will be determined by the location and description of the sample, as described in the project-specific sampling and analysis plan. This sample identification should not be submitted to the laboratory, but should be left blank. If a computer CoC version is used, the sample identification can be input, but printed with this block black. A cross-referenced list of the CoC Sample Number and sample identification must be maintained separately.

Date Collected: Record the collection date in order to track the holding time of the sample. Note: For trip blanks, record the date it was placed in company with samples.

Time Collected: When collecting samples, record the time the sample is first collected. Use of the 24-hour military clock will avoid a.m. or p.m. designations (e.g., 1815 instead of 6:15 p.m.). Record local time; the laboratory is responsible for calculating holding times to local time.

Lab ID: This is for laboratory use only.

Box 7 **Matrix/QC:** Identify the matrix (e.g., water, soil, air, tissue, fresh water sediment, marine sediment, or product). If a sample is expected to contain high analyte concentrations (e.g., a tank bottom sludge or distinct product layer), notify the laboratory in the comment section. Mark an "X" for the sample(s) that have extra volume for laboratory QC matrix spike/matrix spike duplicate (MS/MSD) purposes. The sample provided for MS/MSD purposes is usually a field duplicate.

Box 8 **Analytical Parameters:** Enter the parameter by descriptor and the method number desired (e.g., BTEX 8260B, PAHs 8270C, etc.). Whenever practicable, list the parameters as they appear in the laboratory subcontract to maintain consistency and avoid confusion.

If the CoC does not have a specific box for number of sample containers, use the boxes below the analytical parameter, to indicate the number of containers collected for each parameter.

Box 9 **Sampler's Signature:** The person who collected samples must sign here.

Relinquished By: The person who turned over the custody of the samples to a second party other than an express mail carrier, such as FedEx or DHL, must sign and date here.

Received By: Typically, a representative of the receiving laboratory signs and dates here. Or, a field crew member who delivered the samples in person from the field to the laboratory might sign here. A courier, such as FedEx or DHL, does not sign here because they do not open the coolers. It must also be used by the prime contracting laboratory when samples are to be sent to a subcontractor.

Relinquished By: In the case of subcontracting, the primary laboratory will sign and date the Relinquished By space and fill out an additional CoC to accompany the samples being subcontracted.

Received By (Laboratory): This space is for the final destination (e.g., at a subcontracted laboratory). A representative of the final destination (e.g., subcontracted laboratory) must sign and date here.

Box 10 **Lab No. and Questions:** This box is to be filled in by the laboratory only.

Box 11 **Control Number:** This number is the "CoC" followed by the first contractor identification number in that cooler or contained on that CoC. This control number must be unique (i.e., never used twice). Record the date the CoC is completed. It should be the same date the samples are collected.

Box 12 **Total # of Containers:** Sum the number of containers in that row.

Box 13 **Totals:** Sum the number of containers in each column. Because CoC forms contain different formats depending on who produced the form, not all of the information listed in items 1 to 13 may be recorded; however, as much of this information as possible shall be included.

6.0 Quality Control and Assurance

6.1 Recordkeeping, sample labeling, and CoC activities must incorporate quality control measures to ensure accuracy and completeness.

6.2 Deviations from this procedure or the project-specific TO Quality Assurance Project Plan (QAPP) shall be documented in field records. Significant changes shall be approved by the **Program Quality Manager**.

7.0 Records, Data Analysis, Calculations

7.1 The CoC/analytical request form shall be faxed approximately daily to the **TO Laboratory Coordinator** for verification of accuracy. Following the completion of sampling activities, the sample logbook and CoC forms will be transmitted to the **TO Manager** for storage in project files. The **data validators** shall

receive a copy also. The original CoC/analytical request form shall be submitted by the **laboratory** along with the data delivered. Any changes to the analytical requests that are required shall be made in writing to the laboratory. A copy of this written change shall be sent to the data validators and placed in the project files. The reason for the change shall be included in the project files so that recurring problems can be easily identified.

7.2 Deviations from this procedure or the project-specific sampling and analysis plan shall be documented in the records. Significant changes shall be approved by the **Program Quality Manager**.

8.0 Attachments or References

8.1 Attachment 1 – Chain-of-Custody Seal

8.2 Attachment 2 – Generic Chain-of-Custody/Analytical Request Form

8.3 Attachment 3 – Sample Completed Chain-of-Custody

8.4 Attachment 4 – Sample Out-of-Control Form

8.5 Environmental Protection Agency, United States (EPA). 1988. *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA.* Interim Final. EPA/540/G-89/004. Office of Emergency and Remedial Response. October.

8.6 EPA. 1992. *RCRA Groundwater Monitoring Draft Technical Guidance*. EPA/530/R-93/001. Office of Solid Waste. November.

8.7 EPA. 1997. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846. 3rd ed., Final Update IIIA. Office of Solid Waste.

8.8 Water Resources Control Board, State of California. 1988. *Technical Guidance Manual for Solid Waste Water Quality Assessment Test (SWAT) Proposals and Reports*. August.

8.9 Procedure 3-02, *Logbooks.*

Author	Reviewer	Revisions (Technical or Editorial)
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue
Ken O'Donnell, PG Geologist	Claire Mitchell, PE, PMP Senior Engineer	Rev 1 – PFAS sampling update (July 2019)

Attachment 1
Chain of Custody Seal

CHAIN-OF-CUSTODY SEAL

[LABORATORY]	SAMPLE NO.	DATE	SEAL BROKEN BY
	SIGNATURE		DATE
	PRINT NAME AND TITLE *(Inspector, Analyst or Technician*		

Attachment 2
Generic Chain of Custody/Analytical Request Form

MW01376

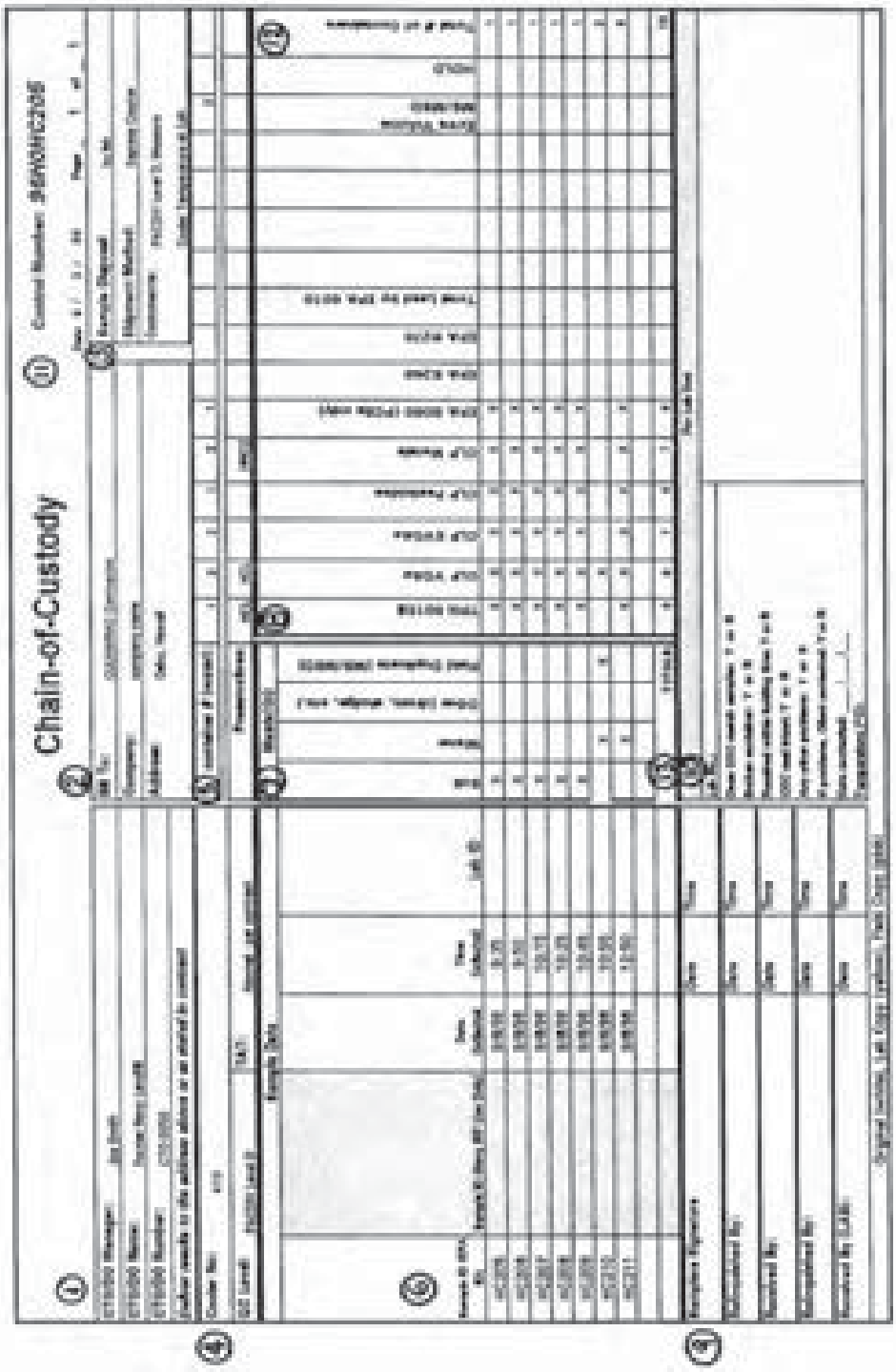
CHAIN OF CUSTODY RECORD Page ____ of ____

Client/Project Name:	Project Location:	Analysis Requested
Project Number:	Field Logbook No.:	
Sampler: (Print Name) / Affiliation:	Chain of Custody Tape No.:	
Signature:	Send Results/Report to:	

Field Sample No./ Identification	Date	Time	Grab	Comp	Sample Container (Size/Matl)	Sample Type (Liquid, Sludge, Etc.)	Preservative	Field Filtered								Lab I.D.	Remarks

Relinquished by: (Print Name)	Date:	Received by: (Print Name)	Date:	Analytical Laboratory (Destination):
Signature:	Time:	Signature:	Time:	
Relinquished by: (Print Name)	Date:	Received by: (Print Name)	Date:	
Signature:	Time:	Signature:	Time:	
Relinquished by: (Print Name)	Date:	Received by: (Print Name)	Date:	
Signature:	Time:	Signature:	Time:	Serial No.

Attachment 3
Sample Completed Chain of Custody



Attachment 4
Sample Out-of-Control Form

OUT OF CONTROL FORM	Status	Date	Initial
	Noted OOC		
	Submit for CA*		
	Resubmit for CA*		
	Completed		

Date Recognized:	By:
Dated Occurred:	Matrix
Parameter (Test Code):	Method:
Analyst:	Supervisor:

Samples Affected (List by Accession AND Sample No.)

1. Type of Event (Check all that apply)		2. Corrective Action (CA)* (Check all that apply)	
	Calibration Corr. Coefficient <0.995		Repeat calibration
	%RSD>20%		Made new standards
	Blank >MDL		Reran analysis
	Does not meet criteria:		Sample(s) redigested and rerun
	Spike		Sample(s) reextracted and rerun
	Duplicate		Recalculated
	LCS		Cleaned system
	Calibration Verification		Ran standard additions
	Standard Additions		Notified
	MS/MSD		Other (please explain)
	BS/BSD		
	Surrogate Recovery		
	Calculations Error		
	Holding Times Missed		
	Other (Please explain	Comments:	

3. Results of Corrective Action	
	Return to Control (indicated with)
	Corrective Actions Not Successful - DATA IS TO BE FLAGGED with _____.

Analyst:	Date:
Supervisor:	Date:
QA Department:	Date:

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Sample Handling, Storage, and Shipping

Procedure 3-04

1.0 Purpose and Scope

1.1 This standard operating procedure describes the actions to be used by personnel engaged in handling, storing, and transporting samples. The objective is to obtain samples of actual conditions with as little alteration as possible.

1.2 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

2.1 Avoid lifting heavy coolers with back muscles; instead, use leg muscles or dollies.

2.2 Wear powderless nitrile gloves, as defined in the project-specific health and safety plan, when handling sample containers to avoid contacting any materials that may have spilled out of the sample containers.

3.0 Terms and Definitions

None.

4.0 Training and Qualifications

4.1 The **Task Order (TO) Manager** and the **Laboratory Project Manager** are responsible for identifying instances of non-compliance with this procedure and ensuring that future sample transport activities comply with this procedure.

4.2 The **Field Manager** is responsible for ensuring that all samples are shipped according to this procedure.

4.3 **Field personnel** are responsible for the implementation of this procedure.

4.4 The **Program Quality Manager** is responsible for ensuring that sample handling, storage, and transport activities conducted during all TOs comply with this procedure.

4.5 All **field personnel** are responsible for the implementation of this procedure.

5.0 Procedure

5.1 Handling and Storage

Immediately following collection, label all samples according to Procedure 3-03, *Recordkeeping, Sample Labeling, and Chain of Custody*. The lids of the containers shall not be sealed with duct tape but may be covered with custody seals or placed directly into self-sealing polyethylene (e.g., Ziploc brand) bags. Place the sample containers in an insulated cooler with water ice in double, sealed self-sealing Ziploc bags. Samples should occupy the lower portion of the cooler, while the ice should occupy the upper portion. Place an absorbent material (e.g., proper absorbent cloth material) on the bottom of the cooler to contain liquids in case of spillage. Fill all empty space between sample containers with PFAS-free fill material. Prior to shipping, wrap glass sample containers on the sides, tops, and bottoms with polyethylene plastic wrap or other appropriate padding and/or surround them in Styrofoam to prevent breakage during transport. Pack all glass containers for water samples in an upright position, never

stacked or on their sides. Prior to shipment, replace the ice in the coolers so that samples will be maintained as close to 4 degrees Celsius (°C) as possible from the time of collection through transport to the analytical laboratory. Ship samples within 24 hours or on a schedule allowing the laboratory to meet holding times for analyses. The procedures for maintaining sample temperatures at 4°C pertain to all field samples.

5.2 Shipping

Follow all appropriate U.S. Department of Transportation regulations (e.g., 49 Code of Federal Regulations [CFR], Parts 171-179) for shipment of air, soil, water, and other samples. Elements of these procedures are summarized below.

5.2.1 Hazardous Materials Shipment

Field personnel must state whether any sample is suspected to be a hazardous material. A sample should be assumed hazardous unless enough evidence exists to indicate it is non-hazardous. If not suspected to be hazardous, shipments may be made as described in the Section 5.2.2 for non-hazardous materials. If hazardous, follow the procedures summarized below.

Any substance or material that is capable of posing an unreasonable risk to life, health, or property when transported is classified as hazardous. Perform hazardous materials identification by checking the list of dangerous goods for that particular mode of transportation. If not on that list, materials can be classified by checking the Hazardous Materials Table (49 CFR 172.102 including Appendix A) or by determining if the material meets the definition of any hazard class or division (49 CFR Part 173), as listed in Attachment 2.

All **persons shipping hazardous materials** must be properly trained in the appropriate regulations, as required by HM-126F, Training for Safe Transportation of Hazardous Materials (49 CFR HM-126F Subpart H). The training covers loading, unloading, handling, storing, and transporting of hazardous materials, as well as emergency preparedness in the case of accidents. **Carriers**, such as commercial couriers, must also be trained. Modes of shipment include air, highway, rail, and water.

When shipping hazardous materials, including bulk chemicals or samples suspected of being hazardous, the proper shipping papers (49 CFR 172 Subpart C), package marking (49 CFR 172 Subpart D), labeling (49 CFR 172 Subpart E), placarding (49 CFR 172 Subpart F, generally for carriers), and packaging must be used. Attachment 1 shows an example of proper package markings. Refer to a copy of 49 CFR each time hazardous materials/potentially hazardous samples are shipped.

According to Section 2.7 of the International Air Transport Association Dangerous Goods Regulations publication, very small quantities of certain dangerous goods may be transported without certain marking and documentation requirements as described in 49 CFR Part 172; however, other labeling and packing requirements must still be followed. Attachment 2 shows the volume or weight for different classes of substances. A “Dangerous Goods in Excepted Quantities” label must be completed and attached to the associated shipping cooler (Attachment 3). Certain dangerous goods are not allowed on certain airlines in any quantity.

As stated in item 4 of Attachment 4, the Hazardous Materials Regulations do not apply to hydrochloric acid (HCl), nitric acid (HNO_3), sulfuric acid (H_2SO_4), and sodium hydroxide (NaOH) added to water samples if their pH or percentage by weight criteria is met. These samples may be shipped as non-hazardous materials as discussed below.

5.2.2 Non-Hazardous Materials Shipment

If the samples are suspected to be non-hazardous based on previous site sample results, field screening results, or visual observations, if applicable, then samples may be shipped as non-hazardous.

When a cooler is ready for shipment to the laboratory, place two copies of the chain of custody (CoC) form inside a self-sealing polyethylene (e.g., Ziploc brand) bag and tape it to the inside of the insulated

cooler. Then, seal the cooler with waterproof tape and label it with “Fragile,” “This-End-Up” (or directional arrows pointing up), or other appropriate notices. Place custody seals on the coolers as discussed in Procedure 3-03, *Recordkeeping, Sample Labeling, and Chain of Custody*.

5.2.3 Shipments from Outside the Continental United States

Shipment of sample coolers to the United States from locations outside the continental United States is controlled by the U.S. Department of Agriculture (USDA) and is subject to their inspection and regulation. A “USDA Soil Import Permit” is required to prove that the receiving analytical laboratory is certified by the USDA to receive and properly dispose of soil. In addition, all sample coolers must be inspected by a **USDA representative**, affixed with a label indicating that the coolers contain environmental samples, and accompanied by shipping forms stamped by the **USDA inspector** prior to shipment.

In addition, the U.S. Customs Service must clear samples shipped from U.S. territorial possessions or foreign countries upon entry into the United States. As long as the commercial invoice is properly completed (see below), shipments typically pass through U.S. Customs Service without the need to open coolers for inspection.

Completion and use of proper paperwork will, in most cases, minimize or eliminate the need for the USDA and U.S. Customs Service to inspect the contents. Attachment 5 shows an example of how paperwork may be placed on the outside of coolers for non-hazardous materials. For hazardous materials, refer to Section 5.2.1.

In summary, tape the paperwork listed below to the outside of the coolers to accompany sample shipments. If a shipment is made up of multiple pieces (e.g., more than one cooler), the paperwork need only be attached to one cooler, provided that the **courier** agrees. All other coolers in the shipment need only to be taped and have the address and custody seals affixed.

1. **Courier Shipping Form & Commercial Invoice:** See Attachment 6 and Attachment 7 for examples of the information to be included on the commercial invoices for soil and water, respectively. Place the courier shipping form and commercial invoice inside a clear, plastic, adhesive-backed pouch that adheres to the package (typically supplied by the courier) and place it on the cooler lid as shown in Attachment 5.
2. **Soil Import Permit (soil only):** See Attachment 8 and Attachment 9 for examples of the soil import permit and soil samples restricted entry labels, respectively. The **laboratory** shall supply these documents prior to mobilization. The USDA often stops shipments of soil without these documents. Staple together the 2-inch × 2-inch USDA label (described below) and soil import permit and place them inside a clear plastic pouch. The **courier** typically supplies the clear, plastic, adhesive-backed pouches that adhere to the package.

   Placing one restricted entry label as shown in Attachment 5 (covered with clear packing tape) and one stapled to the actual permit is suggested.

   The USDA does not control water samples, so the requirements for soil listed above do not apply.
3. **Custody Seals:** The **laboratory** should supply the seals. **TO personnel** must sign and date these. At least two seals should be placed in such a manner that they stick to both the cooler lid and body. Placing the seals over the tape (as shown in Attachment 5), then covering it with clear packing tape is suggested. This prevents the seal from coming loose and enables detection of tampering.
4. **Address Label:** Affix a label stating the destination (laboratory address) to each cooler.
5. **Special Requirements for Hazardous Materials:** See Section 5.2.1.

Upon receipt of sample coolers at the laboratory, the **sample custodian** shall inspect the sample containers as discussed in Procedure 3-03, *Recordkeeping, Sample Labeling, and Chain of Custody*. The samples shall then be immediately extracted and/or analyzed, or stored in a refrigerated storage

area until they are removed for extraction and/or analysis. Whenever the samples are not being extracted or analyzed, they shall be returned to refrigerated storage.

6.0 Quality Control and Assurance

6.1 Sample handling, storage, and shipping must incorporate quality control measures to ensure conformance to these and the project requirements.

7.0 Records, Data Analysis, Calculations

7.1 Maintain records as required by implementing these procedures.

7.2 Deviations from this procedure or the project-specific sampling and analysis plan shall be documented in field records. Significant changes shall be approved by the **Program Quality Manager**.

8.0 Attachments or Reference

8.1 Attachment 1 – Example Hazardous Material Package Marking

8.2 Attachment 2 – Packing Groups

8.3 Attachment 3 – Label for Dangerous Goods in Excepted Quantities

8.4 Attachment 4 – SW-846 Preservative Exception

8.5 Attachment 5 – Non-Hazardous Material Cooler Marking Figure for Shipment from Outside the Continental United States

8.6 Attachment 6 – Commercial Invoice – Soil

8.7 Attachment 7 – Commercial Invoice – Water

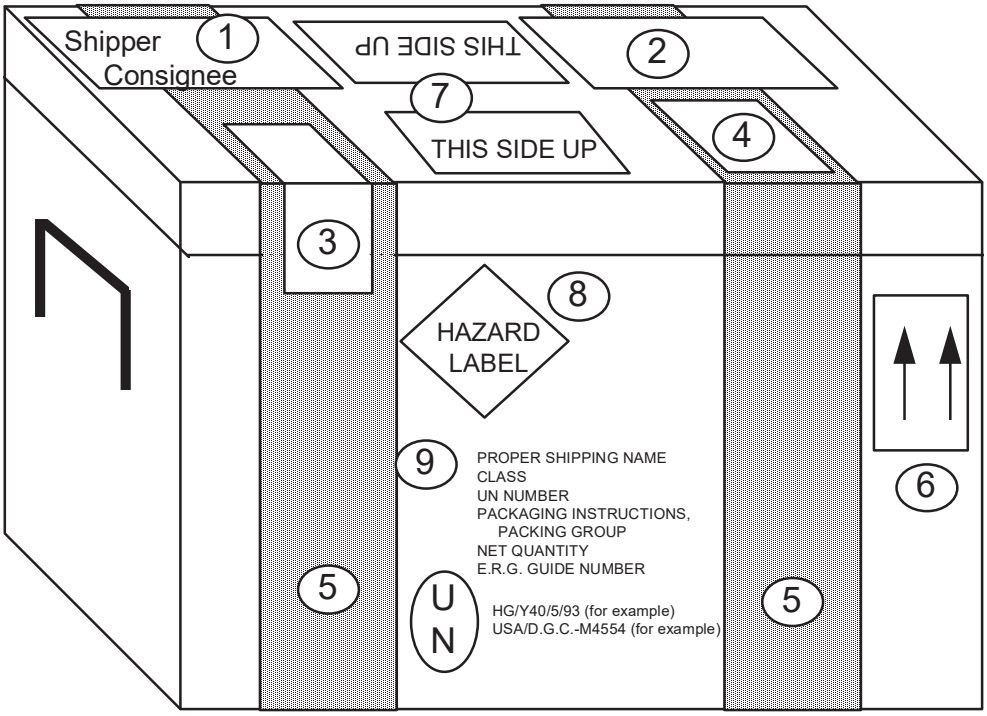
8.8 Attachment 8 – Soil Import Permit

8.9 Attachment 9 – Soil Samples Restricted Entry Labels

8.10 Procedure 3-03, *Recordkeeping, Sample Labeling, and Chain of Custody*.

Author	Reviewer	Revisions (Technical or Editorial)
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue
Ken O'Donnell, PG Geologist	Claire Mitchell, PE, PMP Senior Engineer	Rev 1 – PFAS sampling update (July 2019)

Attachment 1
Example Hazardous Material Package Marking



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

Attachment 2
Packing Groups

PACKING GROUP OF THE SUBSTANCE	PACKING GROUP 1		PACKING GROUP II		PACKING GROUP III	
CLASS or DIVISION of PRIMARY or SUBSIDIARY RISK	Packagings		Packagings		Packagings	
	Inner	Outer	Inner	Outer	Inner	Outer
1: Explosives	---------- Forbidden (Note A) ----------					
2.1: Flammable Gas	---------- Forbidden (Note B) ----------					
2.2: Non-Flammable, non-toxic gas	---------- See Notes A and B ----------					
2.3: Toxic gas	---------- Forbidden (Note A) ----------					
3. Flammable liquid	30 mL	300 mL	30 mL	500 mL	30 mL	1 L
4.1 Self-reactive substances	Forbidden		Forbidden		Forbidden	
4.1: Other flammable solids	Forbidden		30 g	500 g	30 g	1 kg
4.2: Pyrophoric substances	Forbidden		Not Applicable		Not Applicable	
4.2 Spontaneously combustible substances	Not Applicable		30 g	500 g	30 g	1 kg
4.3: Water reactive substances	Forbidden		30 g or 30 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
5.1: Oxidizers	Forbidden		30 g or 30 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
5.2: Organic peroxides (Note C)	See Note A		30 g or 30 mL	500 g or 250 mL	Not Applicable	
6.1: Poisons - Inhalation toxicity	Forbidden		1 g or 1 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
6.1: Poisons - oral toxicity	1 g or 1 mL	300 g or 300 mL	1 g or 1 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
6.1: Poisons - dermal toxicity	1 g or 1 mL	300 g or 300 mL	1 g or 1 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
6.2: Infectious substances	---------- Forbidden (Note A) ----------					
7: Radioactive material (Note D)	---------- Forbidden (Note A) ----------					
8: Corrosive materials	Forbidden		30 g or 30 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
9: Magnetized materials	---------- Forbidden (Note A) ----------					
9: Other miscellaneous materials (Note E)	Forbidden		30 g or 30 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L

Note A: *Packing groups are not used for this class or division.*
Note B: *For inner packagings, the quantity contained in receptacle with a water capacity of 30 mL. For outer packagings, the sum of the water capacities of all the inner packagings contained must not exceed 1 L.*
Note C: *Applies only to Organic Peroxides when contained in a chemical kit, first aid kit or polyester resin kit.*
Note D: *See 6.1.4.1, 6.1.4.2, and 6.2.1.1 through 6.2.1.7, radioactive material in excepted packages.*
Note E: *For substances in Class 9 for which no packing group is indicated in the List of Dangerous Goods, Packing Group II quantities must be used.*

Attachment 3
Dangerous Goods in Excepted Quantities

DANGEROUS GOODS IN EXCEPTED QUANTITIES

This package contains dangerous goods in excepted small quantities and is in all respects in compliance with the applicable international and national government regulations and the IATA Dangerous Goods Regulations.

_____
Signature of Shipper

_____ _____
Title Date

_____

_____
Name and address of Shipper

This package contains substance(s) in Class(es)
(check applicable box(es))

Class:	2	3	4	5	6	8	9
	☐	☐	☐	☐	☐	☐	☐

and the applicable UN Numbers are:

Attachment 4
SW-846 Preservative Exception

Measurement	Vol. Req. (mL)	Container[2]	Preservative[3,4]	Holding Time[5]
MBAS	250	P, G	Cool, 4ºC	48 Hours
NTA	50	P, G	Cool, 4ºC	24 Hours

1. More specific instructions for preservation and sampling are found with each procedure as detailed in this manual. A general discussion on sampling water and industrial wastewater may be found in ASTM, Part 31, p. 72-82 (1976) Method D-3370.

2. Plastic (P) or Glass (G). For metals, polyethylene with a polypropylene cap (no liner) is preferred.

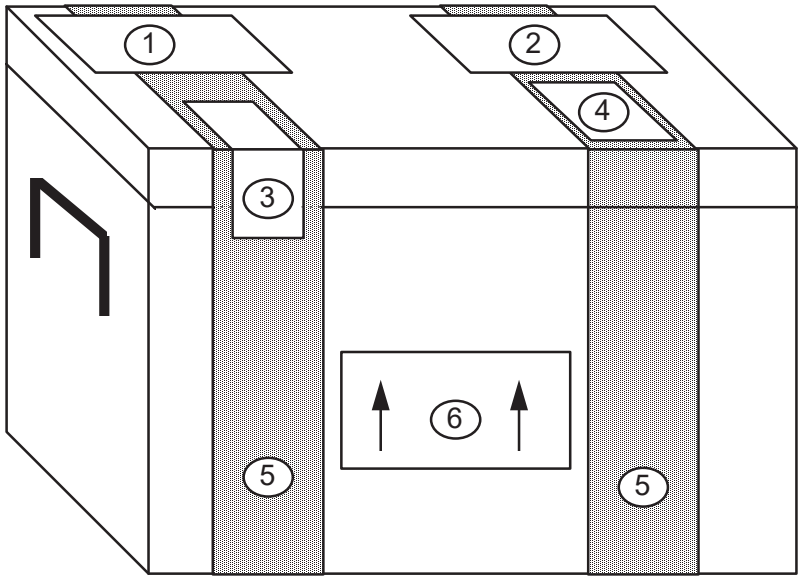
3. Sample preservation should be performed immediately upon sample collection. For composite samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then samples may be preserved by maintaining at 4ºC until compositing and sample splitting is completed.

4. When any sample is to be shipped by common carrier or sent through the United States Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. for the preservation requirements of Table 1, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentration of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

5. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of sample under study are stable for the longer time, and has received a variance from the Regional Administrator. Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show this is necessary to maintain sample stability.

6. Should only be used in the presence of residual chlorine.

Attachment 5
Non-Hazardous Material Cooler Marking Figure for Shipment from Outside the Continental United States



1. **AIR BILL/COMMERCIAL INVOICE**
2. **USDA PERMIT (Letter to Laboratory from USDA)**
3. **CUSTODY SEAL**
4. **USDA 2" X 2" SOIL IMPORT PERMIT**
5. **WATERPROOF STRAPPING TAPE**
6. **DIRECTION ARROWS STICKER - TWO REQUIRED**

Attachment 6
Commercial Invoice – Soil

DATE OF EXPORTATION 1/1/94	EXPORT REFERENCES (i.e., order no., invoice no., etc.) <TO #>
SHIPPER/EXPORTER (complete name and address) Joe Smith Ogden c/o <hotel name> <hotel address>	CONSIGNEE Sample Receipt <Lab Name> <Lab Address>
COUNTRY OF EXPORT Guam, USA	IMPORTER - IF OTHER THAN CONSIGNEE
COUNTRY OF ORIGIN OF GOODS Guam, USA	
COUNTRY OF ULTIMATE DESTINATION USA	
INTERNATIONAL AIR WAYBILL NO.	(NOTE: All shipments must be accompanied by a Federal Express International Air Waybill)

MARKS/NOS	NO. OF PKGS	TYPE OF PACKAGING	FULL DESCRIPTION OF GOODS	QTY	UNIT OF MEASURE	WEIGHT	UNIT VALUE	TOTAL VALUE
	3	coolers	Soil samples for laboratory analysis only				$1.00	$3.00
	TOTAL NO. OF PKGS.					TOTAL WEIGHT		TOTAL INVOICE VALUE
	3							$3.00
								Check one ☐ F.O.B. ☐ C&F ☐ C.I.F.

THESE COMMODITIES ARE LICENSED FOR THE ULTIMATE DESTINATION SHOWN.

DIVERSION CONTRARY TO UNITED STATES LAW IS PROHIBITED.

I DECLARE ALL THE INFORMATION CONTAINED IN THIS INVOICE TO BE TRUE AND CORRECT

SIGNATURE OF SHIPPER/EXPORTER (Type name and title and sign)

Joe Smith, Ogden	Joe Smith	1/1/94
Name/Title	Signature	Date

Attachment 7
Commercial Invoice – Water

DATE OF EXPORTATION 1/1/94	EXPORT REFERENCES (i.e., order no., invoice no., etc.) <TO #>
SHIPPER/EXPORTER (complete name and address) Joe Smith Ogden c/o <hotel name> <hotel address>	CONSIGNEE Sample Receipt <Lab Name> <Lab Address>
COUNTRY OF EXPORT Guam, USA	IMPORTER - IF OTHER THAN CONSIGNEE
COUNTRY OF ORIGIN OF GOODS Guam, USA	
COUNTRY OF ULTIMATE DESTINATION USA	

INTERNATIONAL AIR WAYBILL NO. _____ (NOTE: All shipments must be accompanied by a Federal Express International Air Waybill)

MARKS/NOS	NO. OF PKGS	TYPE OF PACKAGING	FULL DESCRIPTION OF GOODS	QTY	UNIT OF MEASURE	WEIGHT	UNIT VALUE	TOTAL VALUE
	3	coolers	Water samples for laboratory analysis only				$1.00	$3.00
	TOTAL NO. OF PKGS.					TOTAL WEIGHT		TOTAL INVOICE VALUE
	3							$3.00

Check one
☐ F.O.B.
☐ C&F
☐ C.I.F.

THESE COMMODITIES ARE LICENSED FOR THE ULTIMATE DESTINATION SHOWN.

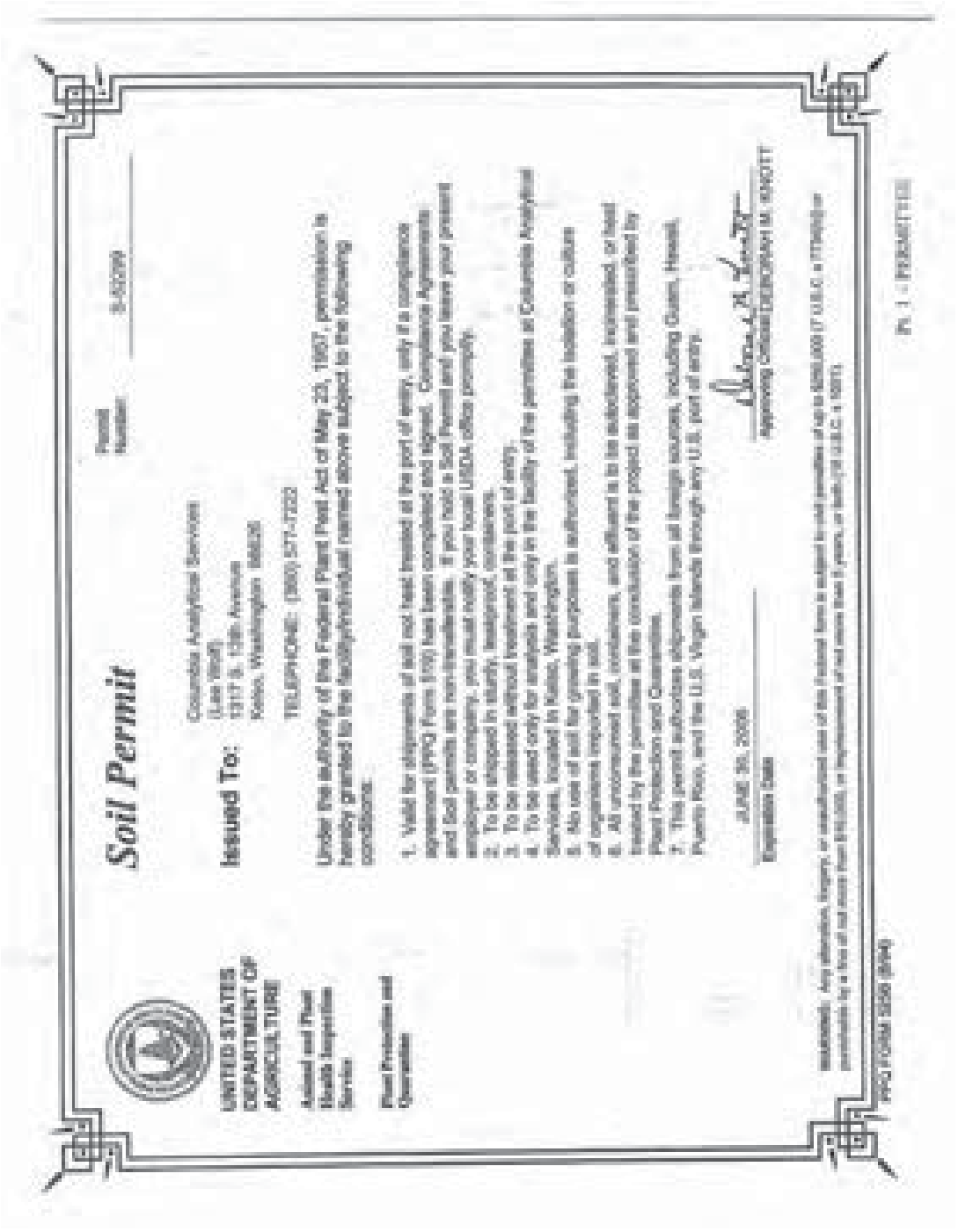
DIVERSION CONTRARY TO UNITED STATES LAW IS PROHIBITED.

I DECLARE ALL THE INFORMATION CONTAINED IN THIS INVOICE TO BE TRUE AND CORRECT

SIGNATURE OF SHIPPER/EXPORTER (Type name and title and sign)

Joe Smith, Ogden _____ Joe Smith _____ 1/1/94 _____

Attachment 8
Soil Import Permit



Soil Permit

Permit Number: S-50099

Issued To:
Columbia Analytical Services
(Lee Wolf)
5317 S. 12th Avenue
Kelso, Washington 98626
TELEPHONE: (360) 577-7222

Under the authority of the Federal Plant Pest Act of May 23, 1957, permission is hereby granted to the facility/individual named above subject to the following conditions:

1. Valid for shipments of soil not held treated at the port of entry, only if a compliance agreement (PPQ Form 519) has been completed and signed. Compliance Agreements and Soil permits are non-transferable. If you hold a Soil Permit and you leave your present employer or company, you must notify your local USDA office promptly.
2. To be shipped in sturdy, leakproof, containers.
3. To be released without treatment at the port of entry.
4. To be used only for analysis and only in the facility of the permittee at Columbia Analytical Services, located in Kelso, Washington.
5. No use of soil for growing purposes is authorized, including the isolation or culture of organisms imported in soil.
6. All unconsumed soil, containers, and effluent is to be autoclaved, incinerated, or held treated by the permittee at the conclusion of the project as approved and prescribed by Plant Protection and Quarantine.
7. This permit authorizes shipments from all foreign sources, including Guam, Hawaii, Puerto Rico, and the U.S. Virgin Islands through any U.S. port of entry.

JUNE 26, 2006
Expiration Date

Approving Official DEBORAH M. KNOTT

WARNING: Any alteration, forgery, or unauthorized use of this Federal form is subject to civil penalties of up to $250,000 (7 U.S.C. a 7734(b)) or imprisonment for a term of not more than five years, or both (18 U.S.C. § 1001).

Pt. 1 - PERMITTEE

APHIS FORM 550 (BW)

Attachment 9
Soil Samples Restricted Entry Labels

U.S. DEPARTMENT OF AGRICULTURE

ANIMAL AND PLANT HEALTH INSPECTION SERVICE

PLANT PROTECTION AND QUARANTINE

HYATTSVILLE, MARYLAND 20782

SOIL SAMPLES

RESTRICTED ENTRY

The material contained in this package is imported under authority of the Federal Plant Pest Act of May 23, 1957.

For release without treatment if addressee is currently listed as approved by Plant Protection and Quarantine.

PPQ FORM 550 *Edition of 12/77 may be used*

(JAN 83)

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Investigation Derived Waste Management

Procedure 3-05

1.0 Purpose and Scope

This standard operating procedure (SOP) describes activities and responsibilities of the client with regard to management of investigation-derived waste (IDW). The purpose of this procedure is to provide guidance for the minimization, handling, labelling, temporary storage, inventory, classification, and disposal of IDW generated under the client contract. This procedure will also apply to personal protective equipment (PPE), sampling equipment, decontamination fluids, non-IDW trash, non-indigenous IDW, and hazardous waste generated during implementation of removal or remedial actions. The information presented will be used to prepare and implement work plans (WPs) for IDW-related field activities. The results from implementation of WPs will then be used to develop and implement final IDW disposal plans.

If there are procedures whether it be from AECOM, state and/or federal that are not addressed in this SOP and are applicable to IDW then those procedures may be added as an appendix to the project specific SAP.

This procedure shall serve as management-approved professional guidance for the client and is consistent with protocol in the Uniform Federal Policy-Quality Assurance Project Plan (DoD 2005). As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved by both the Task Order (TO) Manager and the Quality Assurance (QA) Manager or Technical Director and documented.

This procedure was developed to serve as management-approved professional guidance for the management of IDW generated under the client contract. It focuses on the requirements for minimizing, segregating, handling, labeling, storing, and inventorying IDW in the field. Certain drum inventory requirements related to the screening, sampling, classification, and disposal of IDW are also noted in this procedure.

2.0 Safety

The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the project Health and Safety Plan (HASP). In the absence of a HASP, work will be conducted according to the TO WP and/or direction from the **Site Safety Officer (SSO)**.

All **Field Personnel** responsible for IDW management must adhere to the HASP and must wear the PPE specified in the site-specific HASP. Generally, this includes, at a minimum, steel-toed boots or steel-toed rubber boots, safety glasses, American National Standards Institute-standard hard hats, and hearing protection (if heavy equipment is in operation). If safe alternatives are not achievable, discontinue site activities immediately.

3.0 Terms and Definitions

None.

4.0 Training and Qualifications

4.1 The **TO Manager** is responsible for ensuring that IDW management activities comply with this procedure. The **TO Manager** is responsible for ensuring that all personnel involved in IDW management shall have the appropriate education, experience, and training to perform their assigned tasks.

4.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

4.3 The **Field Manager** is responsible for ensuring that all IDW is managed according to this procedure.

4.4 All **Field Personnel** are responsible for the implementation of this procedure.

All AECOM personnel who will perform any duties related to management of Resource Conservation and Recovery Act (RCRA) hazardous wastes or shipping of Department of Transportation (DOT) Hazardous Materials will be properly trained in accordance with 40 CFR § 262.34 and §265.16 for RCRA Waste Generators, as well as 49 CFR § 172.704 for DOT Hazardous Materials Shippers. All RCRA Hazardous Wastes are by definition DOT Hazardous Materials. See Section 6.1 for details on determining the IDW waste classification.

5.0 Equipment and Supplies

The equipment and supplies required for implementation of this SOP include the following:

- Containers for waste (e.g., [U.S. Department of Transportation] DOT approved 55-gallon open and closed top drums) and material to cover waste to protect from weather (e.g., plastic covering);
- Hazardous /non-hazardous waste drum labels (weatherproof);
- Permanent marking pens;
- Inventory forms for project file;
- Plastic garbage bags, zip lock storage bags, roll of plastic sheeting; and
- Steel-toed boots, chemical resistant gloves, coveralls, safety glasses, and any other PPE required in the HASP.

6.0 Procedure

The following procedures are used to handle the IDW.

6.1 Drum Handling

6.1.1 IDW shall be containerized using DOT approved drums. The drums shall be made of steel or polyethylene, be completely painted or opaque, and have removable lids (i.e., United Nations Code 1A2 or 1H2). Always consider IDW physical and chemical characterises to make sure the drum material is compatible. Typically, 55-gallon drums are used, however small drums may be used depending on the amount of waste generated. Large overpack drums may be used if smaller drums become damaged. New drums are preferred. The use of recycled drums should be avoided.

6.1.2 Recycled drums should not be used for hazardous waste, PCBs or other regulated shipments. For short-term storage of liquid IDW prior to discharge, double-walled bulk steel or plastic storage tanks may be used. For this scenario, consider the scheduling and cost-effectiveness of this type of bulk storage, treatment, and discharge system versus longer-term drum storage.

6.1.3 For long-term IDW storage at other project locations, the DOT approved drums with removable lids are recommended. Verify the integrity of the foam or rubber sealing ring located on the underside of some drum lids prior to sealing drums containing IDW liquids.

6.1.4 If the sealing ring is only partially attached to the drum lid, or if a portion of the sealing ring is missing, select another drum lid with a sealing ring that is in sound condition.

6.1.5 To prevent damage to drums, loss of drum integrity/containment, and/or presenting hazards to drum handlers, the following "Rules-of-Thumb" should be applied when filling drums.

- Liquid, soil, PPE/plastics, and construction debris must be segregated by media into individual drums.
- A **void space of 4 to 6 inches** from the top of the drum (the upper drum ring on most drums) will be left in the drum to allow room for ice expansion when filling drums with water or oil/water emulsions. Under freezing temperatures, expanding ice in a full drum can deform the bottom of a drum such that it is no longer DOT compliant, cause ruptures and/or dislodge the drum lid and present a containment breach. The consequences of this damage can be both economic and environmental.
- Compatibility between the chemical component(s) of the IDW and the drum material must be considered before choosing the type of drum/container to use. Steel drums are susceptible to corrosion and loss of integrity when in contact with high pH water. Lime-based products (cement, concrete, grout, etc.) should not be disposed in steel drums containing water or soil water mixtures, and liquid IDW should not be disposed in steel drums used to mix lime-based products (separate reusable containers for mixing should be used when possible). If high (>12) or low (<2) pH conditions are possible, IDW liquids should be monitored for pH using a calibrated pH meter or pH test strips. The use of plastic drum liners or polyethylene drums is also recommended for high or low pH liquid IDW.
- Soil drums will be filled to no more than two thirds of the drum capacity. Drums completely full of soil can weigh over 600 pounds. Although drum handling tools and carts provide some assistance, moving such excessive weights present significant hazards, including; muscle strain, crushing (foot and fingers), and loss of drum control, such as sliding off of lift gates.
- Drums should not be overfilled filled with PPE and plastic (tubing, old macrocores) such that the material is excessively compacted. Pinch points are presented as the drum is closed under force, and the compressed material can spring up when the drums are opened.

6.1.6 Stacking full or partially full drums is prohibited.

6.1.7 To prepare IDW drums for labelling, wipe clean the outer wall surfaces and drum lids of all material that might prevent legible and permanent labelling. If potentially contaminated material adheres to the outer surface of a drum, wipe that material from the drum, and segregate the paper towel or rag used to remove the material with visibly soiled PPE and disposable sampling equipment. Label all IDW drums and place them on pallets prior to storage.

6.2 Labelling

6.2.1 Containers used to store IDW must be properly labelled. Two general conditions exist: 1) from previous studies or on-site data, waste characteristics are known to be either hazardous or nonhazardous; or 2) waste characteristics are unknown until additional data are obtained.

6.2.2 For situations where the waste characteristics are known, the waste containers should be packaged and labelled in accordance with state regulations and any federal regulations that may govern the labelling of waste.

6.2.3 The following information shall be placed on all non-hazardous waste labels:

- Description of waste (i.e., purge water, soil cuttings);
- Contact information (i.e., contact name and telephone number);
- Date when the waste was first accumulated.

6.2.4 The following information shall be placed on all hazardous waste labels:

- Description of waste (i.e., purge water, soil cuttings);
- Generator information (i.e., name, address, contact telephone number);
- EPA identification number (supplied by on-site client representative);
- Date when the waste was first accumulated.

6.2.5 When the final characterization of a waste is unknown, a notification label should be placed on the drum with the words "waste characterization pending analysis" and the following information included on the label:

- Description of waste (i.e., purge water, soil cuttings);
- Contact information (i.e., contact name and telephone number);
- Date when the waste was first accumulated.

6.2.6 Once the waste has been characterized, the label should be changed as appropriate for a nonhazardous or hazardous waste.

6.2.7 Waste labels should be constructed of a weatherproof material and filled out with a permanent marker to prevent being washed off or becoming faded by sunlight (faded entries should be remarked during inspections performed as specified in Section 6.2.4). It is recommended that waste labels be placed on the side of the container, since the top is more subject to weathering. However, when multiple containers are accumulated together, it may also be helpful to include labels on the top of the containers to facilitate organization and disposal. In addition to a label, each drum should be numbered on the side and top with a paint pen or wax pencil for easy identification.

6.2.8 Each container of waste generated shall be recorded in the field notebook used by the person responsible for labelling the waste. After the waste is disposed of, either by transportation off-site or disposal on-site in an approved disposal area, an appropriate record shall be made in the same field notebook to document proper disposition of IDW.

6.3 Types of Site Investigation Waste

Several types of waste are generated during site investigations that may require special handling. These include solid, liquid, and used PPE, as discussed further below.

Solid Waste

Soil cuttings from boreholes will typically be placed in containers unless site specific requirements allow for soil cuttings to be placed back into the borehole after drilling is complete. Drilling mud generated during investigation activities shall be collected in containers. Covers should be included on the containers and must be secured at all times and only open during filling activities. The containers shall be labelled in accordance with this SOP. An inventory containing the source, volume, and description of material put in the containers shall be logged on prescribed forms and kept in the project file.

Non-hazardous solid waste can be disposed on-site in the designated site landfill or in a designated evaporation pond if it is liquefied. Hazardous wastes must be disposed off-site at an approved hazardous waste landfill.

Liquid Waste

Groundwater generated during monitoring well development, purging, and sampling can be collected in truck-mounted containers and/or other transportable containers (i.e., 55-gallon drums). Lids or bungs on drums must be secured at all times and only open during filling or pumping activities. The containers shall be labelled in accordance with this SOP. Non-hazardous liquid waste can be disposed of in one of the designated lined evaporation ponds on-site. Hazardous wastes must be handled separately and disposed off-site at an approved hazardous waste facility.

Personal Protective Equipment

PPE that is generated throughout investigation activities shall be placed in plastic garbage bags. If the solid or liquid waste that was being handled is characterized as hazardous waste, then the corresponding PPE should also be disposed as hazardous waste. If not, all PPE should be disposed as non-hazardous waste in the designated on-site landfill. Trash that is generated as part of field activities may be disposed of in the landfill as long as the trash was not exposed to hazardous media.

6.1 IDW Waste Classification

State and federal regulations require specific handling and storage requirements for wastes classified as hazardous, such as secondary containment and waste removal deadlines (see Section 6.2.2). The Site owner/operator must determine whether the IDW may contain a listed hazardous waste based on the source of contamination, contaminants, and waste manifests or any other documentation of wastes generated at the Site. It is presumed that the IDW will be considered a solid waste (40 CFR 261.2) but this should be verified during the work plan development. If the available documentation indicates that a listed hazardous waste was generated at the Site, then the IDW will be considered a hazardous waste regulated under RCRA.

If there is inconclusive documentation concerning the IDW generated at the Site, then the U.S. EPA has stated the IDW is not a listed hazardous waste. However, in this case, further evaluation is necessary to evaluate whether the IDW in question exhibits a characteristic of hazardous waste. This is determined by analytical testing or knowledge. An IDW that may be characteristically hazardous should be evaluated for the following hazardous characteristics:

- Characteristic of ignitability (40 CFR §261.21)
- Characteristic of corrosivity (40 CFR §261.22)
- Characteristic of reactivity (40 CFR §261.23)
- Characteristic of toxicity (40 CFR §261.24)

If the RDW contains a listed hazardous waste, then U.S. EPA's contained-in policy (53 FR 31138, 31142, 31148, 57 FR 21453, 61 FR 18795) for contaminated environmental media should be evaluated. U.S. EPA considers IDW to contain hazardous waste:

- when it exhibits a characteristic of hazardous waste; or
- when it is impacted with concentrations of hazardous constituents from listed hazardous wastes that are above health-based levels.

Generally, IDW that does not (or no longer) contain hazardous waste are not subject to RCRA, but in some circumstances, the IDW that contained hazardous waste when first generated remain subject to land disposal restrictions (LDR) (40 CFR §268.45). There are also special LDR standards specific to contaminated debris (40 CFR §268.45).

6.2 Waste Accumulation On-Site

6.2.1 Solid, liquid, or PPE waste generated during investigation activities that are classified as nonhazardous or "characterization pending analysis" should be disposed of as soon as possible. Until off-site transport and disposal is arranged, drums should be moved to a staging location accessible by pickup by truck. This location should be relatively flat, have a hard surface (densely compact dirt, concrete, or asphalt), and be secure (by a fence or building).

6.2.2 Solid, liquid, or PPE waste generated during investigation activities that are classified as hazardous **shall not** be accumulated on-site longer than **90 days**. All hazardous waste containers shall be stored in a secured storage area. The following requirements for the hazardous waste storage area must be implemented:

- Proper hazardous waste signs shall be posted as required by any state or federal statutes that may govern the labelling of waste;
- Secondary containment to contain spills;
- Spill containment equipment must be available;
- Fire extinguisher;
- Adequate aisle space for unobstructed movement of personnel.

6.2.3 When possible, drums should be segregated in the storage area by media and or classification (liquid, solid, non-hazardous, hazardous, etc.) to facilitate type identification during characterization sampling and pickup and reduce the need to rearrange drums if multiple pickups by type are required.

6.2.4 Throughout the project, an inventory shall be maintained to itemize the type and quantity of the waste generated. During active site work, weekly storage area inspections should be performed and documented to ensure compliance with the requirements specified above. Monthly storage area inspections should be performed following the completion of active site work and the date the IDW is removed from the storage area by the waste hauler. Containers should be inventoried and inspected regularly. Labels should be checked to make sure they remain legible. Inspection notes should include the condition of the staging area as this will be important when coordinating the labour and equipment the waste hauler will require. Anomalies should be documented and photographed.

6.3 Waste Disposal

6.3.1 Solid, liquid, and PPE waste will be characterized for disposal through the use of client knowledge, laboratory analytical data created from soil or groundwater samples gathered during the field activities, and/or composite samples from individual containers. The selected disposal facility will prepare a waste profile based on the characterization results. The waste generator (Navy representative or authorized agent) will review and sign the profile.

6.3.2 All waste generated during field activities will be stored, transported, and disposed of according to applicable state, federal, and local regulations. All wastes classified as hazardous will be disposed of at a licensed treatment storage and disposal facility or managed in other approved manners.

6.3.3 Disposal facilities for waste generated during activities under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) will require EPA approval under the Off-Site Rule (OSR) procedures (40 CFR 300.440) to ensure the facility is operating in compliance with RCRA or other Federal and State requirements. After the

waste profile is finalized, the generator will submit it with an OSR request form to the EPA project manager for approval. An example OSR request form is provided in Attachment A. IDW may not be shipped to the facility until approval is granted by the EPA. OSR approvals per waste profile are valid for 90 days.

6.3.4 In general, waste disposal should be carefully coordinated with the facility receiving the waste. Facilities receiving waste have specific requirements that vary even for non-hazardous waste, so characterization should be conducted to support both applicable regulations and facility requirements.

6.4 Regulatory Requirements

The following federal and state regulations shall be used as resources for determining waste characteristics and requirements for waste storage, transportation, and disposal:

- Code of Federal Regulations (CFR), Title 40, Part 261;
- CFR, Title 49, Parts 172, 173, 178, and 179.

6.5 Waste Transport

A state-certified hazardous waste hauler shall transport all wastes classified as hazardous. Typically, the facility receiving any waste can coordinate a hauler to transport the waste. Shipped hazardous waste shall be disposed of in accordance with all RCRA/USEPA requirements. All waste manifests or bills of lading will be signed either by the client or the client's designee.

7.0 Quality Control and Assurance

7.1 Management of IDW must incorporate quality control measures to ensure conformance to these and the project requirements.

8.0 Records, Data Analysis, Calculations

8.1 Maintain records as required by implanting the procedures in this SOP.

8.2 Deviations from this procedure or the sampling and analysis plan shall be documented in field records. Significant changes shall be approved by the **Program Quality Manager**.

9.0 Attachments or References

Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual.* Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

Department of Energy, United States (DOE). 1994. *The Off-Site Rule*. EH-231-020/0194. Office of Environmental Guidance. March.

1999. *Management of Remediation Waste under the Resource Conservation and Recovery Act (RCRA)*. Office of Environmental Policy and Assistance. 20 December.

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Author	Reviewer	Revisions (Technical or Editorial)
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (May 2012)
Joshua Millard Senior Geologist	Andrew Borden Geologist	Rev 1 – Technical (Jan 2017)
Ken O'Donnell, PG Geologist	Claire Mitchell, PE, PMP Senior Engineer	Rev 2 – PFAS sampling update (July 2019)

ATTACHMENT A – OFF SITE RULE REQUEST FORM

United States Environmental Protection Agency – Region 1

Off-Site Rule Compliance Request Form

Date: (mm/dd/yy)		Supporting Documentation Required-Attached? (yes/no)	

RECEIVING FACILITY INFORMATION:		
1	**Name of Facility receiving CERCLA waste:**	
2	**Address of Facility:**	
3	**City:**	
4	**State:**	
5	**Zip Code:**	
6	**EPA/State Facility ID:**(e.g. Haz. Waste/Municipal Waste ID)	
7	**Other Pertinent ID Numbers:** (e.g. License #, permit #)	
8	**Phone Number** (if available):	
9	**Contact Name** (if available):	
10	**FAX Number** (if available):	
11	**E-mail address** (if available):	

GENERATING FACILITY INFORMATION:		
12	**CERCLA Site Name:**	
13	**CERCLA Site Address:**	
14	**City:**	
15	**State:**	
16	**Zip Code:**	
17	**CERCLA Site ID:** (i.e. alpha-numeric)	
18	**EPA CERCLA ID #:**	
19	**Waste Media:** (e.g., Soil, Water, Air, etc.)	
20	**CERCLA Hazardous Waste Contaminates:** (e.g. tce, lead)	
21	**Amount of CERCLA Waste:** (e.g. gallons, pounds, tons, ft³, yd³)	
22	**EPA representative making waste determination:** (e.g. OSC, RPM & TeL#)	
23	**Basis of Waste Determination:** (e.g. analyses, TCLP, etc.)	

[Form: Off-Site Compliance Request] [Rev. G – August 25, 2016] [MacLeod.Donald@epa.gov]

For more information on the Off-Site Rule, please contact the appropriate Regional Off-Site Contact (ROC) listed at **http://www.epa.gov/waste/hazard/wastetypes/wasteid/offsite/index.htm**

	Regional Off-Site Contacts (listed as of April 8, 2014)	
Region # U.S. & DC,PR,VI	**Contact Name**	**Telephone #**
1 CT,MA,ME,NH,RI,VT	Donald MacLeod (macleod.donald@epa.gov)	617.918.1405
2 NY,NJ,PR,VI	Beckett Greenin (Region2_OSR@epa.gov)	732.321.4341
3 DC,DE,MD,PA,VA,WV	Stacie Pratt (pratt.stacie@epa.gov)	215.814.5173
4 AL,FL,GA,KY,MS,NC,SC,TN	Paula Whiting (whiting.paula@epa.gov)	404.562.9277
5 IL,IN,MI,MN,OH,WI	William Damico (damico.william@epa.gov)	312.353.8207
6 AR,LA,NM,OK,TX	Wilkin (Ann) Shannon (shannon.wilkin@epa.gov)	214.665.2282
7 IA,KS,MO,NE	Nicole Moran (moran.nicole@epa.gov)	913.551.7641
8 CO,MT,ND,SD,UT,WY	Linda Jacobson (jacobson.linda@epa.gov)	303.312.6503
9 AZ,CA,HI,NV	Kandice Bellamy (bellamy.kandice@epa.gov)	415.972.3304
10 AK,ID,OR,WA	Kevin Schanilec (schanilec.kevin@epa.gov) Ofelia Erickson (erickson.ofelia@epa.gov)	206.553.1061 206.553.2583

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Equipment Decontamination

Procedure 3-06

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes methods of equipment decontamination, to be used for activities where samples for chemical analysis are collected or where equipment will need to be cleaned before leaving the site or before use in subsequent activities.

1.2 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

It is the responsibility of the **Site Safety and Health Officer (SSHO)** to set up the site zones (i.e., exclusion, transition, and clean) and decontamination areas. Generally, the decontamination area is located within the transition zone, upwind of intrusive activities, and serves as the washing area for both personnel and equipment to minimize the spread of contamination into the clean zone. Typically, for equipment, a series of buckets are set up on a visqueen-lined bermed area. Separate spray bottles containing cleaning solvents as described in this procedure or the Task Order (TO) Quality Assurance Project Plan (QAPP) and deionized water are used for final rinsing of equipment. Depending on the nature of the hazards and the site location, decontamination of heavy equipment, such as augers, pump drop pipe, and vehicles, may be accomplished using a variety of techniques.

All **Field Personnel** responsible for equipment decontamination must adhere to the site-specific Accident Prevention Plan (APP)/Site Safety and Health Plan (SSHP) and must wear the personal protective equipment (PPE) specified in the site-specific APP/SSHP. Generally, this includes, at a minimum, Tyvek® coveralls, steel-toed boots with boot covers or steel-toed rubber boots, safety glasses, American National Standards Institute-standard hard hats, and hearing protection (if heavy equipment is in operation). Air monitoring by the **SSHO** may result in an upgrade to the use of respirators and cartridges in the decontamination area; therefore, this equipment must be available on site. If safe alternatives are not achievable, discontinue site activities immediately.

In addition to the aforementioned precautions, the following sections describe safe work practices that will be employed.

2.1 Chemical Hazards associated with Equipment Decontamination

- Avoid skin contact with and/or incidental ingestion of decontamination solutions and water.
- Utilize PPE as specified in the site-specific APP/SSHP to maximize splash protection.
- Refer to material safety data sheets, safety personnel, and/or consult sampling personnel regarding appropriate safety measures (i.e., handling, PPE including skin and respiratory).
- Take the necessary precautions when handling detergents and reagents.

2.2 Physical Hazards associated with Equipment Decontamination

- To avoid possible back strain, it is recommended to raise the decontamination area 1 to 2 feet above ground level.

- To avoid heat stress, over exertion, and exhaustion, it is recommended to rotate equipment decontamination among all site personnel.
- Take necessary precautions when handling field sampling equipment.

3.0 Terms and Definitions

None.

4.0 Training and Qualifications

4.1 The **TO Manager** is responsible for ensuring that decontamination activities comply with this procedure. The **TO Manager** is responsible for ensuring that all personnel involved in equipment decontamination shall have the appropriate education, experience, and training to perform their assigned tasks.

4.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

4.3 The **Field Manager** is responsible for ensuring that all field equipment is decontaminated according to this procedure.

4.4 All **Field Personnel** are responsible for the implementation of this procedure.

5.0 Procedure

Decontamination of equipment used in soil/sediment sampling, groundwater monitoring, well drilling and well development, as well as equipment used to sample groundwater, surface water, sediment, waste, wipe, asbestos, and unsaturated zone, is necessary to prevent cross-contamination and to maintain the highest integrity possible in collected samples. Planning a decontamination program requires consideration of the following factors:

- Location where the decontamination procedures will be conducted
- Types of equipment requiring decontamination
- Frequency of equipment decontamination
- Cleaning technique and types of cleaning solutions appropriate to the contaminants of concern
- Method for containing the residual contaminants and wash water from the decontamination process
- Use of a quality control measure to determine the effectiveness of the decontamination procedure

The following subsections describe standards for decontamination, including the frequency of decontamination, cleaning solutions and techniques, containment of residual contaminants and cleaning solutions, and effectiveness.

5.1 Decontamination Area

Select an appropriate location for the decontamination area at a site based on the ability to control access to the area, the ability to control residual material removed from equipment, the need to store clean equipment, and the ability to restrict access to the area being investigated. Locate the decontamination area an adequate distance away and upwind from potential contaminant sources to avoid contamination of clean equipment.

5.2 Types of Equipment

Drilling equipment that must be decontaminated includes drill bits, auger sections, drill-string tools, drill rods, split barrel samplers, tremie pipes, clamps, hand tools, and steel cable. Decontamination of monitoring well development and groundwater sampling equipment includes submersible pumps, bailers,

interface probes, water level meters, bladder pumps, airlift pumps, peristaltic pumps, and lysimeters. Other sampling equipment that requires decontamination includes, but is not limited to, hand trowels, hand augers, slide hammer samplers, shovels, stainless-steel spoons and bowls, soil sample liners and caps, wipe sampling templates, composite liquid waste samplers, and dippers. Equipment with a porous surface, such as rope, cloth hoses, and wooden blocks, cannot be thoroughly decontaminated and shall be properly disposed of after one use.

5.3 Frequency of Equipment Decontamination

Decontaminate down-hole drilling equipment and equipment used in monitoring well development and purging prior to initial use and between each borehole or well. Down-hole drilling equipment, however, may require more frequent cleaning to prevent cross-contamination between vertical zones within a single borehole. When drilling through a shallow contaminated zone and installing a surface casing to seal off the contaminated zone, decontaminate the drilling tools prior to drilling deeper. Initiate groundwater sampling by sampling groundwater from the monitoring well where the least contamination is suspected. Decontaminate groundwater, surface water, and soil sampling devices prior to initial use and between collection of each sample to prevent the possible introduction of contaminants into successive samples.

5.4 Cleaning Solutions and Techniques

Decontamination can be accomplished using a variety of techniques and fluids. The preferred method of decontaminating major equipment, such as drill bits, augers, drill string, and pump drop-pipe, is steam cleaning. To steam clean, use a portable, high-pressure steam cleaner equipped with a pressure hose and fittings. For this method, thoroughly steam wash equipment and rinse it with potable tap water to remove particulates and contaminants.

A rinse decontamination procedure is acceptable for equipment such as bailers, water level meters, new and re-used soil sample liners, and hand tools. The decontamination procedure shall consist of the following: (1) wash with a PFAS-free detergent (Alconox®, Liquinox®, or other suitable detergent) and deionized water solution, and (2) rinse in triplicate with deionized water. If possible, disassemble equipment prior to cleaning. Add an additional wash as needed at the beginning of the process if equipment is very soiled.

Decontaminating submersible pumps requires additional effort because internal surfaces become contaminated during usage. Decontaminate these pumps by washing and rinsing the outside surfaces using the procedure described for small equipment or by steam cleaning. Decontaminate the internal surfaces by recirculating fluids through the pump while it is operating. This recirculation may be done using a relatively long (typically 4 feet) large-diameter pipe (4-inch or greater) equipped with a bottom cap. Fill the pipe with the decontamination fluids, place the pump within the capped pipe, and operate the pump while recirculating the fluids back into the pipe. The decontamination sequence shall include: (1) detergent and deionized water solution, and (2) rinse in triplicate with deionized water rinse. Change the decontamination fluids after each decontamination cycle.

Solvents other than isopropyl alcohol may be used, depending upon the contaminants involved. For example, if polychlorinated biphenyls or chlorinated pesticides are contaminants of concern, hexane may be used as the decontamination solvent; however, if samples are also to be analyzed for volatile organics, hexane shall not be used. In addition, some decontamination solvents have health effects that must be considered. Decontamination water shall consist of deionized water. Decontamination solvents to be used during field activities will be specified in the TO QAPP.

Rinse equipment used for measuring field parameters, such as pH (indicates the hydrogen ion concentration – acidity or basicity), temperature, specific conductivity, and turbidity with deionized water after each measurement. Also wash new, unused soil sample liners and caps with a fresh detergent solution and rinse them with deionized water to remove any dirt or cutting oils that might be on them prior to use.

5.5 Containment of Residual Contaminants and Cleaning Solutions

A decontamination program for equipment exposed to potentially hazardous materials requires a provision for catchment and disposal of the contaminated material, cleaning solution, and wash water.

When contaminated material and cleaning fluids must be contained from heavy equipment, such as drill rigs and support vehicles, the area must be properly floored, preferably with a concrete pad that slopes toward a sump pit. If a concrete pad is impractical, planking can be used to construct solid flooring that is then covered by a nonporous surface and sloped toward a collection sump. If the decontamination area lacks a collection sump, use plastic sheeting and blocks or other objects to create a bermed area for collection of equipment decontamination water. Situate items, such as auger flights, which can be placed on metal stands or other similar equipment, on this equipment during decontamination to prevent contact with fluids generated by previous equipment decontamination. Store clean equipment in a separate location to prevent recontamination. Collect decontamination fluids contained within the bermed area and store them in secured containers as described below.

Use wash buckets or tubs to catch fluids from the decontamination of lighter-weight drilling equipment and hand-held sampling devices. Collect the decontamination fluids and store them on site in secured containers, such as U.S. Department of Transportation-approved drums, until their disposition is determined by laboratory analytical results. Label containers in accordance with Procedure 3-05, *IDW Management*.

6.0 Quality Control and Assurance

A decontamination program must incorporate quality control measures to determine the effectiveness of cleaning methods. Quality control measures typically include collection of equipment blank samples or wipe testing. Equipment blanks consist of analyte-free deionized water that has been poured over or through the sample collection equipment after its final decontamination rinse. Wipe testing is performed by wiping a PFAS-free cotton cloth over the surface of the equipment after cleaning. These quality control measures provide "after-the fact" information that may be useful in determining whether or not cleaning methods were effective in removing the contaminants of concern.

7.0 Records, Data Analysis, Calculations

Any project where sampling and analysis is performed shall be executed in accordance with an approved sampling and analysis plan. This procedure may be incorporated by reference or may be incorporated with modifications described in the plan.

Deviations from this procedure or the sampling and analysis plan shall be documented in field records. Significant changes shall be approved by the **Program Quality Manager**.

8.0 Attachments or References

8.1 ASTM Standard D5088. 2008. *Standard Practice for Decontamination of Field Equipment Used at Waste Sites.* ASTM International, West Conshohocken, PA. 2008. DOI: 10.1520/D5088-02R08. www.astm.org.

8.2 Procedure 3-05, *IDW Management*.

Author	Reviewer	Revisions (Technical or Editorial)
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue
Ken O'Donnell, PG Geologist	Claire Mitchell, PE, PMP Senior Engineer	Rev 1 – PFAS sampling update (July 2019)

Land Surveying

Procedure 3-07

1.0 Purpose and Scope

1.1 The purpose of this document is to define the standard operating procedure (SOP) for acquiring land surveying data to facilitate the location and mapping of geologic, hydrologic, geotechnical data, and analytical sampling points and to establish topographic control over project sites.

1.2 This procedure is the Program-approved professional guidance for work performed by AECOM under the client contract.

1.3 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review. If there are procedures whether it be from Resolution Consultants, state and/or federal that are not addressed in this SOP and are applicable to surface water sampling then those procedures may be added as an appendix to the project-specific Quality Assurance Project Plan (QAPP).

1.4 It is fully expected that the procedures outlined in this SOP will be followed. Procedural modifications may be warranted depending upon field conditions, equipment limitations, or limitations imposed by the procedure. Substantive modification to this SOP will be approved in advance by the Program Quality Manager. Deviations to this SOP will be documented in the field records.

1.5 If there are procedures, whether it be from Resolution Consultants, state and/or federal, that are not addressed in this SOP and are applicable to land surveying then those procedures may be added as an appendix to the project-specific QAPP.

2.0 Safety

2.1 Depending upon the site-specific contaminants, various protective programs must be implemented prior to conducting fieldwork. All **field sampling personnel** must review the project-specific Accident Prevention Plan (APP)/Site Safety and Health Plan (SSHP) paying particular attention to the control measures planned for the specific field tasks. Conduct preliminary area monitoring to determine the potential hazard to field sampling personnel. If significant contamination is observed, minimize contact with potential contaminants in both the vaper and liquid phase through the use of respirators and disposable clothing.

2.2 In addition, observe standard health and safety practices according to the project-specific APP/SSHP. Suggested minimum protection includes inner disposable vinyl gloves, outer chemical-protective nitrile gloves, rubberized steel-toed boots, and an American National Standards Institute-standard hard hat. Half-face respirators and cartridges and Tyvek® suits may be necessary depending on the contaminant concentrations, and shall always be available on site.

2.3 Daily safety briefs will be conducted at the start of each working day before any work commences. These daily briefs will be facilitated by the **Site Safety and Health Officer (SSHO)** or designee to discuss the day's events and any potential health risk areas covering every aspect of the work to be completed. Weather conditions are often part of these discussions. As detailed in the APP/SSHP, everyone on the field team has the authority to stop work if an unsafe condition is perceived until the conditions are fully remedied to the satisfaction of the SSHO.

2.4 The health and safety considerations for the work associated with land surveying include:

- Slip, trips and falls associated with work in the field;

- Biological hazards associated with work in the field; and,
- Potential hazards associated with chemicals of concern (COCs) that may be located in the survey area,

3.0 Terms and Definitions

3.1 Boundary Survey

Boundary surveys are conducted by Certified Land Surveyors in order to delineate a legal property line for a site or section of a site.

3.2 Global Positioning System

A global positioning system (GPS) is a system of satellites, computers, and receivers that is able to determine the latitude and longitude of a receiver on Earth by calculating the time difference for signals from different satellites to reach the receiver.

4.0 Interferences

4.1 Commercially available GPS units typically have real-time sub-meter accuracy. Field corrections can be made as described in Section 8.3 below.

5.0 Training and Qualifications

5.1 Qualifications and Training

5.1.1 The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 Responsibilities

5.2.1 The **Task Order (TO) Manager** is responsible for ensuring that land surveying activities comply with this procedure. The TO Manager is responsible for ensuring that all field sampling personnel involved in land surveying shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

5.2.3 The **Site Supervisor (SS)** is responsible for ensuring that all field personnel follow these procedures. In virtually all cases, subcontractors will conduct these procedures. The SS or designee is responsible for overseeing the activities of the subcontractor and ensuring that sampling points and topographic features are properly surveyed.

6.0 Equipment and Supplies

6.1 The following equipment list contains materials that may be needed in carrying out the procedures outlined in this SOP. Not all equipment listed below may be necessary for a specific activity. Additional equipment may be required, pending field conditions.

- Personal protective equipment (PPE) and other safety equipment, as required by the APP/SSHP;
- Commercially available GPS unit; and,
- Field Logbook.

7.0 Calibration or Standardization

7.1 An authorized manufacturer's representative shall inspect and calibrate survey instruments in accordance with the manufacturer's specifications regarding procedures and frequencies. At a minimum, instruments shall be calibrated no more than six months prior to the start of the survey work.

7.2 Standards for all survey work shall be in accordance with National Oceanic and Atmospheric Administration standards and, at a minimum, with accuracy standards set forth below. The horizontal accuracy for the location of all grid intersection and planimetric features shall be (±) 0.1 feet. The horizontal accuracy for boundary surveys shall be 1 in 10,000 feet (1:10,000). The vertical accuracy for ground surface elevations shall be (±) 0.1 feet. Benchmark elevation accuracy and elevation of other permanent features, including monitoring wellheads, shall be (±) 0.01 feet.

8.0 Procedure

8.1 Theodolite/Electronic Distance Measurement (EDM)

Follow the procedures listed below during theodolite/EDM land surveying:

- A land surveyor registered in the state or territory in which the work is being performed shall directly supervise all surveying work.
- Reference surveys to the local established coordinate systems.
- Reference surveyed points to site designated vertical datum, such as Mean Sea Level (Lower Low Water Level) and North American Vertical Datum of 1988 (NAVD 88).
- Jointly determine appropriate horizontal and vertical control points prior to the start of survey activities. If discrepancies in the survey (e.g., anomalous water level elevations) are observed, the surveyor may be required to verify the survey by comparison to a known survey mark. If necessary, a verification survey may be conducted by a qualified third party.
- All field notes, sketches, and drawings shall clearly identify the horizontal and vertical control points by number designation, description, coordinates, and elevations. Map all surveyed locations using a base map or other site mapping, as specified by the project Quality Assurance Project Plan (QAPP).
- Begin and end all surveys at the designated horizontal and vertical control points to determine the degree of accuracy of the surveys.
- Iron pins used to mark control points shall be made of reinforcement steel or an equivalent material and shall be 18 inches long with a minimum diameter of 5/8 inch. Drive pins to a depth of 18 inches into the soil.
- Stakes used to mark survey lines and points shall be made from 3-foot lengths of 2-inch by 2-inch lumber and pointed at one end. Clearly mark them with brightly coloured weatherproof flagging and paint.
- Clearly mark the point on a monitoring well casing or well riser that is surveyed by filing grooves into the casing/riser on either side of the surveyed point, or by marking the riser with a permanent ink marker.

8.2 Global Positioning System to Conduct Land Survey

Follow the procedures listed below during land surveying using GPS:

- A land surveyor registered in the state or territory in which the work is being performed shall directly supervise all surveying work.
- Reference surveys to the local established coordinate systems.
- All field notes, sketches, and drawings shall clearly identify the horizontal and vertical control points by number designation, description, coordinates, and elevations. Map all surveyed locations using a base map or other site mapping, as specified in the project QAPP.
- Begin and end all surveys at the designated horizontal and vertical control points (as applicable) to determine the degree of accuracy of the surveys.

- Iron pins used to mark control points shall be made of reinforcement steel or an equivalent material and shall be 18 inches long with a minimum diameter of 5/8 inch. Drive pins to a depth of 18 inches into the soil.
- Stakes used to mark survey lines and points shall be made from 3-foot lengths of 2-inch by 2-inch lumber and pointed at one end. Clearly mark them with brightly coloured weatherproof flagging and paint.
- Clearly mark the point on a monitoring well casing that is surveyed by filing grooves into the casing on either side of the surveyed point.

8.3 Global Positioning System to Position Sample Locations or Locate Site Features

Experienced field personnel may use a GPS system unit to position sample locations (e.g. grid positioned samples, soil boring locations) at a site. The decision to use field personnel or a licensed land surveyor will depend on the objectives of the survey (e.g. vertical elevation is not required) and the levels of precision required. Typically, when a level of accuracy greater than 0.03 meter is required, a licensed surveyor will be required. When a level of accuracy of (±) 1 meter is sufficient to meet project requirements (i.e. when laying sampling grids, identifying significant site features, or locating features identified in geographic information system [GIS] figures) experienced field personnel may use commercially available, consumer-grade GPS units. Follow the procedures listed below to locate samples or site features using GPS:

- A commercially available Trimble Geo 7X high-accuracy Global Navigation Satellite System.
- If waypoints are to be imported into a GIS database, the same grid projection system should be used.
- If a permanent reference point near the site is available, it is recommended that a waypoint at this location be taken every day waypoints are stored.
- When laying out a sampling grid from a GIS map, upload the coordinates from GIS to the GPS unit, including coordinates for an easily identified, permanent, nearby feature (i.e. building corner, roadway intersection, or United States Geological Survey benchmark).
- If during the initial site walk, the permanent feature identified does not overlay within (±) 1 meter as identified in the GPS unit, field corrections of the waypoints should be made.
- Field corrections can be made by adding/subtracting the difference in x,y coordinates between the field measurement of the permanent site feature and the anticipated x,y coordinates. This correction should then be applied to the x,y coordinates for each sampling location to be marked. Corrected x,y coordinates can then be uploaded into the GPS unit.
- Sampling points and site features can then be located in the field using the GPS units “Go To” function. When the distance to the sampling point or feature remains close to zero, the location can be marked.
- If no field corrections to the sampling location need to be made, or if sampling locations are to be surveyed by a licensed surveyor at a later date, no additional waypoints need to be taken. If significant changes to the sampling location are made, GPS coordinates at the corrected location shall be stored and labelled.
- GPS files containing field coordinates must be uploaded to a storage device such as PC at the end of each day. A new GPS file must be created for each day in the field.
- Field logs shall indicate manufacturer and model number for GPS unit used, map datum and projection used, and any field corrections made. If the GPS unit cannot lock onto a Wide Area Augmentation System (WAAS) system at the site, this should also be noted.

9.0 Quality Control and Assurance

GPS field data to be differentially corrected and imported into GIS and checked for accuracy on a daily basis.

10.0 Data and Records Management

The surveyor shall record field notes daily using generally accepted practices. The data shall be neat, legible, in indelible ink, and easily reproducible. Copies of the surveyor's field notes and calculation forms generated during the work shall be obtained and placed in the project files.

Surveyor's field notes shall, at a minimum, clearly indicate:

- The date of the survey;
- General weather conditions;
- The name of the surveying firm;
- The names and job titles of personnel performing the survey work;
- Equipment used, including serial numbers; and,
- Field book designations, including page numbers.

A land surveyor registered in the state or territory in which the work was done shall sign, seal, and certify the drawings and calculations submitted by the surveyor.

Dated records of land surveying equipment calibration shall be provided by the surveyor and placed in the project files. Equipment serial numbers shall be provided in the calibration records.

11.0 Attachments or References

Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

Author	Reviewer	Revisions (Technical or Editorial)
Robert Shoemaker Senior Scientist	Naomi Ouellette, Project Manager	Rev 0 – Initial Issue
Joshua Millard Geologist	James Bourdeau GIS Specialist	Rev 1 – Technical
Ken O'Donnell, PG Geologist	Claire Mitchell, PE, PMP Senior Engineer	Rev 2 – PFAS sampling update (July 2019)

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Soil and Rock Classification

Procedure 3-16

1.0 Purpose and Scope

1.1 The purpose of this document is to define the standard operating procedure (SOP) to thoroughly describe the physical characteristics of the sample and classify it according to the Unified Soil Classification System (USCS).

1.2 This procedure is the Program-approved professional guidance for work performed by AECOM under the client contract.

1.3 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review. If there are procedures whether it be from AECOM, state and/or federal that are not addressed in this SOP and are applicable to surface water sampling then those procedures may be added as an appendix to the project-specific Quality Assurance Project Plan (QAPP).

1.4 It is fully expected that the procedures outlined in this SOP will be followed. Procedural modifications may be warranted depending upon field conditions, equipment limitations, or limitations imposed by the procedure. Substantive modification to this SOP will be approved in advance by the Program Quality Manager. Deviations to this SOP will be documented in the field records.

2.0 Safety

2.1 Depending upon the site-specific contaminants, various protective programs must be implemented prior to sampling. All **field sampling personnel** responsible for sampling activities must review the project-specific Accident Prevention Plan (APP)/Site Safety and Health Plan (SSHP), paying particular attention to the control measures planned for the sampling tasks. Conduct preliminary area monitoring to determine the potential hazard to field sampling personnel. If significant contamination is observed, minimize contact with potential contaminants in both the vapor and liquid phase through the use of respirators and disposable clothing.

2.2 In addition, observe standard health and safety practices according to the project-specific APP/SSHP. Suggested minimum protection during well sampling activities includes inner disposable vinyl gloves, outer chemical-protective nitrile gloves, rubberized steel-toed boots, and an American National Standards Institute-standard hard hat. Half-face respirators and cartridges and Tyvek® suits may be necessary depending on the contaminant concentrations, and shall always be available on site.

2.3 Daily safety briefs will be conducted at the start of each working day before any work commences. These daily briefs will be facilitated by the **Site Safety and Health Officer (SSHO)** or designee to discuss the day's events and any potential health risk areas covering every aspect of the work to be completed. Weather conditions are often part of these discussions. As detailed in the APP/SSHP, everyone on the field team has the authority to stop work if an unsafe condition is perceived until the conditions are fully remedied to the satisfaction of the SSHO.

2.4 The health and safety considerations for the work associated with soil classification include:

- At no time during classification activities are personnel to reach for debris near machinery that is in operation, place any samples in their mouth, or come in contact with the soils/rocks without the use of gloves.

- Stay clear of all moving equipment and be aware of pinch points on machinery. Avoid wearing loose fitting clothing.
- When using cutting tools, cut away from yourself. The use of appropriate, task specific cutting tools is recommended.
- To avoid heat/cold stress as a result of exposure to extreme temperatures and PPE, drink electrolyte replacement fluids (1 to 2 cups per hour is recommended) and in case of extreme cold, wear insulating clothing.

3.0 Terms and Definitions

None.

4.0 Interference

None.

5.0 Training and Qualifications

5.1 The **Task Order (TO) Manager** is responsible for ensuring that the soil and rock classification procedures comply with this procedure. The **TO Manager** is responsible for ensuring that all personnel involved in soil and rock classification shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

5.3 The **Site Supervisor (SS)** is responsible for ensuring that all project **field personnel** follow these procedures.

5.4 Field personnel are responsible for the implementation of this procedure. Minimum qualifications for **field sampling personnel** require that one individual on the field team shall have a minimum of 6 months of experience with soil and rock classification.

5.5 The **project geologist** and/or **task manager** is responsible for directly supervising the soil and rock classification procedures to ensure that they are conducted according to this procedure, and for recording all pertinent data collected. If deviations from the procedure are required because of anomalous field conditions, they must first be approved by the **Program Quality Manager** and then documented in the field logbook and associated report or equivalent document.

6.0 Equipment and Supplies

6.1 The following equipment list contains materials which may be needed in carrying out the procedures outlined in this SOP. Not all equipment listed below may be necessary for a specific activity. Additional equipment may be required, pending field conditions.

- Personal protective equipment (PPE) and other safety equipment, as required by the APP/SSHP
- Field log book and pen with indelible ink
- Boring log
- Munsell Soil Color Chart
- Scoopula, spatula, and/or other small hand tools
- California Sampler
- Hand-held penetrometer

7.0 Calibration or Standardization

None.

8.0 Procedure

8.1 Soil Classification

The basic purpose of the classification of soil is to thoroughly describe the physical characteristics of the sample and to classify it according to an appropriate soil classification system. The USCS was developed so that soils could be described on a common basis by different investigators and serve as a "shorthand" description of soil. A classification of a soil in accordance with the USCS includes not only a group symbol and name, but also a complete word description.

Describing soil on a common basis is essential so that soil described by different site qualified personnel is comparable. Site individuals describing soil as part of site activities *must* use the classification system described herein to provide the most useful geologic database for all present and future subsurface investigations and remedial activities.

The site geologist or other qualified individual shall describe the soil and record the description in a boring log, logbook, and/or electronic field data collection device. The essential items in any written soil description are as follows:

- Classification group name (e.g., silty sand)
- Color, moisture, and odor
- Range of particle sizes and maximum particle size
- Approximate percentage of boulders, cobbles, gravel, sand, and fines
- Plasticity characteristics of the fines
- In-place conditions, such as consistency, density, and structure
- USCS classification symbol

The USCS serves as ”shorthand“ for classifying soil into 15 basic groups:

GW[1] Well graded (poorly sorted) gravel (>50 percent gravel, <5percent fines)

GP[1] Poorly graded (well sorted) gravel (>50percent gravel, <5percent fines)

GM[1] Silty gravel (>50 percent gravel, >15 percent silt)

GC[1] Clayey gravel (>50 percent gravel, >15 percent clay)

SW[1] Well graded (poorly sorted) sand (>50 percent sand, <5 percent fines)

SP[1] Poorly graded (well sorted) sand (>50 percent sand, <5 percent fines)

SM[1] Silty sand (>50 percent sand, >15 percent silt)

SC[1] Clayey sand (>50 percent sand, >15 percent clay)

ML[2] Inorganic, low plasticity silt (slow to rapid dilatancy, low toughness, and plasticity)

[1] If percentage of fine is 5 percent to 15 percent, a dual identification shall be given (e.g., a soil with more than 50 percent poorly sorted gravel and 10 percent clay is designated GW-GC.

CL[2] Inorganic, low plasticity (lean) clay (no or slow dilatancy, medium toughness and plasticity)

MH[2] Inorganic elastic silt (no to slow dilatancy, low to medium toughness and plasticity)

CH[2] Inorganic, high plasticity (fat) clay (no dilatancy, high toughness, and plasticity)

OL Organic low plasticity silt or organic silty clay

OH Organic high plasticity clay or silt

PT Peat and other highly organic soil

Figure8-1 defines the terminology of the USCS. Flow charts presented in Figure 8-2 and indicate the process for describing soil. The particle size distribution and the plasticity of the fines are the two properties of soil used for classification. In some cases, it may be appropriate to use a borderline classification (e.g., SC/CL) if the soil has been identified as having properties that do not distinctly place the soil into one group.

8.1.1 Estimation of Particle Size Distribution

One of the most important factors in classifying a soil is the estimated percentage of soil constituents in each particle size range. Being proficient in estimating this factor requires extensive practice and frequent checking. The steps involved in determining particle size distribution are listed below:

1. Select a representative sample (approximately 1/2 of a 6-inch long by 2.5-inch diameter sample liner).
2. Remove all particles larger than 3 inches from the sample. Estimate and record the percent by volume of these particles. Only the fraction of the sample smaller than 3 inches is classified.
3. Estimate and record the percentage of dry mass of gravel (less than 3 inches and greater than 1/4 inch).
4. Considering the rest of the sample, estimate, and record the percentage of dry mass of sand particles (about the smallest particle visible to the unaided eye).
5. Estimate and record the percentage of dry mass of fines in the sample (do not attempt to separate silts from clays).
6. Estimate percentages to the nearest 5 percent. If one of the components is present in a quantity considered less than 5 percent, indicate its presence by the term “trace”.
7. The percentages of gravel, sand, and fines must add up to 100 percent. “Trace” is not included in the 100 percent total.

8.1.2 Soil Dilatancy, Toughness, and Plasticity

8.1.2.1 Dilatancy

To evaluate dilatancy, follow these procedures:

1. From the specimen, select enough material to mold into a ball about 1/2 inch (12 millimeters [mm]) in diameter. Mold the material, adding water if necessary, until it has a soft, but not sticky, consistency.
2. Smooth the soil ball in the palm of one hand with the blade of a knife or small spatula. Shake horizontally, striking the side of the hand vigorously against the other hand several times. Note the reaction of water appearing on the surface of the soil. Squeeze the sample by closing the hand or

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[2] If the soil is estimated to have 15 percent to 25 percent sand or gravel, or both, the words “with sand” or “with gravel” (whichever predominates) shall be added to the group name (e.g., clay with sand, CL; or silt with gravel, ML). If the soil is estimated to have 30 percent or more sand or gravel, or both, the words “sandy” or “gravely” (whichever predominates) shall be added to the group name (e.g., sandy clay, CL). If the percentage of sand is equal to the percent gravel, use “sandy.”

pinching the soil between the fingers, and note the reaction as none, slow, or rapid in accordance with the criteria in Table 8-1. The reaction is the speed with which water appears while shaking and disappears while squeezing.

Table 8-1: Criteria for Describing Dilatancy

Description	Criteria
None	No visible change in specimen.
Slow	Water appears slowly on the surface of the specimen during shaking and does not disappear or disappears slowly upon squeezing.
Rapid	Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing.

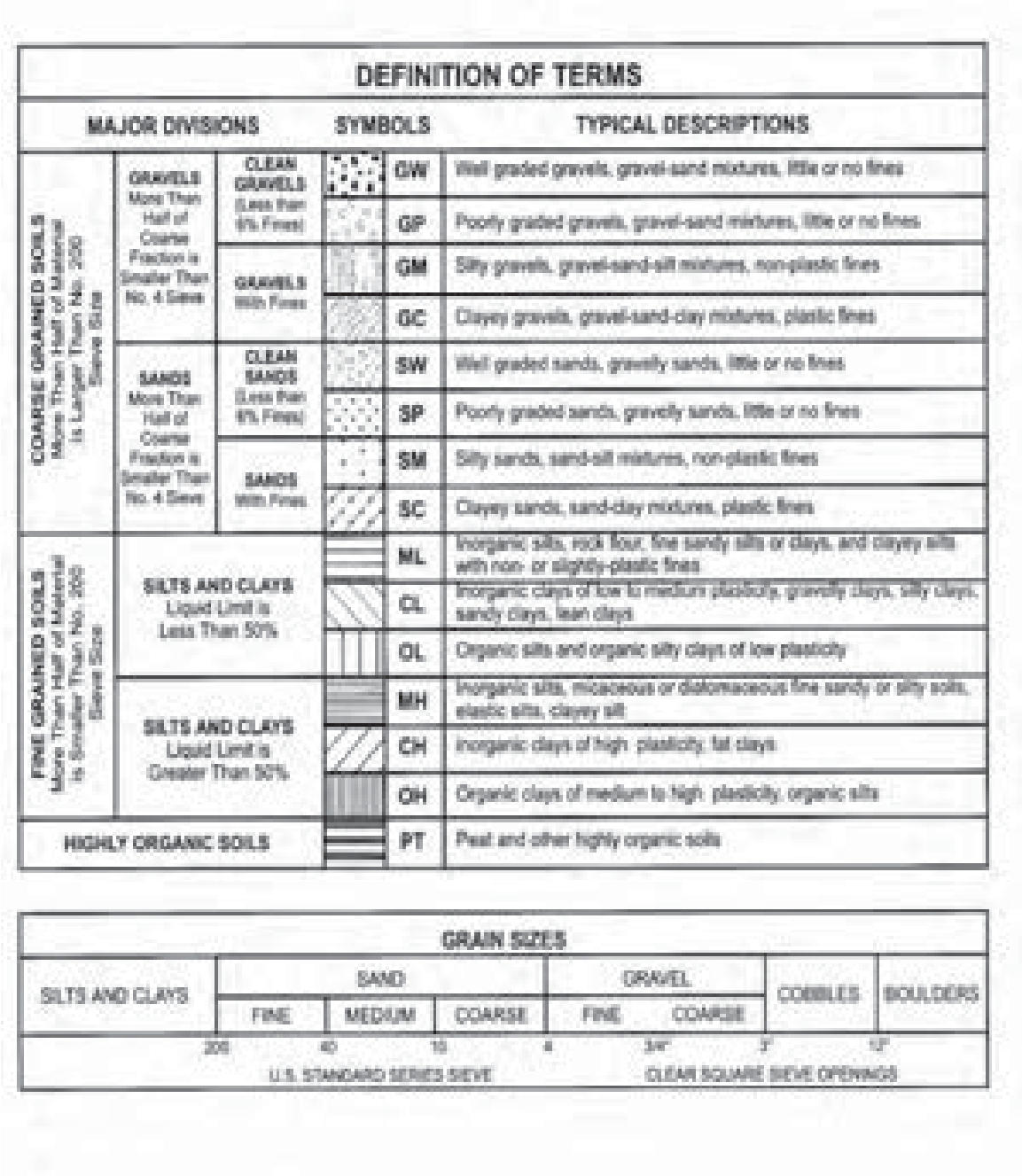
8.1.2.2 Toughness

Following the completion of the dilatancy test, shape the test specimen into an elongated pat and roll it by hand on a smooth surface or between the palms into a thread about 1/8 inch (3 mm) in diameter. (If the sample is too wet to roll easily, spread it into a thin layer and allow it to lose some water by evaporation.) Fold the sample threads and re-roll repeatedly until the thread crumbles at a diameter of about 1/8 inch. The thread will crumble at a diameter of 1/8 inch when the soil is near the plastic limit. Note the pressure required to roll the thread near the plastic limit. Also, note the strength of the thread. After the thread crumbles, lump the pieces together and knead it until the lump crumbles. Note the toughness of the material during kneading. Describe the toughness of the thread and lump as low, medium, or high in accordance with the criteria in Table 8-2.

Table 8-2: Criteria for Describing Toughness

Description	Criteria
Low	Only slight pressure is required to roll the thread near the plastic limit. The thread and the lump are weak and soft.
Medium	Medium pressure is required to roll the thread near the plastic limit. The thread and the lump have medium stiffness.
High	Considerable pressure is required to roll the thread near the plastic limit. The thread and the lump have very high stiffness.

Figure8-1: Unclassified Soil Classification System (USCS)



DEFINITION OF TERMS

MAJOR DIVISIONS			SYMBOLS	TYPICAL DESCRIPTIONS
COARSE GRAINED SOILS More Than Half of Material is Larger Than No. 200 Sieve Size	GRAVELS More Than Half of Coarse Fraction is Smaller Than No. 4 Sieve	CLEAN GRAVELS (Less than 6% Fines)	GW	Well graded gravels, gravel-sand mixtures, little or no fines
			GP	Poorly graded gravels, gravel-sand mixtures, little or no fines
		GRAVELS With Fines	GM	Silty gravels, gravel-sand-silt mixtures, non-plastic fines
			GC	Clayey gravels, gravel-sand-clay mixtures, plastic fines
	SANDS More Than Half of Coarse Fraction is Smaller Than No. 4 Sieve	CLEAN SANDS (Less than 6% Fines)	SW	Well graded sands, gravelly sands, little or no fines
			SP	Poorly graded sands, gravelly sands, little or no fines
		SANDS With Fines	SM	Silty sands, sand-silt mixtures, non-plastic fines
			SC	Clayey sands, sand-clay mixtures, plastic fines
FINE GRAINED SOILS More Than Half of Material is Smaller Than No. 200 Sieve Size	SILTS AND CLAYS Liquid Limit is Less Than 50%		ML	Inorganic silts, rock flour, fine sandy silts or clays, and clayey silts with non- or slightly-plastic fines
			CL	Inorganic clays of low to medium plasticity, gravelly clays, silty clays, sandy clays, lean clays
			OL	Organic silts and organic silty clays of low plasticity
	SILTS AND CLAYS Liquid Limit is Greater Than 50%		MH	Inorganic silts, micaceous or diatomaceous fine sandy or silty soils, elastic silts, clayey silt
			CH	Inorganic clays of high plasticity, fat clays
			OH	Organic clays of medium to high plasticity, organic silts
HIGHLY ORGANIC SOILS			PT	Peat and other highly organic soils

GRAIN SIZES

SILTS AND CLAYS	SAND			GRAVEL		COBBLES	BOULDERS
	FINE	MEDIUM	COARSE	FINE	COARSE		

200 40 10 4 3/4" 3" 12"

U.S. STANDARD SERIES SIEVE — CLEAR SQUARE SIEVE OPENINGS

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

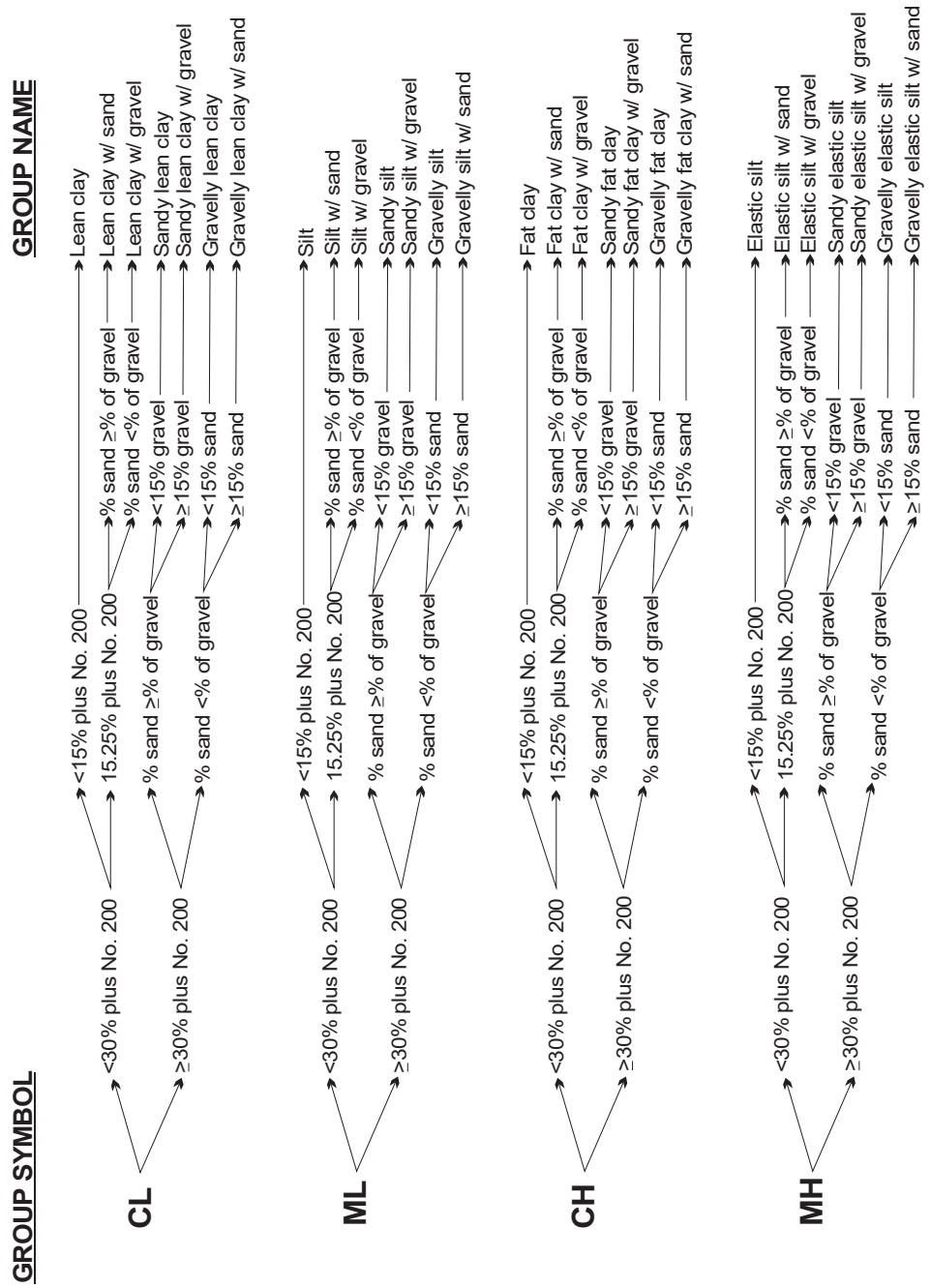
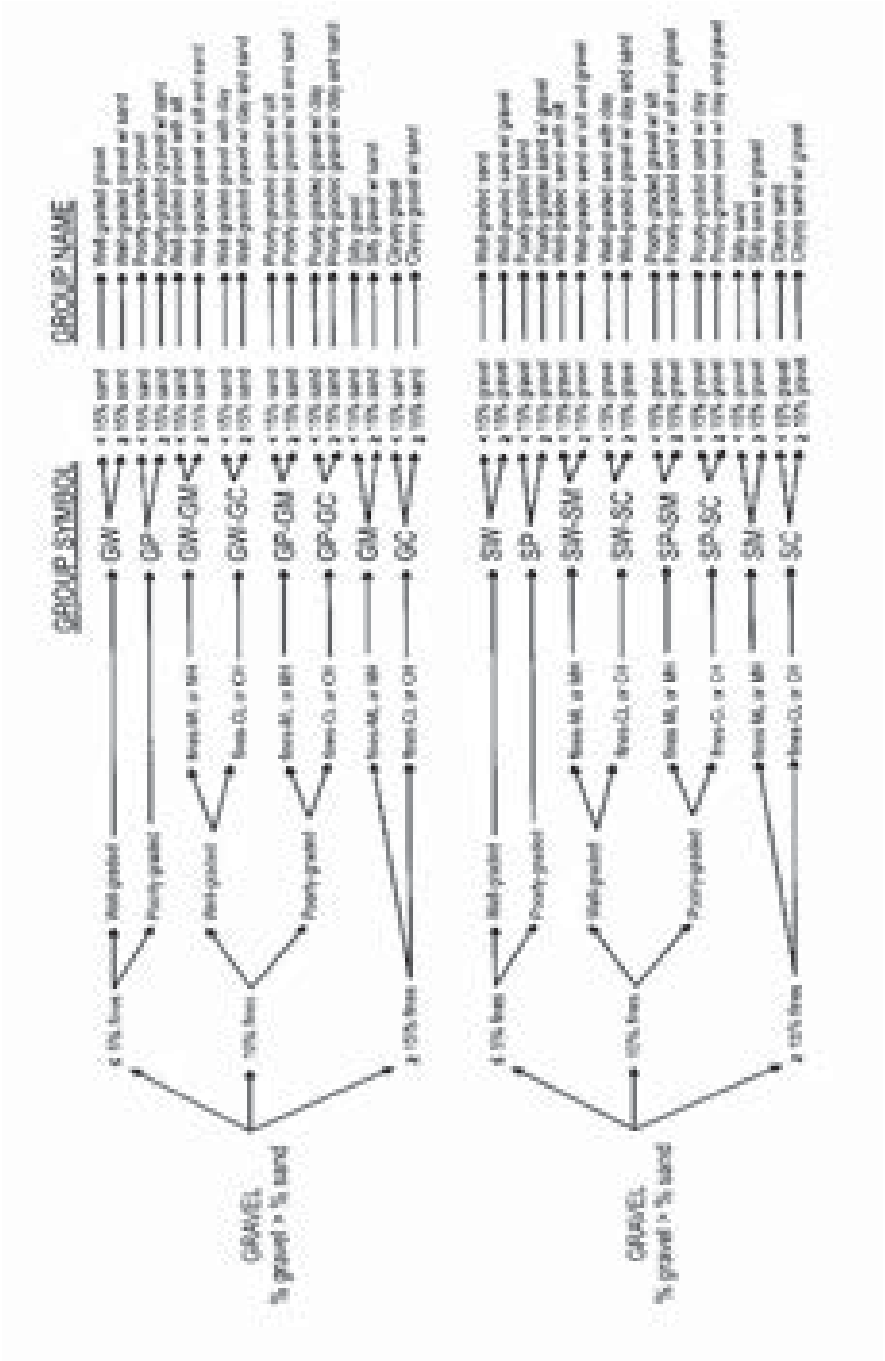


Figure 8-3: Flow Chart for Soil with Gravel



8.1.2.3 Plasticity

The plasticity of a soil is defined by the ability of the soil to deform without cracking, the range of moisture content over which the soil remains in a plastic state, and the degree of cohesiveness at the plastic limit. The plasticity characteristic of clays and other cohesive materials is defined by the liquid limit and plastic limit. The liquid limit is defined as the soil moisture content at which soil passes from the liquid to the plastic state as moisture is removed. The test for the liquid limit is a laboratory, not a field, analysis.

The plastic limit is the soil moisture content at which a soil passes from the plastic to the semi-solid state as moisture is removed. The plastic limit test can be performed in the field and is indicated by the ability to roll a 1/8-inch (0.125-inch) diameter thread of fines, the time required to roll the thread, and the number of times the thread can be re-rolled when approaching the plastic limit.

The plasticity tests are not based on natural soil moisture content, but on soil that has been thoroughly mixed with water. If a soil sample is too dry in the field, add water prior to performing classification. If a soil sample is too sticky, spread the sample thin and allow it to lose some soil moisture.

Table 8-3 presents the criteria for describing plasticity in the field using the rolled thread method.

Table 8-3: Criteria for Describing Plasticity

Description	Criteria
Non-Plastic	A 1/8-inch thread cannot be rolled.
Low Plasticity	The thread can barely be rolled.
Medium Plasticity	The thread is easy to roll and not much time is required to reach the plastic limit.
High Plasticity	It takes considerable time rolling the thread to reach the plastic limit.

8.1.3 Angularity

The following criteria describe the angularity of the coarse sand and gravel particles:

- **Rounded** particles have smoothly-curved sides and no edges.
- **Subrounded** particles have nearly plane sides but have well-rounded corners and edges.
- **Subangular** particles are similar to angular but have somewhat rounded or smooth edges.
- **Angular** particles have sharp edges and relatively plane sides with unpolished surfaces. Freshly broken or crushed rock would be described as angular.

8.1.4 Color, Moisture, and Odor

The natural moisture content of soil is very important. Table **8-4** shows the terms for describing the moisture condition and the criteria for each.

Table 8-4: Soil Moisture Content Qualifiers

Qualifier	Criteria
Dry	Absence of moisture, dry to the touch
Moist	Damp but no visible water
Wet	Visible water, usually soil is below water table

Color is described by hue and chroma using the Munsell Soil Color Chart (Munsell 2000). For uniformity, all site geologists shall utilize this chart for soil classification. Doing so will facilitate correlation of geologic units between boreholes logged by different geologists. The Munsell Color Chart is a small booklet of numbered color chips with names like “5YR 5/6, yellowish-red.” Note mottling or banding of colors. It is particularly important to note and describe staining because it may indicate contamination.

In general, wear a respirator if strong organic odors are present. If odors are noted, describe them if they are unusual or suspected to result from contamination. An organic odor may have the distinctive smell of decaying vegetation. Unusual odors may be related to hydrocarbons, solvents, or other chemicals in the subsurface. An organic vapor analyzer may be used to detect the presence of volatile organic contaminants.

8.1.5 In-Place Conditions

Describe the conditions of undisturbed soil samples in terms of their density/consistency (i.e., compactness), cementation, and structure utilizing the following guidelines:

8.1.5.1 Density/Consistency

Density and consistency describe a physical property that reflects the relative resistance of a soil to penetration. The term “density” is commonly applied to coarse to medium-grained sediments (i.e., gravels, sands), whereas the term “consistency” is normally applied to fine-grained sediments (i.e., silts, clays). There are separate standards of measure for both density and consistency that are used to describe the properties of a soil.

The density or consistency of a soil is determined by observing the number of blows required to drive a 1 3/8-inch (35 mm) diameter split barrel sampler 18 inches using a drive hammer weighing 140 lbs. (63.5 kilograms [kg]) dropped over a distance of 30 inches (0.76 meters). Record the number of blows required to penetrate each 6 inches of soil in the field boring log during sampling. The first 6 inches of penetration is considered to be a seating drive; therefore, the blow count associated with this seating drive is recorded, but not used in determining the soil density/consistency. The sum of the number of blows required for the second and third 6 inches of penetration is termed the “standard penetration resistance,” or the “N-value.” The observed number of blow counts must be corrected by an appropriate factor if a different type of sampling device (e.g., Modified California Sampler with liners) is used. For a 2 3/8-inch inner diameter (I.D.) Modified California Sampler equipped with brass or stainless-steel liners and penetrating a cohesionless soil (sand/gravel), the N-value from the Modified California Sampler must be divided by 1.43 to provide data that can be compared to the 1 3/8-inch diameter sampler data.

For a cohesive soil (silt/clay), the N-value for the Modified California Sampler should be divided by a factor of 1.13 for comparison with 1 3/8-inch diameter sampler data.

Drive the sampler and record blow counts for each 6-inch increment of penetration until one of the following occurs:

- A total of 50 blows have been applied during any one of the three 6-inch increments; a 50-blow count occurrence shall be termed “refusal” and noted as such on the boring log.
- A total of 150 blows have been applied.
- The sampler is advanced the complete 18 inches without the limiting blow counts occurring, as described above.

If the sampler is driven less than 18 inches, record the number of blows per partial increment on the boring log. If refusal occurs during the first 6 inches of penetration, the number of blows will represent the N-value for this sampling interval. Table 8-5 8-5 and Table 8-6 present representative descriptions of soil density/consistency vs. N-values.

Table 8-5: Measuring Soil Density with a California Sampler – Relative Density (Sands, Gravels)

Description	Field Criteria (N-Value)	
	1 3/8 in. ID Sampler	2 in. ID Sampler using 1.43 factor
Very Loose	0–4	0–6
Loose	4–10	6–14
Medium Dense	10–30	14–43

Description	Field Criteria (N-Value)	
	1 3/8 in. ID Sampler	2 in. ID Sampler using 1.43 factor
Dense	30–50	43–71
Very Dense	> 50	> 71

Table 8-6: Measuring Soil Density with a California Sampler – Fine Grained Cohesive Soil

Description	Field Criteria (N-Value)	
	1 3/8 in. ID Sampler	2 in. ID Sampler using 1.13 factor
Very Soft	0–2	0–2
Soft	2–4	2–4
Medium Stiff	4–8	4–9
Stiff	8–16	9–18
Very Stiff	16–32	18–36
Hard	> 32	> 36

For undisturbed fine-grained soil samples, it is also possible to measure consistency with a hand-held penetrometer. The measurement is made by placing the tip of the penetrometer against the surface of the soil contained within the sampling liner or Shelby tube, pushing the penetrometer into the soil a distance specified by the penetrometer manufacturer, and recording the pressure resistance reading in pounds per square foot (psf). The values are as follows (Table 8-7):

Table 8-7: Measuring Soil Consistency with a Hand-Held Penetrometer

Description	Pocket Penetrometer Reading (psf)
Very Soft	0–250
Soft	250–500
Medium Stiff	500–1000
Stiff	1000–2000
Very Stiff	2000–4000
Hard	>4000

Consistency can also be estimated using thumb pressure using Table 8-8.

Table 8-8: Measuring Soil Consistency Using Thumb Pressure

Description	Criteria
Very Soft	Thumb will penetrate soil more than 1 inch (25 mm)
Soft	Thumb will penetrate soil about 1 inch (25 mm)
Firm	Thumb will penetrate soil about 1/4 inch (6 mm)
Hard	Thumb will not indent soil but readily indented with thumbnail
Very Hard	Thumbnail will not indent soil

8.1.5.2 Cementation

Cementation is used to describe the friability of a soil. Cements are chemical precipitates that provide important information as to conditions that prevailed at the time of deposition, or conversely, diagenetic effects that occurred following deposition. Seven types of chemical cements are recognized by Folk (1980). They are as follows:

- Quartz – siliceous

- Chert – chert-cemented or chalcedonic
- Opal – opaline
- Carbonate – calcitic, dolomitic, sideritic (if in doubt, calcareous should be used)
- Iron oxides – hematitic, limonitic (if in doubt, ferruginous should be used)
- Clay minerals – if the clay minerals are detrital or have formed by recrystallization of a previous clay matrix, they are not considered to be a cement. Only if they are chemical precipitates, filling previous pore space (usually in the form of accordion-like stacks or fringing radial crusts) should they be included as “kaolin-cemented,” “chlorite-cemented,” etc.
- Miscellaneous minerals – pyritic, collophane-cemented, glauconite-cemented, gypsiferous, anhydrite-cemented, baritic, feldspar-cemented, etc.

The degree of cementation of a soil is determined qualitatively by utilizing finger pressure on the soil in one of the sample liners to disrupt the gross soil fabric. The three cementation descriptors are as follows:

- Weak – friable; crumbles or breaks with handling or slight finger pressure
- Moderate – friable; crumbles or breaks with considerable finger pressure
- Strong – not friable; will not crumble or break with finger pressure

8.1.5.3 Structure

This variable is used to qualitatively describe physical characteristics of soil that are important to incorporate into hydrogeological and/or geotechnical descriptions of soil at a site. Appropriate soil structure descriptors are as follows:

- Granular – spherically shaped aggregates with faces that do not accommodate adjoining faces
- Stratified – alternating layers of varying material or color with layers at least 6 mm (1/4 inch) thick; note thickness
- Laminated – alternating layers of varying material or color with layers less than 6 mm (1/4 inch) thick; note thickness
- Blocky – cohesive soil that can be broken down into small angular or subangular lumps that resist further breakdown
- Lensed – inclusion of a small pocket of different soil, such as small lenses of sand, should be described as homogeneous if it is not stratified, laminated, fissured, or blocky. If lenses of different soil are present, the soil being described can be termed homogeneous if the description of the lenses is included
- Prismatic or Columnar – particles arranged about a vertical line, ped is bounded by planar, vertical faces that accommodate adjoining faces; prismatic has a flat top; columnar has a rounded top
- Platy – particles are arranged about a horizontal plane

8.1.5.4 Other Features

- Mottled – soil that appears to consist of material of two or more colors in blotchy distribution
- Fissured – breaks along definite planes of fracture with little resistance to fracturing (determined by applying moderate pressure to sample using thumb and index finger)
- Slickensided – fracture planes appear polished or glossy, sometimes striated (parallel grooves or scratches)

8.1.6 Development of Soil Description

Develop standard soil descriptions according to the following examples. There are three principal categories under which all soil can be classified. They are described below.

8.1.6.1 Coarse-grained Soil

Coarse-grained soil is divided into sands and gravels. A soil is classified as a sand if over 50 percent of the coarse fraction is "sand-sized." It is classified as a gravel if over 50 percent of the coarse fraction is composed of "gravel-sized" particles.

The written description of a coarse-grained soil shall contain, in order of appearance: Typical name including the second highest percentage constituent as an adjective, if applicable (underlined); grain size of coarse fraction; Munsell color and color number; moisture content; relative density; sorting; angularity; other features, such as stratification (sedimentary structures) and cementation, possible formational name, primary USCS classification, secondary USCS classification (when necessary), and approximate percentages of minor constituents (i.e., sand, gravel, shell fragments, rip-up clasts) in parentheses.

Example: POORLY-SORTED SAND WITH SILT, medium- to coarse-grained, light olive gray, 5Y 6/2, saturated, loose, poorly sorted, subrounded clasts, SW/SM (minor silt with approximately 20 percent coarse-grained sand-sized shell fragments, and 80 percent medium-grained quartz sand, and 5 percent to 15 percent ML).

8.1.6.2 Fine-grained Soil

Fine-grained soil is further subdivided into clays and silts according to its plasticity. Clays are rather plastic, while silts have little or no plasticity.

The written description of a fine-grained soil should contain, in order of appearance: Typical name including the second highest percentage constituent as an adjective, if applicable (underlined); Munsell color; moisture content; consistency; plasticity; other features, such as stratification, possible formation name, primary USCS classification, secondary USCS classification (when necessary), and the percentage of minor constituents in parentheses.

Example: SANDY LEAN CLAY, dusky red, 2.5 YR 3/2, moist, firm, moderately plastic, thinly laminated, CL (70 percent fines, 30 percent sand, with minor amounts of disarticulated bivalves [about 5 percent]).

8.1.6.3 Organic Soil

For highly organic soil, describe the types of organic materials present as well as the type of soil constituents present using the methods described above. Identify the soil as an organic soil, OL/OH, if the soil contains enough organic particles to influence the soil properties. Organic soil usually has a dark brown to black color and may have an organic odor. Often, organic soils will change color, (e.g., from black to brown) when exposed to air. Some organic soils will lighten in color significantly when air-dried. Organic soils normally will not have a high toughness or plasticity. The thread for the toughness test will be spongy.

8.2 Example: ORGANIC CLAY, black, 2.5Y, 2.5/1, wet, soft, low plasticity, organic odor, OL (100 percent fines), weak reaction to HCl.

8.3 Rock Classification

The purpose of rock classification is to thoroughly describe the physical and mineralogical characteristics of a specimen and to classify it according to an established system. The generalized rock classification system described below was developed because, unlike the USCS for soils, there is no universally accepted rock classification system. In some instances, a more detailed and thorough rock classification system may be appropriate. Any modifications to this classification system, or the use of an alternate classification system should be considered during preparation of the site work plan. Both the TO Manager

and the QA Manager or Technical Director must approve any modifications to this classification system, or the use of another classification system.

Describing rock specimens on a common basis is essential so that rocks described by different site geologists are comparable. Site geologists describing rock specimens as a part of investigative activities must use the classification system described herein, or if necessary, another more detailed classification system. Use of a common classification system provides the most useful geologic database for all present and future subsurface investigations and remedial activities.

In order to provide a more consistent rock classification between geologists, a rock classification template has been designated as shown in **Error! Reference source not found.**. The template includes classification of rocks by origin and mineralogical composition. When classifying rocks, all site geologists shall use this template.

The site geologist shall describe the rock specimen and record the description in a boring log or logbook. The items essential for classification include (i.e., metamorphic foliated):

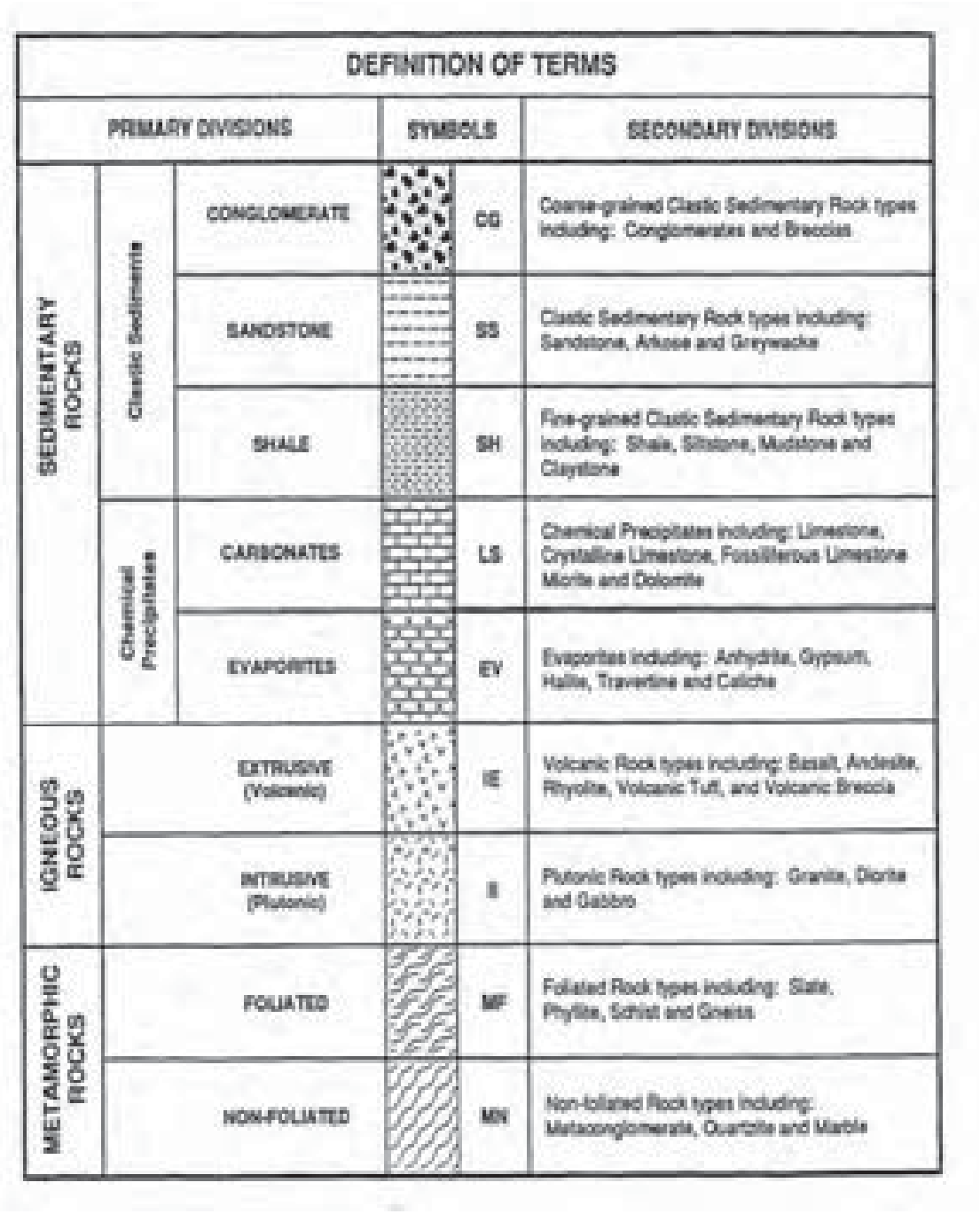
- Classification Name (i.e., schist)
- Color
- Mineralogical composition and percent
- Texture/Grain size (i.e., fine-grained, pegmatitic, aphlitic, glassy)
- Structure (i.e., foliated, fractured, lenticular)
- Rock Quality Designation (sum of all core pieces greater than two times the diameter of the core divided by the total length of the core run, expressed as a percentage)
- Classification symbol (i.e., MF)

Example: Metamorphic foliated schist: Olive gray, 5Y, 3/2, Garnet 25 percent, Quartz 45 percent, Chlorite 15 percent, Tourmaline 15 percent, Fine-grained with Pegmatite garnet, highly foliated, slightly wavy, MF.

9.0 Quality Control and Assurance

None

Figure 8-4: Rock Classification System



DEFINITION OF TERMS					
PRIMARY DIVISIONS				SYMBOLS	SECONDARY DIVISIONS
SEDIMENTARY ROCKS	Clastic Sediments	CONGLOMERATE		CG	Coarse-grained Clastic Sedimentary Rock types including: Conglomerates and Breccias
		SANDSTONE		SS	Clastic Sedimentary Rock types including: Sandstone, Arkose and Greywacke
		SHALE		SH	Fine-grained Clastic Sedimentary Rock types including: Shale, Siltstone, Mudstone and Claystone
	Chemical Precipitates	CARBONATES		LS	Chemical Precipitates including: Limestone, Crystalline Limestone, Fossiliferous Limestone Marlte and Dolomite
		EVAPORITES		EV	Evaporites including: Anhydrite, Gypsum, Halite, Travertine and Caliche
IGNEOUS ROCKS		EXTRUSIVE (Volcanic)		IE	Volcanic Rock types including: Basalt, Andesite, Rhyolite, Volcanic Tuff, and Volcanic Breccia
		INTRUSIVE (Plutonic)		II	Plutonic Rock types including: Granite, Diorite and Gabbro
METAMORPHIC ROCKS		FOLIATED		MF	Foliated Rock types including: Slate, Phyllite, Schist and Gneiss
		NON-FOLIATED		MN	Non-foliated Rock types including: Metaconglomerate, Quartzite and Marble

10.0 Data and Records Management

10.1 Document soil classification information collected during soil sampling onto the field boring logs, field trench logs, and into the field notebook. Copies of this information shall be sent to the **TO Manager** for the project files.

10.2 Field notes will be kept during coring activities in accordance with SOP 3-03 – Recordkeeping, Sample Labeling, and Chain of Custody. The information pertinent to soil classification activities includes chronology of events, sample locations (x,y,z), time/date, sampler name, methods (including type of core liner/barrel, if applicable), sampler penetration and acceptability, sample observations, and the times and type of equipment decontamination. Deviations to the procedures detailed in the SOP should be recorded in the field logbook.

11.0 Attachments or References

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Author	Reviewer	Revisions (Technical or Editorial)
Robert Shoemaker Senior Scientist	Naomi Ouellette, Project Manager	Rev 0 – Initial Issue
Ken O'Donnell, PG Geologist	Claire Mitchell, PE, PMP Senior Engineer	Rev 1 – PFAS sampling update (July 2019)

Direct Push Sampling Techniques

Procedure 3-17

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) provides guidance on the use of direct push techniques.

1.2 This procedure is the Program-approved professional guidance for work performed by AECOM under the client contract.

1.3 This procedure shall serve as management-approved professional guidance and is consistent with protocol in the Uniform Federal Policy-Quality Assurance Project Plan (UFP-QAPP; DoD 2005). As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved by both the Task Order (TO) Manager and the Quality Assurance (QA) Manager or Technical Director, and documented.

1.4 If there are procedures whether it be from AECOM, state and/or federal that are not addressed in this SOP and are applicable to direct push sampling then those procedures may be added as an appendix to the project-specific QAPP.

2.0 Safety

2.1 Field personnel shall perform work in accordance with the Accident Prevention Plan (APP)/Site Safety and Health Plan (SSHP). During monitoring well installation, subcontractors in direct contact with potentially contaminated media shall wear the proper personal protective equipment (PPE) as outlined in the APP/SSHP. Failure to comply will result in disciplinary action.

2.2 If circumstances warrant, a real-time immediate response instrument, such as a Miniram Dust Monitor, organic vapor analyzer, HNu, Thermo, Draeger or Sensidyne tubes, or explosimeter, should be used to monitor the work area. When real/time instrument response exceeds the permissible exposure limit, personnel shall don the appropriate PPE and alternate control measures to ensure personnel safety. If safe control measures are not achievable, field activities shall be discontinued immediately. Company-specific APP/SSHPs offer guidelines on air surveillance and on selection of PPE. In addition, the site-specific APP/SSHP includes an air monitoring program and suggested PPE.

2.3 In addition to the aforementioned precautions and depending upon the type of contaminant expected, employ the following safe work practices:

Particulate, Metal Compounds, or PFAS/PFOS

1. Avoid skin contact and/or incidental ingestion of soil.
2. Wear protective clothing, steel-toed boots, powderless Nitrile gloves with non-water-repellent protective leather gloves, safety glasses, and hearing protection as warranted.

VOCs

1. Avoid breathing constituents venting from holes by approaching upwind, and/or by use of respiratory protection.
2. Pre-survey the area with a flame ionization detector (FID) or photoionization detector (PID) prior to sampling.
3. If monitoring results indicate organic vapors that exceed action levels as specified in the site-specific APP/SSHP, sampling activities may need to be conducted in Level C protection. At a

minimum, skin protection will be required by use of gloves and Tyvek or other media that is protective against the media being encountered.

Flammable or Explosive Conditions

1. Monitor explosive gases as continuously as possible using an explosimeter and oxygen meter.
2. Place all ignition sources upwind or crosswind of the borehole.
3. If explosive gases exceed the designated action levels as specified in the site-specific APP/SSHP, cease operations and evaluate conditions.

Physical Hazards Associated with Soil Sampling

1. To avoid possible back strain associated with sample collection, use the large muscles of the legs, not the back, when retrieving soil samplers.
2. Stay clear of all moving equipment, and avoid wearing loose fitting clothing.
3. To avoid slip/trip/fall hazards, be wary of open trenches, pits, or holes.
4. Be aware of restricted mobility due to PPE.
5. To avoid hand, wrist, arm, shoulder, and back trauma due to the use of slide hammers or hand augers, rotate sampling among field personnel

3.0 Terms and Definitions

3.1 Direct push techniques are methods for subsurface sampling or monitoring that involve the application of downward pressure (usually supplied through hydraulic means) without the benefit of cutting tool rotation to enter soil. A variety of systems are available under several trade names, such as GeoProbe®. Equipment may be skid-mounted, trailered, or mounted directly on the frame of a vehicle.

4.0 Interferences

4.1 Potential interferences could result from cross-contamination between samples or sample locations. Minimization of the cross contamination will occur through the following:

- The use of clean sampling tools at each location as necessary.
- Avoidance of material that is not representative of the media to be sampled.

5.0 Training and Qualifications

5.1 Qualifications and Training

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 Responsibilities

5.2.1 The TO Manager is responsible for ensuring that these standard direct push technique procedures are followed during projects conducted under the ER Program and that a qualified individual conducts or supervises the projects. A qualified individual for subsurface sampling or monitoring using direct push techniques is defined as a person with a degree in geology, hydrogeology, or geotechnical/civil engineering with at least 1 year of experience supervising soil boring construction using conventional drilling or direct push techniques. The TO Manager or designee is responsible for ensuring that all personnel involved in direct push sampling techniques shall have the appropriate education, experience, and training to perform their assigned tasks as specified in Chief of Naval Operations Instruction 5090.1c (DON 2007).

5.2.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

5.2.3 The **Site Supervisor (SS)** is responsible for ensuring that all field personnel follow these procedures.

5.2.4 All **Field Personnel** are responsible for the implementation of this procedure.

5.2.5 The **Field Personnel** and/or **SS** are responsible for directly supervising the direct push sampling activities to ensure that they are conducted according to this procedure, and for recording all pertinent data collected during sampling.

6.0 Equipment and Supplies

In addition to those materials provided by the subcontractor, the project **SS/Field Personnel** will require:

- Boring Logs;
- Spoons or scoops;
- Sample kit (bottles, labels, custody records and tape, cooler, wet ice), if laboratory analysis is required;
- Sample collection pan;
- Folding rule or tape measure;
- Polyethylene plastic sheeting;
- Utility knife;
- Equipment decontamination materials (as described in SOP 3-06, *Equipment Decontamination*);
- Health and safety equipment (as required by APP/SSHP); and
- Non-water-repellent field project notebook/pen.

7.0 Procedure

Direct push techniques may be used as a cost-effective alternative to conventional drilling techniques for obtaining subsurface soil and groundwater samples and for monitoring subsurface conditions.

7.1 Method Selection

Base the decision to use direct push techniques on: (1) their ability to achieve the required information at the required level of quality control and (2) their cost-effectiveness compared to conventional drilling methods. Major limitations of direct push techniques are their inability to penetrate rock or cobbles and a shallow maximum depth of penetration. The capabilities of direct push systems vary significantly among vendors. Consider these differences in capabilities when evaluating the method for a subsurface exploration program.

Use direct push techniques to obtain groundwater samples for confirmatory analyses only if the screen placement method protects the screen from clogging during installation and allows the installation of a sand-pack around the exterior of the well screen.

7.2 Inspection of Equipment

Inspect direct push equipment prior to use for signs of fluid leakage, which could introduce contaminants to the soil. If, at any time during equipment operation, fluid is observed leaking from the rig, cease operations and immediately repair or contain the leak. Collect, containerize, and label soil and other materials affected by the leak for proper disposal (see SOP 3-05, *IDW Management*).

7.3 Preparation of Work Site

Inspect the work site prior to commencing operations to ensure that no overhead hazards exist that could impact the direct push equipment, and the work area should cleared and/or marked by the local underground utility locating service (e.g., DigSafe). In addition, clear locations planned for subsurface exploration using either geophysical methods and/or hand excavate locations to a depth of 2 to 3 feet prior to soil penetration, unless it is certain (by virtue of subsurface clearing activities) that no utilities or other hazardous obstructions will be encountered in the first 2 to 3 feet. Hand excavation may be waived when it is not practical.

Locate the direct push rig so that it is downslope from the penetration point, if the work is to be performed on a grade. Locate the rig downwind or crosswind of the penetration point, if possible. Cover the area surrounding, and in the vicinity of, the penetration point with plastic. Establish required exclusion zones using plastic tape or cones to designate the various areas.

7.4 Equipment Decontamination

To avoid cross-contamination, thoroughly decontaminate equipment used for direct push exploration and sampling as described in SOP 3-06, *Equipment Decontamination*. Decontaminate sampling tools and downhole equipment between each sampling event and between penetration points. At a minimum, steam clean or wash and rinse the equipment. Collect, containerize, and label all wash and rinse water for proper disposal. Clean equipment (e.g., drive rods and samplers) shall not come into contact with contaminated soils or other contaminated materials. Keep equipment on plastic or protect it in another suitable fashion. Store push rods and other equipment removed from a hole on plastic sheeting until properly decontaminated.

7.5 Soil Sampling

This SOP assumes that the subcontractor will perform sampling; therefore, detailed procedures regarding sample acquisition are not provided. Vendors of direct push equipment offer a variety of sampling systems designed specifically for their equipment. Both continuous and discreet soil samples may be obtained using sampling equipment similar to that described in Procedure 3-21, *Surface and Subsurface Soil Sampling*. The preferred methods for soil sampling using direct push techniques use stainless steel split-tube samplers that are driven through the horizon to be sampled. Use plastic sample tubes (e.g., Macro-Core Samplers) only for screening purposes or, in the case of confirmatory sampling, if samples will not be analyzed for volatile organic compounds (VOCs) or semivolatile organic compounds (SVOCs).

7.6 Groundwater Sampling

Direct push vendors offer numerous methods for obtaining groundwater samples. Key differences among methods involve: (1) the maximum well diameter achievable; (2) the ability to protect the well screen from exposure to contaminated overburden soils during installation; (3) the ability to install packing around the screen; (4) flexibility in the size, materials of construction, and design of well screens; and (5) the ability to convert sampling points into permanent monitoring wells. The limitations and abilities of a given system must be thoroughly understood and matched to the needs of the project before committing to the collection of groundwater samples using direct push techniques.

Use direct push techniques only to collect screening samples unless it is confirmed that the system:

1. Effectively protects the well screen from exposure to contaminated overburden soils during installation
2. Allows the installation of effective packing around the well screen
3. Allows the well screen to be effectively sealed against the downward infiltration of overlying groundwater or surface precipitation
4. Is constructed of materials compatible with the intended sampling and analysis goals of the project

5. Allows the use of a well screen properly sized and slotted for the needs of the project

Additional information on the collection of groundwater samples can be found in SOP 3-14 Monitoring Well Sampling.

It is the responsibility of the **TO Manager** to evaluate and determine the appropriateness of direct push systems prior to committing to their use on any project involving groundwater sampling. As part of this evaluation, it is recommended to obtain concurrence from regulatory authorities in advance for the method selection.

7.7 Borehole Abandonment

Methods for abandoning boreholes created with direct push systems will vary among vendors. Coordinate the desired method for abandonment with the vendor in the planning stages of the project to ensure proper abandonment.

Some direct push boreholes will close naturally as the drive rods and sampling tools are withdrawn. This may occur in loose, unconsolidated soils, such as sands. Close all boreholes using one of the procedures described in this procedure, unless natural caving precludes such closure.

The three methods for closing direct push boreholes are:

1. Add granulated or pelletized bentonite and hydrate in layers, proceeding from the bottom of the hole to the surface.
2. Pour premixed cement/water (or cement/water/bentonite) mixture into the hole.
3. Fill the entire hole with granular or pelletized bentonite and hydrate by means of a previously emplaced water tube that is gradually withdrawn as water is supplied to the bentonite.

The second method is recommended. For shallow holes less than 10 feet in depth, pour a cement/water/bentonite mix directly into the opening using a funnel. For deeper holes, use a conductor (tremie) pipe to carry the grout mix to the far reaches of the borehole. Lower the conductor pipe to within 2 inches of the bottom and gradually withdraw it as grout is added, keeping the lower end of the pipe submerged in grout at all times.

The recommended grout mixture for well abandonment is 7 to 9 gallons of water per 94-pound bag of Portland cement, with 3 percent to 5 percent by weight of powdered bentonite added to the mixture. Commercial products, such as Volcay are acceptable with pre-approval of the **TO Manager**.

Seal boreholes to within 0.5 to 2.0 feet of the surface. Inspect the abandoned borehole after 24 hours to ensure that grout shrinkage does not occur. If significant shrinkage has occurred, re-grout the borehole. Fill the remaining portion of the hole with local topsoil or appropriate paving materials.

8.0 Quality Control and Assurance

8.1 Collection of representative samples will be ensured through adherence to the procedures in this SOP and the sampling strategy outlined in the QAPP. The field quality control samples identified in the QAPP must be collected. These samples may include field duplicates, equipment rinsate blanks, trip blanks, and matrix spike/matrix spike duplicates

9.0 Records, Data Analysis, Calculations

9.1 Various forms are required to ensure that adequate documentation is made of the sample collection activities. These forms may include:

- Boring logs;
- Non-water-repellent field logbook;
- Sample collection records;

- Chain-of-custody forms; and
- Shipping labels.

9.2 Boring logs (Attachment 1) will provide visual and descriptive information for samples collected at each soil boring and are often the most critical form of documentation generated during a soil sampling program.

9.3 The field logbook is kept as a general log of activities and should not be used in place of the boring log.

9.4 Chain-of-custody forms are transmitted with the samples to the laboratory for sample tracking purposes.

9.5 Shipping labels are required is sample coolers are to be transported to a laboratory by a third party (courier service).

10.0 Attachments or References

10.1 Attachment 1 – Boring Log

10.2 Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

10.3 SOP 3-05, *IDW Management.*

10.4 SOP 3-06, *Equipment Decontamination.*

10.5 SOP 3-21, *Surface and Subsurface Soil Sampling.*

Author	Reviewer	Revisions (Technical or Editorial)
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (May 2012)
Ken O'Donnell, PG Geologist	Claire Mitchell, PE, PMP Senior Engineer	Rev 1 – PFAS sampling update (July 2019)

Attachment 1
Boring Log

Boring ID:

Page 1 of

Project Name:	Drilling Company:	Type of Surface Material:
Project Number:	Drilling Method:	Patching Material:
Date Started Drilling:	Rig Type:	Drilling Water Level:
Date Finished Drilling:	Core Size:	Boring Total Depth (bgs):
Physical Location:		Logged By:

Depth Range	Recovery (in)	PID (ppm)	Moisture Content	GA Class	USCS	GA Class: Garfield Avenue Sites classification & Modified Unified Soil Classification System	
						Ground Surface Cover and Thickness:	Sample Name & #:
0-1							
1-2							
2-3							
3-4							
4-5							
5-6							
6-7							
7-8							
8-9							
9-10							
10-11							
11-12							
12-13							
13-14							
14-15							
15-16							
16-17							
17-18							
18-19							
19-20							

Stratigraphic Unit Intervals:		Comments:
1)	5)	
2)	6)	
3)	7)	
4)	8)	

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Headspace Screening for Total VOCs

Procedure 3-19

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes the basic techniques for using headspace analysis to screen for volatile organics in contaminated soils using a portable Photo Ionization Detector (PID) or Flame Ionization Detector (FID).

1.2 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

2.1 The health and safety considerations for the work associated with this SOP will be addressed in the project Accident Prevention Plan (APP)/Site Safety and Health Plan (SSHP). Work will also be conducted according to the Task Order (TO) Quality Assurance Project Plan (QAPP) and/or direction from the **Site Safety and Health Officer (SSHO)**. Note that headspace screening usually requires Level D personal protection unless there is a potential for airborne exposure to site contaminants. Under circumstances where potential airborne exposure is possible respiratory protective equipment may be required based on personal air monitoring results. Upgrades to Level C will be coordinated with the **SSHO** or **TO Manager**.

2.2 Health and safety hazards and corresponding precautions include, but are not limited to, the following:

2.2.1 Dermal contact with contaminated soil. Personnel should treat all soil as potentially contaminated and wear chemically impervious gloves. Minimize skin contact with soil by using sampling instruments such as stainless-steel spades or spoons. Do not touch any exposed skin with contaminated gloves.

2.2.2 Inhalation hazards. Appropriate air monitoring should be conducted to ensure that organic vapor concentrations in the breathing zone do not exceed action levels as specified in the APP/SSHP. When ambient temperatures are low enough to require warming samples using the vehicle heater, the vehicle's windows should be opened enough to prevent the build-up of any organic vapors. Use the PID or FID to verify the airborne concentrations in the vehicle remain below applicable action levels. Note that many volatile organic compounds (VOCs) are flammable and all precautions must be observed to eliminate any potential ignition sources.

2.2.3 Shipping limitations. Follow applicable regulations when shipping FID/PID equipment. When shipping an FID by air, the hydrogen tank must be bled dry. Calibration gas canisters are considered dangerous goods and must be shipped according to International Air Transport Association and Department of Transportation regulations. Consult your Safety, Health, and Environment (SH&E) Coordinator and check with your shipping company to determine the correct shipping procedures

3.0 Terms and Definitions

None.

4.0 Interferences

4.1 Regardless of which gas is used for calibration, the instrument will respond to all analytes present in the sample that can be detected by the type of lamp used in the PID.

4.2 Moisture will generate a positive interference in the concentration measured for a PID and is characterized by a slow increase in the reading as the measurement is made. Care must be taken to minimize uptake of moisture to the extent possible. Refer to the manufacturers' instructions for care, cleaning, and maintenance.

4.3 Uptake of soil into the PID must be avoided as it will compromise instrument performance by blocking the probe, causing a positive interference, or dirtying the PID lamp. Refer to the manufacturers' instructions for care, cleaning, and maintenance.

4.4 The user should listen to the pitch of the sampling pump. Any changes in pitch may indicate a blockage and corrective action should be initiated.

5.0 Training and Qualifications

5.1 Qualifications and Training

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 Responsibilities

5.2.1 The **TO Manager** is responsible for ensuring that the collection of headspace readings comply with this procedure. The **TO Manager** is responsible for ensuring that all personnel involved in the collection of headspace readings shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

5.2.3 The **Site Supervisor (SS)** is responsible for ensuring that all headspace readings are conducted according to this procedure as well as verifying that the PID/FID is in proper operating condition prior to use and for implementing the calibration.

5.2.4 All **Field Personnel** are responsible for the implementation of this procedure.

6.0 Equipment and Supplies

6.1 The following materials must be on hand in good operating condition and/or in sufficient quantity to ensure that proper field analysis procedures may be followed:

- Calibrated PID/FID instrument;
- Top-sealing "Zip-Loc" type plastic bags – or – 16 ounces of soil or "mason-" type glass jars and aluminum foil;
- Project field book and/or boring logs;
- Personal Protective Equipment (PPE) as specified in the project APP/SSHP; and
- Safety Data Sheets (SDSs) for any chemicals or site-specific contaminants.

7.0 Procedure

7.1 Preparation

Review available project information to determine the types of organic vapors that will likely be encountered to select the right instrument. The two basic types of instruments are FIDs and PIDs.

FIDs work well with organic compounds that have relatively lightweight molecules, but may have problems detecting halogenated compounds or heavier organic compounds; FIDs can detect methane for example. Since the FID uses a flame to measure organic compounds, ensure that work is conducted

in an atmosphere, which is free of combustible vapors. If ambient temperatures are below 40°F, the flame of the FID may be difficult to light.

When using a PID, select an instrument that can measure the ionization potential of the anticipated contaminants of concern. PIDs work well with a range of organic compounds and can detect some halogenated hydrocarbons; PIDs cannot detect methane. The correct ultraviolet (UV) light bulb must be selected according to the types of organic vapors that will likely be encountered. The energy of the UV light must equal or exceed the ionization potential of the organic molecules that the PID will measure. The NIOSH Pocket Guide to Chemical Hazards is one source for determining ionization potentials for different chemicals. Bulbs available for PIDs include 9.4 eV, 10.6 (or 10.2) eV, and 11.7 eV bulbs. The 10.6 eV bulb is most commonly used as it detects a fairly large range of organic molecules and does not burn out as easily as the 11.7 eV bulb. The 9.4 eV bulb is the most rugged but detects only a limited range of compounds. Under very humid or very cold ambient conditions, the window covering the UV light may fog up, causing inaccurate readings. Ask the **SSHO** about correction factors when high humidity conditions exist.

After selecting the correct instrument, calibrate the PID/FID according to the manufacturer's instructions. Record background/ambient levels of organic vapors measured on the PID/FID after calibration and make sure to subtract the background concentration (if any) from your readings. Check the PID/FID readings against the calibration standard every 20 readings or at any time when readings are suspected to be inaccurate, and recalibrate, if necessary. Be aware that, after measuring highly contaminated soil samples, the PID/FID may give artificially high readings for a time.

7.2 Top-Sealing Plastic Bag

Place a quantity of soil in a top-sealing plastic bag and seal the bag immediately. The volume of soil to be used should be determined by the **TO Manager** or **SS**. The volume of soil may vary between projects but should be consistent for all samples collected for one project. Ideally, the bag should be at least 1/10th-filled with soil and no more than half-filled with soil. Once the bag is sealed, shake the bag to distribute the soil evenly. If the soil is hard or clumpy, use your fingers to gently work the soil (through the bag) to break up the clumps. Do not use a sampling instrument or a rock hammer since this may create small holes in the plastic bag and allow organic vapors to escape. Alternatively, the sample may be broken up before it is placed in the bag. Use a permanent marker to record the following information on the outside of the bag:

- Site identification information (i.e., borehole number);
- Depth interval; and
- Time the sample was collected. For example: "SS-12, 2-4 ft, @1425".

Headspace should be allowed to develop before organic vapors are measured with a PID/FID. The amount of time required for sufficient headspace development will be determined by the project-specific sampling plan and the ambient temperature. Equilibration time should be the same for all samples to allow an accurate comparison of organic vapor levels between samples. However, adjustments to equilibration times may be necessary when there are large variations in ambient temperature from day to day. When ambient temperatures are below 32°F, headspace development should be within a heated building or vehicle. When heating samples, be sure there is adequate ventilation to prevent the build-up or organic vapors above action levels.

Following headspace development, open a small opening in the seal of the plastic bag. Insert the probe of a PID/FID and seal the bag back up around the probe as tightly as possible. Alternatively, the probe can be inserted through the bag to avoid loss of volatiles. Since PIDs and FIDs are sensitive to moisture, avoid touching the probe to the soil or any condensation that has accumulated inside of the bag. Since the PID/FID consumes organic vapors, gently agitate the soil sample during the reading to release fresh organic vapors from the sample. Erratic meter response may occur at high organic vapor concentrations

or conditions of elevated headspace moisture, in which case, headspace data should be discounted. Record the highest reading on the field form or in the field notebook as described in Section 9.

7.3 Jar and Aluminum Foil (Alternate Method)

Half-fill a clean glass jar with the soil sample to be screened. Quickly cover the jar's opening with one to two sheets of clean aluminum foil and apply the screw cap to tightly seal the jar. Allow headspace development for at least ten minutes. Vigorously shake the jar for 15 seconds, both at the beginning and at the end of the headspace development period. Where ambient temperatures are below 32°F (0°C), headspace development should be within a heated area. When heating samples, be sure there is adequate ventilation to prevent the build-up of organic vapors above action levels.

Subsequent to headspace development, remove the jar lid and expose the foil seal. Quickly puncture the foil seal with the instrument sampling probe, to a point about one-half of the headspace depth. Exercise care to avoid uptake of water droplets or soil particulates. As an alternative, use a syringe to withdraw a headspace sample, and then inject the sample into the instrument probe or septum-fitted inlet. This method is acceptable contingent upon verification of methodology accuracy using a test gas standard. Following probe insertion through the foil seal or sample injection to probe, record the highest meter response on the field form or in the field notebook. Using foil seal/probe insertion method, maximum response should occur between two and five seconds. Erratic meter response may occur at high organic vapor concentrations or conditions of elevated headspace moisture, in which case, headspace data should be discounted.

8.0 Quality Control and Assurance

Quality Assurance/Quality Control (QA/QC) will include the collection of duplicate samples. In general, one duplicate will be collected per 20 samples. Organic vapor concentrations measured in the primary and duplicate samples should be similar within plus or minus 20 percent. The frequency of headspace duplicate collection will be determined by the project manager/task manager. The PID/FID instrument must be calibrated according to the manufacturer's instructions before beginning screening and checked or recalibrated every 20 analyses or when readings are suspected to be inaccurate. Record ambient organic vapor levels in the field notebook and on the field form. Periodically check ambient organic vapor levels. If ambient levels have changed more than 20 percent, recalibrate the PID/FID. Make sure readings are not collected near a vehicle exhaust or downwind of a drill rig exhaust. If grossly contaminated soil is encountered, decontaminate sampling instruments between samples and/or change contaminated gloves to avoid cross contaminating less contaminated samples.

9.0 Records, Data Analysis, Calculations

9.1 All data generated (results and duplicate comparisons) will be recorded in the field notebook and/or on the field form. Any deviation from the outlined procedure will also be noted. Field conditions (ambient temperature, wind, etc.) should also be recorded in the field notebook.

9.2 Readings may be recorded in a field notebook, on a boring log, or on an appropriate form specific to the project. The form should include the following information:

- When the PID/FID was calibrated (date/time) and calibration standard used;
- Background/ambient concentrations measured after PID/FID calibration;
- Location of sample (i.e., bore-hole number);
- Depth interval of sample measured;
- Lithology of material measured; and
- PID/FID reading and units of measure.

9.3 Note that if PID/FID measurements are recorded on a boring log, it is not necessary to duplicate information in the column where the PID/FID readings are recorded (e.g., borehole number, depth interval, lithology type).

9.4 All documentation will be stored in the project files and retained following completion of the project.

10.0 Attachments or References

SOP 3-20 Operation and Calibration of a Photoionization Detector

Author	Reviewer	Revisions (Technical or Editorial)
Robert Shoemaker Senior Scientist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (May 2012)
Ken O'Donnell, PG Geologist	Claire Mitchell, PE, PMP Senior Engineer	Rev 1 – PFAS sampling update (July 2019)

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Operation and Calibration of a Photoionization Detector

Procedure 3-20

1.0 Purpose and Scope

1.1 Purpose and Applicability

1.1.1 This standard operating procedure (SOP) describes the procedures that will be followed by field staff for operation and calibration of a photoionization detector (PID). The PID is primarily used by AECOM personnel for safety and survey monitoring of ambient air, determining the presence of volatiles in soil and water, and detecting leakage of volatiles.

1.1.2 PIDs routinely used by field personnel include the Photovac Microtip, Thermoelectron 580EZ, MiniRAE 2000, and MiniRae 3000. Personnel responsible for using the PID should first read and thoroughly familiarize themselves with the instrument instruction manual.

1.2 Principle of Operation

1.2.1 The PID is a non-specific vapor/gas detector. The unit generally consists of a hand-held probe that houses a PID, consisting of an ultraviolet (UV) lamp, two electrodes, and a small fan which pulls ambient air into the probe inlet tube. The probe is connected to a readout/control box that consists of electronic control circuits, a readout display, and the system battery. Units are available with UV lamps having an energy from 9.5 electron volts (eV) to 11.7 eV.

1.2.2 The PID analyzer measures the concentration of trace gas present in the atmosphere by photoionization. Photoionization occurs when an atom or molecule absorbs a photon of sufficient energy to release an electron and become a positive ion. This will occur when the ionization potential of the molecule (in electron volts (eV) is less than the energy of the photon. The source of photons is an ultraviolet lamp in the probe unit. Lamps are available with energies ranging from 9.5 eV to 11.7 eV. All organic and inorganic vapor/gas compounds having ionization potentials lower than the energy output of the UV lamp are ionized and the resulting potentiometric change is seen as a positive reading on the unit. The reading is proportional to the concentration of organics and/or inorganics in the vapor.

1.2.3 Sample gases enter the probe through the inlet tube and enter the ion chamber where they are exposed to the photons emanating from the UV lamp. Ionization occurs for those molecules having ionization potentials near to or less than that of the lamp. A positive- biased polarizing electrode causes these positive ions to travel to a collector electrode in the chamber. Thus the ions create an electrical current which is amplified and displayed on the meter. This current is proportional to the concentration of trace gas present in the ion chamber and to the sensitivity of that gas to photoionization.

1.2.4 In service, the analyzer is first calibrated with a gas of known composition equal to, close to, or representative of that to be measured. Gases with ionization potentials near to or less than the energy of the lamp will be ionized. These gases will thus be detected and measured by the analyzer. Gases with ionization potentials greater than the energy of the lamp will not be detected. The ionization potentials of the major components of air, i.e., oxygen, nitrogen, and carbon dioxide, range from about 12.0 eV to 15.6 eV and are not ionized by any of the lamps available. Gases with ionization potentials near to or slightly higher than the lamp are partially ionized, with low sensitivity.

1.3 Specifications

1.3.1 Refer to the manufacturer's instructions for the technical specifications of the instrument being used. The operating concentration range is typically 0.1 to 2,000 ppm isobutylene equivalent.

2.0 Safety

2.1 The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the project Accident Prevention Plan (APP)/Site Safety and Health Plan (SSHP). Work will also be conducted according to the Task Order (TO) Quality Assurance Project Plan (QAPP) and/or direction from the **Site Safety and Health Officer (SSHO)**.

2.2 Only PIDs stamped Division I Class I may be used in explosive atmospheres. Refer to the project APP/SSHP for instructions pertaining to instrument use in explosive atmospheres.

3.0 Terms and Definitions

None.

4.0 Interferences

4.1 Regardless of which gas is used for calibration, the instrument will respond to all analytes present in the sample that can be detected by the type of lamp used in the PID.

4.2 Moisture will generate a positive interference in the concentration measured for a PID and is characterized by a slow increase in the reading as the measurement is made. Care must be taken to minimize uptake of moisture to the extent possible. Refer to the manufacturers' instructions for care, cleaning, and maintenance.

4.3 Uptake of soil into the PID must be avoided as it will compromise instrument performance by blocking the probe, causing a positive interference, or dirtying the PID lamp. Refer to the manufacturers' instructions for care, cleaning, and maintenance.

4.4 The user should listen to the pitch of the sampling pump. Any changes in pitch may indicate a blockage and corrective action should be initiated.

5.0 Training and Qualifications

5.1 Qualifications and Training

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 Responsibilities

5.2.1 The **TO Manager** is responsible for ensuring that the operation and calibration activities comply with this procedure. The **TO Manager** is responsible for ensuring that all personnel involved in the operation and calibration shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

5.2.3 The **Site Supervisor (SS)** is responsible for ensuring that all operation and calibration activities are conducted according to this procedure.

5.2.4 All **Field Personnel** are responsible for the implementation of this procedure.

6.0 Equipment and Supplies

- Calibration Gas: Compressed gas cylinder of isobutylene in air or similar stable gas mixture of known concentration. The selected gas should have an ionization potential similar to that of the vapors to be monitored, if known. The concentration should be at 50-75% of the range in which the instrument is to be calibrated;
- Regulator for calibration gas cylinder;

- Approximately 6 inches of Teflon® tubing;
- Tedlar bag (optional);
- Commercially-supplied zero grade air (optional);
- "Magic Marker" or “Sharpie” or other waterproof marker;
- Battery charger;
- Moisture traps;
- Spare lamps;
- Manufacturer’s instructions; and
- Field data sheets or logbook/pen.

7.0 Procedure

7.1 Preliminary Steps

7.1.1 Preliminary steps (battery charging, check-out, calibration, maintenance) should be conducted in a controlled or non-hazardous environment.

7.2 Calibration

7.2.1 The PID must be calibrated in order to display concentrations in units equivalent to ppm. First a supply of zero air (ambient air or from a supplied source), containing no ionizable gases or vapors is used to set the zero point. A span gas, containing a known concentration of a photoionizable gas or vapor, is then used to set the sensitivity.

7.2.2 Calibrate the instrument according to the manufacturer’s instructions. Record the instrument model and identification number, the initial and adjusted meter readings, the calibration gas composition and concentration, and the date and the time in the field records.

7.2.3 If the calibration cannot be achieved or if the span setting resulting from calibration is 0.0, then the lamp must be cleaned (Section 7.4).

7.3 Operation

7.3.1 Turn on the unit and allow it to warm up (minimum of 5 minutes). Check to see if the intake fan is functioning; if so, the probe will vibrate slightly and a distinct sound will be audible when holding the probe casing next to the ear. Also, verify on the readout display that the UV lamp is lit.

7.3.2 Calibrate the instrument as described in Section 7.2, following the manufacturer’s instructions. Record the calibration information in the field records.

7.3.3 The instrument is now operational. Readings should be recorded in the field records.

7.3.4 When the PID is not being used or between monitoring intervals, the unit may be switched off to conserve battery power and UV lamp life; however, a “bump” test should be performed each time the unit is turned on and prior to taking additional measurements. To perform a bump test, connect the outlet tubing from a Tedlar bag containing a small amount of span gas to the inlet tubing on the unit and record the reading. If the reading is not within the tolerance specified in the project plan, the unit must be recalibrated.

7.3.5 At the end of each day, recheck the calibration. The check will follow the same procedures as the initial calibration (Section 7.2) except that no adjustment will be made to the instrument. Record the information in the field records.

7.3.6 Recharge the battery after each use (Section 7.4).

7.3.7 When transporting, ensure that the instrument is packed in its stored condition in order to prevent damage.

7.4 Routine Maintenance

7.4.1 Routine maintenance associated with the use of the PID includes charging the battery, cleaning the lamp window, replacing the detector UV lamp, replacing the inlet filter, and replacing the sample pump. Refer to the manufacturer's instructions for procedures and frequency.

7.4.2 All routine maintenance should be performed in a non-hazardous environment.

7.5 Troubleshooting Tips

7.5.1 One convenient method for periodically confirming instrument response is to hold the sensor probe next to the tip of a magic marker. A significant reading should readily be observed.

7.5.2 Air currents or drafts in the vicinity of the probe tip may cause fluctuations in readings.

7.5.3 A fogged or dirty lamp, due to operation in a humid or dusty environment, may cause erratic or fluctuating readings. The PID should never be operated without the moisture trap in place.

7.5.4 Moving the instrument from a cool or air-conditioned area to a warmer area may cause moisture to condense on the UV lamp and produce unstable readings.

7.5.5 A zero reading on the meter should not necessarily be interpreted as an absence of air contaminants. The detection capabilities of the PID are limited to those compounds that will be ionized by the particular probe used.

7.5.6 Many volatile compounds have a low odor threshold. A lack of meter response in the presence of odors does not necessarily indicate instrument failure.

7.5.7 When high vapor concentrations enter the ionization chamber in the PID the unit can become saturated or "flooded". Remove the unit to a fresh air environment to allow the vapors to be completely ionized and purged from the unit.

8.0 Quality Control and Assurance

8.1 The end use of the data will determine the quality assurance requirements that are necessary to produce data of acceptable quality. These quality assurance requirements will be defined in the site-specific QAPP.

8.2 Calibration of the PID will be conducted at the frequency specified in the project plan. In the absence of project-specific guidance, calibration will be performed at the beginning of each day of sampling and will be checked at the end of the sampling day or whenever instrument operation is suspect. The PID will sample a calibration gas of known concentration. The instrument must agree with the calibration gas within ±10%. If the instrument responds outside this tolerance, it must be recalibrated.

8.3 Checks of the instrument response (Section 7.5) should be conducted periodically and documented in the field records.

9.0 Records, Data Analysis, Calculations

Safety and survey monitoring with the PID will be documented in a bound field logbook, or on standardized forms, and retained in the project files. The following information is to be recorded:

- Project name and number;
- Instrument manufacturer, model, and identification number;
- Operator's signature;

- Date and time of operation;
- Calibration gas used;
- Calibration check at beginning and end of day (meter readings before adjustment);
- Span setting after calibration adjustment;
- Meter readings (monitoring data obtained);
- Instances of erratic or questionable meter readings and corrective actions taken; and
- Instrument checks and response verifications – e.g., battery check, magic marker response (Section 7.5) or similar test.

10.0 Attachments or References

United States Environmental Protection Agency. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual (EISOPQAM). USEPA, Region 4, SESD, Enforcement and Investigations Branch, Athens, GA. November 2001.

Author	Reviewer	Revisions (Technical or Editorial)
Robert Shoemaker Senior Scientist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (May 2012)
Ken O'Donnell, PG Geologist	Claire Mitchell, PE, PMP Senior Engineer	Rev 1 – PFAS sampling update (July 2019)

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Surface and Subsurface Soil Sampling Procedures

Procedure 3-21

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes the procedures for soil sampling. The procedure includes surface and subsurface sampling by various methods using hand auguring, test pit, direct-push, sonic drilling, and split-spoon equipment.

1.2 For project specific information (e.g., sampling depths, equipment to be used, and frequency of sampling), refer to the Quality Assurance Project Plan (QAPP), which takes precedence over these procedures. Surface soil sampling, typically considered to be up to two feet below ground surface by United States Environmental Protection Agency (USEPA) standards, is typically accomplished using hand tools such as shovels or hand augers. Test pit samples are considered subsurface samples, although normally collected via hand tools similar to surface soil sampling or by excavation machinery. Direct-push and split-spoon sampling offer the benefit of collecting soil samples from a discrete or isolated subsurface interval without the need of extracting excess material above the target depth. These methods dramatically reduce time and cost associated with disposal of material from soil cuttings when compared to test pit sampling. In addition, direct-push, sonic drilling, and split-spoon sampling methods can obtain samples at targeted intervals greater than 15 feet in depth, allowing for discrete depth soil sampling while speeding up the sampling process. Direct-push methods work best in medium to fine-grained cohesive materials, such as medium to fine sands, silts, and silty clay soils. Sonic drilling sampling works well in all types of soil and bedrock. Split-spoon sampling works well in all types of soil but is somewhat slower than direct-push and sonic drilling methods. With the exception of volatile organic compounds (VOCs) samples, the soil sample interval is composited so that each sample contains a homogenized representative portion of the sample interval. Due to potential loss of analytes, samples for VOC analysis are not composited. Samples for chemical analysis can be collected by any of the above-mentioned sampling methods, as disturbed soil samples. Undisturbed samples are best collected with direct push or by Shelby Tube (not covered in this SOP). They are collected, sealed, and sent directly to the laboratory for analysis without homogenizing.

2.0 Safety

2.1 The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the project Accident Prevention Plan (APP)/Site Safety and Health Plan. Work will also be conducted according to the Task Order (TO) QAPP and/or direction from the **Site Safety and Health Officer (SSHO)**.

2.2 Before soil sampling commences, appropriate entities (e.g. DigSafe, local public works departments, company facilities) must be contacted to assure the anticipated soil sampling locations are marked for utilities, including electrical, telecommunications, water, sewer, and gas.

3.0 Terms and Definitions

None.

4.0 Interferences

4.1 Low recovery of soil from sampling equipment will prevent an adequate representation of the soil profile and sufficient amount of soil sample. If low recovery is a problem, the hole may be offset and re-advanced, terminated, or continued using a larger diameter sampler.

4.2 Asphalt in soil samples can cause false positive results for hydrocarbons. To ensure samples are free of asphalt, do not collect samples that may contain asphalt. If the collection of samples potentially containing asphalt is unavoidable, note the sampling depths at which the presence of asphalt are suspected.

4.3 Instrumentation interferences addressed in SOPs for Calibration of the Photoionization Detector (PID), Headspace Screening for Total Volatile Organics, and Equipment Decontamination must also be considered.

4.4 Cross contamination from sampling equipment must be prevented by using sampling equipment constructed of stainless steel that is adequately decontaminated between samples.

5.0 Training and Qualifications

5.1 Qualifications and Training

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 Responsibilities

5.2.1 The **TO Manager** is responsible for ensuring that soil sampling activities comply with this procedure. The TO Manager is responsible for ensuring that all personnel involved in soil sampling shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

5.2.3 The **Site Supervisor (SS)** is responsible for ensuring that all soil sampling activities are conducted according to this procedure.

5.2.4 All **Field Personnel** are responsible for the implementation of this procedure.

6.0 Equipment and Supplies

The depth at which samples will be collected and the anticipated method of sample collection (direct-push, split-spoon, hand auger, shovel, or test pits) will be presented in the QAPP. The following details equipment typically needed for soil sampling, based on the various methods. See the QAPP for specific detail of equipment and supply needs.

6.1 Depending on the nature of suspected contamination, field screening instrumentation may be used for direct sampling. Appropriate instrumentation and calibration standards should be available. If volatile organic contaminants are suspected and a PID will be used, refer to the equipment and instrumentation listed in SOP 3-20 Operation and Calibration of a Photoionization Detector. Equipment in this SOP includes but is not limited to:

- PID/FID;
- Calibration gas; and
- Tedlar® gas bags (for calibration).

6.2 If field screening methods include jar headspace screening for volatile organics, refer to the equipment and procedure in SOP 3-19 Headspace Screening for Total VOCs. Equipment in this SOP includes but is not limited to:

- Clean soil (“drillers jars”) jars;
- HDPE sample jars for PFAS/PFOS samples; and
- Aluminum foil for non-PFAS/PFOS sampling.

6.3 Appropriate decontamination procedures must be followed for sampling equipment. Refer to SOP 3-06 Equipment Decontamination. Equipment in this SOP includes but is not limited to:

- Alconox® or Liquinox®;
- Isopropyl Alcohol;
- Deionized Ultra-Filtered (DIUF) Water (confirmed PFAS-free);
- Plastic buckets or washbasins;
- Brushes; and
- Polyethylene sheeting.

6.4 The following general equipment is needed for all soil sampling, regardless of method:

- Stainless steel bowls;
- Stainless steel trowels;
- Appropriate sample containers for laboratory analysis;
- Personal Protective Equipment (PPE);
- Non-water-repellent logbook;
- Cooler and ice for preservation; and
- Stakes and flagging to document sampling location.

6.5 The following additional equipment is needed for volatile organic sampling:

- Electronic pan scale and weights for calibration; and
- Syringes or other discrete soil core samplers.

6.6 The following additional equipment may be needed for surface and test pit soil sampling:

- Hand Auger

6.7 The following additional equipment may be needed for soil sampling from direct push and/or split-spoon equipment:

- Tape measure or folding carpenter's rule for recording the length of soil recovered.

Note: All subsurface drilling equipment will be provided and maintained by the subcontractor.

7.0 Procedure

7.1 General Soil Sampling Procedure for All Soil Sampling Methods

7.1.1 Record the weather conditions and other relevant on-site conditions.

7.1.2 Select the soil sampling location, clear vegetation, if necessary, and record the sampling location identification number and pertinent location details.

7.1.3 Verify that the sampling equipment is properly decontaminated, in working order, and situated at the intended sampling location.

7.1.4 Place polyethylene sheeting on the ground and assemble all necessary sampling equipment on top of it. Cover surfaces onto which soils or sampling equipment will be placed (i.e., tables with polyethylene sheeting).

7.1.5 Follow the appropriate procedures listed below for either surface, split-spoon, sonic drilling, direct push, or test pit sample collection (7.2, 7.3, 7.4, 7.5, and 7.6, respectively).

7.1.6 Collect soil samples according to procedures listed in Section 7.7 depending on project specific analyses.

7.1.7 Record date/time, sample ID, and sample descriptions in the field logbook or field form. A sketch or description of the location may also be recorded so the sample location can be re-constructed, especially if the location will not be recorded using global positioning satellite (GPS) equipment.

7.1.8 Immediately label the sample containers and place them on ice, if required for preservation. Complete the chain-of-custody form(s) as soon as possible.

7.1.9 Dispose of all excess excavated soil in accordance with the QAPP.

7.1.10 If required, mark the sample location with a clearly labelled wooden stake or pin flag. If the location is on a paved surface, the location may be marked with spray paint.

7.1.11 Decontaminate the sampling equipment according to SOP 3-06 Equipment Decontamination.

7.2 Surface Sampling

7.2.1 The criteria used for selecting surface soil locations for sampling may include the following:

- Visual observations (soil staining, fill materials);
- Other relevant soil characteristics;
- Site features;
- Screening results;
- Predetermined sampling approach (i.e., grid or random); and
- Sampling objectives as provided in the QAPP.

7.2.2 The following procedures are to be used to collect surface soil samples. Surface soils are considered to be soils that are up to two feet below ground surface, though state regulations and project objectives may define surface soils differently; therefore, the QAPP should be consulted for direction on the depth from which to collect the surface soil samples. Sampling and other pertinent data and information will be recorded in the field logbook and/or on field forms. Photographs may be taken as needed or as specified in the QAPP.

1. Gently scrape any vegetative covering until soil is exposed. Completely remove any pavement.
2. Remove soil from the exposed sampling area with a stainless-steel trowel, hand auger, or shovel. Put soils within the sampling interval in a stainless-steel bowl for homogenizing. Monitor the breathing zone and sampling area as required in the APP/SSHP.
3. For VOC analyses, collect representative soil samples directly from the recently-exposed soil using a syringe or other soil coring device (e.g., TerraCore®, EnCore®). Follow procedures in Section 7.7.1 for VOC sampling.
4. Collect sufficient soil to fill all remaining sample jars into a stainless-steel bowl. Homogenize the soil samples to obtain a uniform soil composition which is representative of the total soil sample collected according to the following procedure:

    a) Remove all rocks and non-soil objects using a stainless-steel spoon or scoop.

b) Form a cone shaped mound with the sample material, then flatten the cone and split the sample into quarters.

c) Use the stainless-steel spoon/scoop to mix the quarter samples that are opposite.

d) After mixing the opposite quarters, reform the cone shaped mound.

e) Repeat this procedure a minimum of five (5) times, removing any non-soil objects and breaking apart any clumps.

7.3 Split-Spoon Sampling

7.3.1 At each boring location, the frequency and depth of split-spoon samples will be determined from the QAPP. Split-spoon samples may be collected continuously, intermittently, or from predetermined depths.

7.3.2 Split-spoon samplers shall be driven into undisturbed soil by driving the spoon ahead of the drill augers/casing. In cohesive soils, or soils where the borehole remains open (does not collapse), two split-spoon samples may be taken prior to advancing the augers/casing.

7.3.3 After split-spoons are retrieved, open the split-spoon and measure the recovery of soil. If a PID will be used for screening, immediately scan the recovered sample for VOCs using the PID. Scan the recovered soil boring by making a hole in the soil with a decontaminated trowel and placing the PID inlet very close to the hole. Be very careful not to get soil on the tip of the PID. Take PID readings every 6 inches along the split-spoon and/or in any areas of stained or disturbed soil. Record the highest PID reading and the depth at which it was observed along with all other pertinent observations. If required in the QAPP, VOC and headspace samples should be collected (see Section 7.7.1) prior to logging the sample.

7.3.4 If headspace screening for VOCs is required in the QAPP, collect a soil sample (as defined in the QAPP) and perform headspace screening according to SOP 3-19 Headspace Screening for Total VOCs.

7.3.5 Soils collected using the split-spoon sampler will be logged by the field representative using the procedure required in the QAPP.

7.3.6 Collect the remainder of the sample volume required into a stainless-steel bowl. Homogenize the soil so the material is uniform in composition and representative of the total soil sample collected. Follow homogenizing techniques as described in Section 7.2.

7.3.7 The QAPP may specify that intervals to be sent to the laboratory be determined by visual observation and/or highest PID screening or headspace results, which can only be determined once the boring is complete. In this instance, a VOC sample should be collected at each interval. The remainder of the soil from that interval will be set aside in a clearly labelled stainless steel bowl covered with polyethylene sheeting. Once the boring has been completed and the sample interval has been determined, the remainder of the soil can be homogenized according to Section 7.2 and submitted for laboratory analysis.

7.3.8 Once a boring is complete and all required samples have been collected, the boring must be completed as specified in the QAPP (e.g., completed as a monitoring well, backfilled with bentonite, etc.).

7.4 Sonic Drilling Sampling

7.4.1 At each boring location, the frequency and depth of sonic drilling samples will be determined from the QAPP.

7.4.2 Sonic drilling methods, also known as vibratory drilling, use an eccentrically oscillating drill head to produce high-frequency vibratory energy that is then transmitted down a drill string to a core barrel to quickly advance through the subsurface. Sonic drilling utilizes a double-cased system using an inner core barrel and a larger override casing. This ensures that the borehole is continuously cased to the total depth, minimizing the potential for borehole collapse and providing the means to alter casing diameters to telescope through semi-confining units to prevent downhole cross contamination.

7.4.3 Upon retrieval of the core barrel, place the tubular plastic sleeve (confirmed PFAS-free) with sealed bottom over the bottom of the core barrel. The core barrel will then be vibrated, causing the soil sample to be extruded into the sleeve. Place the sleeve on the work surface (i.e. PFAS-free plastic covered table or ground). Open the sleeve and measure the recovery of soil.

7.4.4 If a PID will be used for screening, immediately scan the recovered sample for VOCs using the PID. Scan the recovered soil boring by making a hole in the soil with a decontaminated trowel and placing the PID inlet very close to the hole. Be very careful not to get soil on the tip of the PID. Take PID readings every 6 inches along the soil core and/or in any areas of stained or disturbed soil. Record the highest PID reading and the depth at which it was observed along with all other pertinent observations. If required in the QAPP, VOC and headspace samples should be collected (see Section 7.7.1) prior to logging the sample.

7.4.5 If headspace screening for VOCs is required in the QAPP, collect a soil sample (as defined in the QAPP) and perform headspace screening according to SOP 3-19 Headspace Screening for Total VOCs.

7.4.6 Soils collected using sonic drilling will be logged by the field representative using the procedure required in the QAPP.

7.4.7 Collect the remainder of the sample volume required into a stainless-steel bowl. Homogenize the soil so the material is uniform in composition and representative of the total soil sample collected. Follow homogenizing techniques as described in Section 7.2.

7.4.8 The QAPP may specify that intervals to be sent to the laboratory be determined by visual observation and/or highest PID screening or headspace results, which can only be determined once the boring is complete. In this instance, a VOC sample should be collected at each interval. The remainder of the soil from each interval will be set aside. Once the boring has been completed and the sample interval has been determined, the remainder of the soil can be homogenized according to Section 7.2 and submitted for laboratory analysis.

7.4.9 Once a boring is complete and all required samples have been collected, the boring must be completed as specified in the QAPP (e.g., completed as a monitoring well, backfilled with bentonite, etc.).

7.5 Direct Push Sampling

At each boring location, the frequency of direct-push samples will be determined from the QAPP. Typically, samples with direct-push equipment are collected in 4-foot (ft) intervals, but smaller (e.g., 2 ft) and larger (e.g., 5 ft) intervals are also possible.

1. Sample using Macro-Core samplers with acetate liners to obtain discrete soil samples at the depths specified in the QAPP.
2. Cut open the acetate liner. If required in the QAPP, immediately scan the recovered soil boring for VOCs using a PID by making a hole in the soil with a decontaminated trowel and placing the PID inlet very close to the hole. Be very careful not to get soil on the tip of the PID. Take PID readings every 6 inches along the split-spoon and/or in any areas of stained or disturbed soil. Record the highest PID reading and the depth at which it was observed along with all other pertinent observations. VOC and headspace samples, if required in the QAPP should be collected (see Section 7.7.1) prior to logging the sample.
3. If required in the QAPP, collect a soil sample (as defined in the QAPP) and perform headspace screening according to SOP 3-19 Headspace Screening for Total VOCs.
4. Soils collected using the direct-push sampler will be logged by the by the field representative using the procedure required in the QAPP.
5. Collect the remainder of the sample into a stainless-steel bowl. Homogenize the soil collected so that the material is uniform in composition and representative of the total soil sample collected. Follow homogenizing techniques as described in Section 7.2.

6. Once a boring is complete and all required samples have been collected, the boring must be completed as specified in the QAPP (e.g., completed as a monitoring well, backfilled with bentonite, etc.).

7.6 Test Pit Sampling

7.6.1 Excavate the test pit to the desired depth.

7.6.2 Using the excavator bucket, collect soil samples as specified in the QAPP. Collect a sample and perform screening analyses as required by the QAPP. If VOCs contamination is suspected, perform headspace screening according to SOP 3-19 Headspace Screening for Total VOCs.

7.6.3 Collect the sample from center of the bucket to avoid potential contamination from the bucket.

7.6.4 VOC samples should also be collected from an undisturbed section soil in the excavator bucket. The top layer of exposed soil should be scraped away just prior to collecting the VOC samples.

7.6.5 Collect the remainder of the sample volume required into a stainless-steel bowl. Homogenize the soil so the material is uniform in composition and representative of the total soil sample collected. Follow homogenizing techniques as described in Section 7.2.

7.6.6 Dispose of all excavated soil according to the QAPP.

7.7 Sample Collection Methods

7.7.1 Volatile Organics Sampling

For soils collected for analyses of volatile organics, including Volatile Petroleum Hydrocarbons (VPH) or other purgeable compounds, a closed system is maintained. From collection through analysis, the sample bottles are not opened. The bottle kit for a routine field sample for these analyses will typically include three 40-mL VOA vials and one soil jar. Two 40-mL VOA vials will contain either 5 mL reagent water or 5 mL sodium bisulfate and magnetic stir bars (i.e., low level vials). The third VOA vial will contain 15 mL methanol with no magnetic stir bar (i.e., high level vial). These vials are usually provided by the laboratory and are pre-weighed, with the tare weight recorded on the affixed sample label. No additional sample labels are affixed to the VOA vials, as addition of a label would alter the vial weight. All information is recorded directly on the sample label using an indelible marker. The soil jar is provided for percent solids determination. For VOC or VPH analyses, samples are collected prior to sample homogenization. Collect the VOC sample in accordance with the procedure described below.

1. Determine the soil volume necessary for the required sample weight, typically 5 grams:
    a) Prepare a 5 mL sampling corer (e.g., Terra Core®) or cut-off plastic syringe.
    b) Tare the sampler by placing it on the scale and zeroing the scale.
    c) Draw back the plunger to the 5-gram mark or 5mL (5cc) mark on cut-off syringe and insert the open end of the sampler into an undisturbed area of soil with a twisting motion, filling the sampler with soil. Note the location of the plunger with respect to the milliliter (cc) or other graduation printed on the sampler.
    d) Weigh the filled sampler and remove or add soil until the desired weight is obtained. Note the location of the plunger which corresponds to this weight. Do not use this sample for laboratory analysis.
2. Once the required soil volume has been determined, pull the plunger back to this mark and hold it there while filling the syringe for each sample.
3. Collect 5 grams of soil using the cut-off syringe or Terra Core® sample device. Extrude the 5-grams of soil into one of the low level 40-mL VOA vials. Quickly wipe any soil from the threads of the VOA vial with a clean Kimwipe® and immediately close the vial. It is imperative that the

threads be free from soil or other debris prior to replacing the cap on the vial in order to maintain the closed system necessary for the analysis.

4. Gently swirl the vial so that all of the soil is fully wetted with the preservative.
5. Fill the other low level 40 mL VOA vial in this manner.
6. Repeat the process for the high-level VOA vials, only for the high-level VOA vial three 5-gram aliquots (i.e., 15 grams total) should be extruded into the high-level VOA vial.

   NOTE: Depending on the laboratory, some high-level VOA vials only contain 5 mL or 10 mL of methanol. If this is the case, either 5 grams total or 10 grams total, respectively, should be extruded into the high-level VOA vial. In other words, the mass of soil in grams should be identical to the volume of methanol in mL (i.e., 1:1 ratio of soil to methanol).

7. Collect any additional QC sample collected (e.g., field duplicate, MS, and MSD) in the same manner as above.
8. Fill the 4-oz glass jar with soil from the same area for percent moisture determination.

7.7.2 Soil Sampling Method (All other analyses except VOC/VPH)

When all the required soil for a sampling location has been obtained, the soil can be homogenized as described in section 7.2. Collect sufficient volume to fill all of the remaining sample containers at least ¾ full for all other analyses. Homogenize the soil in a decontaminated stainless-steel bowl, removing rocks, sticks, or other non-soil objects and breaking apart any lumps of soil prior to filling the remaining sample containers.

NOTE: Soil samples must contain greater than 30% solids for the data to be considered valid.

8.0 Quality Control and Assurance

8.1 Sampling personnel should follow specific quality assurance guidelines as outlined in the QAPP. Proper quality assurance requirements should be provided which will allow for collection of representative samples from representative sampling points. Quality assurance requirements outlined in the QAPP typically suggest the collection of a sufficient quantity of field duplicate, field blank, and other samples.

8.2 Quality control requirements are dependent on project-specific sampling objectives. The QAPP will provide requirements for equipment decontamination (frequency and materials), sample preservation and holding times, sample container types, sample packaging and shipment, as well as requirements for the collection of various quality assurance samples such as trip blanks, field blanks, equipment blanks, and field duplicate samples.

9.0 Records, Data Analysis, Calculations

All data and information (e.g., sample collection method used) must be documented on field data sheets, boring logs, or within site logbooks with permanent ink. Data recorded may include the following:

- Weather conditions;
- Arrival and departure time of persons on site;
- Instrument type, lamp (PID), make, model and serial number;
- Calibration gas used;
- Date, time and results of instrument calibration and calibration checks;
- Sampling date and time;
- Sampling location;
- Samples collected;
- Sampling depth and soil type;
- Deviations from the procedure as written; and
- Readings obtained.

10.0 Attachments or References

SOP 3-06, *Equipment Decontamination*

SOP 3-19, *Headspace Screening for Total VOCs*

SOP 3-20, *Operation and Calibration of a Photoionization Detector*

Author	***Reviewer***	***Revisions (Technical or Editorial)***
Robert Shoemaker, PMP Senior Scientist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (May 2012)
Ken O'Donnell, PG Geologist	Claire Mitchell, PE, PMP Senior Engineer	Rev 1 – PFAS sampling update (July 2019)
Robert Shoemaker, PMP Senior Scientist	Josh Millard, PG, CPG	Rev 2 – Addition of Sonic Drilling Methods (January 2020)

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Grab Groundwater Sampling Techniques

Procedure 3-37

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) defines the procedures for collecting grab-groundwater samples from temporary well points installed using direct push or other drilling.

1.2 This procedure is the Program-approved professional guidance for work performed by AECOM under the client contract.

1.3 As guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved by both the **Task Order (TO) Manager** and the **Program Quality Manager** and documented.

1.4 If there are procedures, whether they be from AECOM, state, and/or federal that are not addressed in this SOP and are applicable to direct push sampling, then those procedures may be added as an appendix to the project-specific Quality Assurance Project Plan (QAPP).

2.0 Safety

2.1 **Field Personnel** shall perform work in accordance with the Accident Prevention Plan (APP) and Site Safety and Health Plan (SSHP). During grab groundwater collection, subcontractors in direct contact with potentially contaminated media shall wear the proper personal protective equipment (PPE) as outlined in the APP/SSHP. Failure to comply will result in disciplinary action.

3.0 Terms and Definitions

3.1 Grab groundwater collection techniques are designed to collect screening-level groundwater data in an efficient manner such that informed field decisions can be made when delineating contaminant plumes, inferring source areas, and identifying other potential soil sample locations and/or locations for permanent monitoring well installation.

4.0 Interferences

4.1 Contaminants that are known to adsorb to particulates, such as metals, polychlorinated biphenyls (PCBs), etc., will be impacted by elevated turbidity (i.e., >25 Nephelometric Turbidity Units [NTU]). For grab groundwater samples with turbidity above 25 NTU, AECOM may collect filtered samples using a 0.45-micron field filter as well as unfiltered samples, providing that the use of a filter does not potentially compromise sample quality.

4.2 Gas bubbles present in discharge tubing during purging and sampling are a problem; their presence indicates off-gassing from groundwater or poor purging connections in the airline or groundwater tubing. Sunlight can exacerbate this problem when low pumping rates are used. Check connections at the surface, and if bubbles persist, check connections at the pump. During purging and sampling, observe the flow of groundwater in the sample tubing and keep the tubing filled with groundwater, removing all air pockets and bubbles, to the extent possible. Gas bubbles may be reduced by increasing flow, if possible, and keeping tubing shaded.

4.3 Pump tubing lengths above the top of well casing should be kept as short as possible to minimize heating the groundwater in the tubing by exposure to sun light and ambient air temperatures. Heating

may cause the groundwater to degas, which is unacceptable for the collection of samples for volatile organic compounds (VOCs) and dissolved gases analyses.

5.0 Training and Qualifications

5.1 Qualifications and Training

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 Responsibilities

5.2.1 The **TO Manager** is responsible for ensuring that these standard grab groundwater collection procedures are followed during projects conducted under the Program and that a qualified individual conducts or supervises the projects.

5.2.2 The **Program Quality Manage**r is responsible for ensuring overall compliance with this procedure.

5.2.3 The **Site Supervisor (SS)** is responsible for ensuring that all **Field Personnel** follow these procedures.

5.2.4 All **Field Personnel** are responsible for the implementation of this procedure.

5.2.5 The **Field Personnel** and/or **SS** are responsible for directly supervising the grab groundwater collection procedures to ensure that they are conducted according to this procedure, and for recording all pertinent data collected during sampling.

6.0 Equipment and Supplies

6.1 Bladder Pump

The bladder pump system contains the following components: a pressurized cylinder of inert gas (typically nitrogen), a pump controller, air intake and discharge lines, and bladder pumps. The controller regulates total flow of nitrogen from the pressurized nitrogen cylinder to the pump assembly located in the well. AECOM typically samples one well per nitrogen cylinder. Note that if the bladder pumps are placed at the same depth in each well, multiple wells may be sampled simultaneously with one nitrogen cylinder or air compressor. In this case, a three-way cross splitter with quick-connect air line fittings is attached to the tubing connected to the nitrogen cylinder. Up to three controllers can then be connected to the nitrogen cylinder. If nitrogen cylinders are not available, air compressors may be used to power the bladder pumps.

The tubing bundle connected to the pump has three components: an air line with fittings to the pump and the controller, a sample line, and a support cable. For pumps that use nipple tubing connectors, the support cable may not be necessary. The sample line, through which purge water is removed, must to be composed entirely of high-density polyethylene (HDPE) if samples for VOCs or PFAS are to be collected, depending on the project data quality objectives.

Temporary well points installed using direct push or other drilling methods are typically 1” or 0.75” in diameter. The diameter of the bladder pump should be sufficiently small (e.g., 0.850”, 0.675”, etc.) to allow for the easy deployment of the pump, associated tubing, and water level indicator.

6.2 Peristaltic Pump

Peristaltic pumps are not submerged in the well but remain outside of the well and function by pulling water to the surface. A peristaltic pump has a rotating pump head with stepless variable speed that compresses a short stretch of flexible Pharmaceutical-grade (e.g., Pharmed) silicone tubing to pull water up from the well using mechanical peristalsis. The sample water does not come into direct contact with the pump. HDPE tubing is connected to either end of the silicone tubing. The pumps typically used, the GeoTech GeoPump or GeoPump II, operate off an external 12-volt (V) battery or 120 V alternating current (AC) power source. Commercially available “JumpStart” 12 V batteries are typically preferred

since electrical hookup is typically not available; since they are safe, easy to carry, and easy to recharge; and since the potential contamination issues associated with use of a generator are avoided. Peristaltic pumps cannot be used when the depth to water is greater than 27 feet.

6.3 Tubing

HDPE tubing is preferred for all parameters. Pharmaceutical-grade (e.g., Pharmed) silicon tubing may be required to be used around the rotor head of the peristaltic pump and, if necessary, as a connecting tubing to the flow-through cells. Inner tubing diameter should be kept to the smallest size possible to reduce the generation of air pockets during low flow. Tubing typically used with the peristaltic pumps is HDPE of 1/4-inch outside diameter and 3/16-inch inner diameter.

6.4 Electronic water level indicator: Solinst Model 101 or similar

Inner casing diameter and pump diameter should be considered when selecting a water level indicator that will fit into the well with the pump. A smaller diameter probe will be required for temporary well points. Electronic water level indicators will be confirmed with the vendor to be Teflon-free.

6.5 Flow controllers and compressed inert gases for submersible bladder pumps

QED Model MP-10 flow controller and nitrogen gas are typically used unless nitrogen is an analyte of interest. Portable air compressors may be used in place of compressed gas (e.g., QED Well Wizard).

6.6 Power Source

Marine battery, battery pack, compressed gas, portable air compressor, and a flow-controller are typically used.

6.6.1 Bladder Pumps

For bladder pump operation, the cylinders of inert compressed gas or portable air compressors function with the flow controller as the power source, although the flow controller requires batteries.

6.6.2 Peristaltic Pumps

The peristaltic pumps typically used by AECOM require an external 12 V battery or 120 V AC power source. Commercially available 12 V batteries designed for jump-starting a car battery (“JumpStart” or similar) are preferred since electrical hookup is typically not available; since they are safe, easy to carry, and easily rechargeable; and since the potential contamination issues associated with use of a generator are avoided.

6.7 Turbidity Meter

LaMotte 2020 turbidity meter or similar model.

7.0 Procedure

7.1 Pre-Sampling Activities

Place polyethylene sheeting on the ground and assemble all necessary sampling equipment on top of it. This process helps to prevent contamination of the sampling equipment by the ground surface, reduces wear on the sampling equipment, and reduces the likelihood that contaminated purge water will spill onto the ground surface.

Prior to beginning sampling activities, measure the depth to water and total depth by using the water level indicator and determine the amount of water in the temporary well point. Record this information in the field logbook. If the depth to water is greater than 27 feet, a bladder pump will have to be used.

Wells should be inspected for the presence of light non-aqueous phase liquid (LNAPL). Wells with LNAPL cannot be sampled using bladder pumps or peristaltic pumps and must be sampled with a bailer.

All non-dedicated down-well measuring devices will be thoroughly decontaminated before sampling and between monitoring locations.

7.2 Purging the Temporary Well Point

Temporary well points are typically single-use and are sampled shortly after installation. Purging of temporary well points is completed just prior to sampling in an effort to remove the first water that enters the open borehole.

The following procedures should be followed when collecting grab samples from temporary well points:

Connect all the tubing to the pump. Attach the water discharge line to a 5-gallon purge bucket or carboy using a squeeze clamp or similar device. Connect the pump to the power source (i.e., battery, pump controller and compressed gas cylinder, or air compressor). Lower the tubing or pump to the bottom of the well and begin slowly pumping.

- Note the purge start time.
- Start by surging the tubing up and down several times within the lower part of the screened interval to loft sediment from the bottom of the well. Immediately raise the tubing or pump to the mid-point of the screened interval to remove dislodged sediment from the well. Secure the tubing in place.
- The pump controller should be set to allow for adequate recharge such that a maximum flow rate with no drawdown is achieved and a smooth, laminar discharge flow is achieved. Measure the flow rate using a graduated cylinder and time piece and monitor the water level and pumping rate.
- If drawdown cannot be reasonably controlled at any point during purging, especially to the extent that the well begins to purge dry, collect the groundwater sample immediately.
- If sufficient recharge is available, continue purging. Once drawdown has stabilized and an acceptable flow rate established, begin monitoring turbidity every 5 minutes and continue monitoring flow rate and water level. Water quality parameters should be monitored concurrently using an in-line device (i.e., multi-parameter probe and flow through cell) separate from the turbidity meter at the discharge end of the tubing. Field measurements should be taken after a visible ‘break’ in turbidity is noted in order to avoid running sediment-laden water, which may foul the sensors, through the field meters.
- Purge the temporary well for a period of 20 minutes or until a target turbidity reading of ≤ 25 NTU is achieved, whichever occurs first. Turbidity readings should be collected at a minimum of every 5 minutes.
- If the target turbidity has not been achieved at the end of 20 minutes, assess whether a decreasing turbidity trend exists (i.e., consecutive decreasing readings of >10%). If turbidity is decreasing, continue purging for another 20 minutes. Repeat this process until the target turbidity is achieved or for a maximum purge time of 1 hour (three 20-minute cycles).
- If, at the end of a 20-minute cycle, turbidity readings are not observed to be significantly decreasing and appear instead to stabilize (i.e., three consecutive readings with ±10%), the sample may be collected. The final turbidity must be noted at the time of sample collection.

7.3 Sampling

In keeping with convention, samples should be collected in order of decreasing volatility and reactivity so that the most volatile or reactive samples are collected first. The following are general guidelines.

- Gases (methane/ethane/ethene/hydrogen/CO_2)
- VOCs
- Semivolatile Organic Compounds (SVOCs)

- Pesticides
- PCBs
- Dioxins/furans
- Metals
- Per- and polyfluoroalkyl substances (PFAS)

During sample collection, allow the water to flow directly into and down the side of the sample container without allowing the tubing to touch the inside of the sample container or lid in order to minimize aeration and maintain sample integrity.

- If groundwater turbidity is above 25 NTU at the time of sampling, collect filtered and unfiltered samples using a 0.45-micron filter for analyses that may be impacted by the elevated turbidity (e.g., metals, PCBs). Do not collect filtered samples if the use of a filter may potentially compromise sample quality (e.g., PFAS).

8.0 Quality Control and Assurance

8.1 Collection of representative samples will be ensured through adherence to the procedures in this SOP and the sampling strategy outlined in the QAPP. The field quality control samples identified in the QAPP must be collected. These samples may include field duplicates, equipment rinsate blanks, trip blanks, and matrix spike/matrix spike duplicates.

9.0 Records, Data Analysis, Calculations

9.1 Various forms are required to ensure that adequate documentation is made of the sample collection activities. These forms may include:

- Non-waterproof field logbook;
- Sample collection records;
- Chain-of-custody (CoC) forms; and
- Shipping labels.

9.2 The field logbook is kept as a general log of activities and should not be used in place of the boring log.

9.3 CoC forms are transmitted with the samples to the laboratory for sample tracking purposes.

9.4 Shipping labels are required if sample coolers are to be transported to a laboratory by a third party (courier service).

Author	Reviewer	Revisions (Technical or Editorial)
Suzy Baird Project Scientist	Robert Shoemaker TO Manager	Rev 0 – Initial Issue (February 2013)
Ken O'Donnell, PG Geologist	Claire Mitchell, PE, PMP Senior Engineer	Rev 1 – PFAS sampling update (July 2019)
Matt Costakis, PG Geologist	Claire Mitchell, PE, PMP Senior Engineer	Rev 2 – Purging the Temporary Well Point update (January 2021)

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APPENDIX B

LABORATORY ACCREDITATION

CERTIFICATE OF ACCREDITATION

The ANSI National Accreditation Board

Hereby attests that

Pace Analytical – South Carolina

106 Vantage Point Drive
West Columbia, SC 29172

Fulfills the requirements of

ISO/IEC 17025:2017

and the

U.S. Department of Defense (DoD) Quality Systems Manual for Environmental Laboratories (DoD QSM V5.3)

In the field of

TESTING

This certificate is valid only when accompanied by a current scope of accreditation document.
The current scope of accreditation can be verified at www.anab.org.

R. Douglas Leonard Jr., VP, PILR SBU

Expiry Date: 18 November 2024
Certificate Number: L2224.01

This laboratory is accredited in accordance with the recognized International Standard ISO/IEC 17025:2017. This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated April 2017).

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2017 AND U.S. DEPARTMENT OF DEFENSE (DOD) QUALITY SYSTEMS MANUAL FOR ENVIRONMENTAL LABORATORIES (DOD QSM V5.3)

Pace Analytical – South Carolina

106 Vantage Point Drive
West Columbia, SC 29172
Kelly Nance
803-227-2702

TESTING

Valid to: **November 18, 2024** Certificate Number: **L2224**

Environmental

Non-Potable Water		
Technology	**Method**	**Analyte**
GC	EPA 8151A	2,4,5-T
GC	EPA 8151A	2,4D
GC	EPA 8151A	2,4DB
GC	EPA 8151A	Dalapon
GC	EPA 8151A	Dicamba
GC	EPA 8151A	Dichloroprop
GC	EPA 8151A	Dinoseb
GC	EPA 8151A	MCPA
GC	EPA 8151A	MCPP
GC	EPA 8151A	Silvex(2,4,5 TP)
GC/MS	EPA 8270E	0,0,0-Triethylphosphorothioate
GC/MS	EPA 8270E	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270E	1,2,4-Trichlorobenzene
GC/MS	EPA 8270E	1,2-Dichlorobenzene
GC/MS	EPA 8270E	1,3-Dichlorobenzene
GC/MS	EPA 8270E	1,3,5-Trinitrobenzene
GC/MS	EPA 8270E	1,4-Benzoquinone
GC/MS	EPA 8270E	1,4-Dichlorobenzene
GC/MS	EPA 8270E	1,4-Dinitrobenzene
GC/MS	EPA 8270E	1,4-Naphthoquinone

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E	1-Chloronaphthalene
GC/MS	EPA 8270E	1-Methylnaphthalene
GC/MS	EPA 8270E	1-Naphthylamine
GC/MS	EPA 8270E	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270E	2,3,5,6-Tetrachlorophenol
GC/MS	EPA 8270E	2,4,5-Trichlorophenol
GC/MS	EPA 8270E	2,4,6-Trichlorophenol
GC/MS	EPA 8270E	2,4-Dichlorophenol
GC/MS	EPA 8270E	2,4-Dimethylphenol
GC/MS	EPA 8270E	2,4-Dinitrophenol
GC/MS	EPA 8270E	2,4-Dinitrotoluene
GC/MS	EPA 8270E	2,6-Dichlorophenol
GC/MS	EPA 8270E	2,6-Dinitrotoluene
GC/MS	EPA 8270E	2-Acetylaminofluorene
GC/MS	EPA 8270E	2-Chloronaphthalene
GC/MS	EPA 8270E	2-Chlorophenol
GC/MS	EPA 8270E	2-Methylnaphthalene
GC/MS	EPA 8270E	2-Methylphenol
GC/MS	EPA 8270E	2-Naphthylamine
GC/MS	EPA 8270E	2-Nitroaniline
GC/MS	EPA 8270E	2-Nitrophenol
GC/MS	EPA 8270E	2-Picoline
GC/MS	EPA 8270E	3,3'-Dichlorobenzidine
GC/MS	EPA 8270E	3,3'-Dimethylbenzidine
GC/MS	EPA 8270E	3-Methylcholanthrene
GC/MS	EPA 8270E	3-Nitroaniline
GC/MS	EPA 8270E	4,4'-Methylene-bis-chloroaniline
GC/MS	EPA 8270E	4,6-Dinitro-2-methylphenol
GC/MS	EPA 8270E	4-Aminobiphenyl
GC/MS	EPA 8270E	4-Bromophenylphenylether
GC/MS	EPA 8270E	4-Chloro-3-methylphenol
GC/MS	EPA 8270E	4-Chloroaniline
GC/MS	EPA 8270E	4-Chlorophenylphenylether
GC/MS	EPA 8270E	4-Nitroaniline
GC/MS	EPA 8270E	4-Nitrophenol
GC/MS	EPA 8270E	5-Nitro-o-toluidine

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E	7,12-Dimethylbenzo(a)anthracene
GC/MS	EPA 8270E	Acenaphthene
GC/MS	EPA 8270E	Acenaphthylene
GC/MS	EPA 8270E	Acetophenone
GC/MS	EPA 8270E	Aniline
GC/MS	EPA 8270E	Anthracene
GC/MS	EPA 8270E	Aramite
GC/MS	EPA 8270E	Atrazine
GC/MS	EPA 8270E	Azobenzene
GC/MS	EPA 8270E	Benzaldhyde
GC/MS	EPA 8270E	Benzidine
GC/MS	EPA 8270E	Benzo(a)Anthracene
GC/MS	EPA 8270E	Benzo(a)pyrene
GC/MS	EPA 8270E	Benzo(b)fluoranthene
GC/MS	EPA 8270E	Benzo(g,h,i)perylene
GC/MS	EPA 8270E	Benzo(k)fluoranthene
GC/MS	EPA 8270E	Benzoic acid
GC/MS	EPA 8270E	Benzyl alcohol
GC/MS	EPA 8270E	Biphenyl
GC/MS	EPA 8270E	bis(2-Chloroethoxy)methane
GC/MS	EPA 8270E	bis(2-Chloroethyl)ether
GCMS	EPA 8270E	Bis (2-Chloro-1-methylethyl) ether
GC/MS	EPA 8270E	bis(2-Ethylhexyl)phthalate
GC/MS	EPA 8270E	Butylbenzylphthalate
GC/MS	EPA 8270E	Caprolactam
GC/MS	EPA 8270E	Carbazole
GC/MS	EPA 8270E	Chrysene
GC/MS	EPA 8270E	Chlorobenzilate
GC/MS	EPA 8270E	DEET
GC/MS	EPA 8270E	Diallate
GC/MS	EPA 8270E	Dibenzo(a,h)acridine
GC/MS	EPA 8270E	Dibenzo(a,h)anthracene
GC/MS	EPA 8270E	Dibenzo(a,e)pyrene
GC/MS	EPA 8270E	Dibenzofuran
GC/MS	EPA 8270E	Diethylphthalate
GC/MS	EPA 8270E	Dimethoate

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E	Dimethylphthalate
GC/MS	EPA 8270E	Di-n-butylphthalate
GC/MS	EPA 8270E	Di-n-octylphthalate
GC/MS	EPA 8270E	Dinoseb
GC/MS	EPA 8270E	Disulfoton
GC/MS	EPA 8270E	Ethyl methacrylate
GC/MS	EPA 8270E	Ethyl methanesulfonate
GC/MS	EPA 8270E	Famphur
GC/MS	EPA 8270E	Fluoranthene
GC/MS	EPA 8270E	Fluorene
GC/MS	EPA 8270E	Hexachlorobenzene
GC/MS	EPA 8270E	Hexachlorobutadiene
GC/MS	EPA 8270E	Hexachlorocyclopentadiene
GC/MS	EPA 8270E	Hexachloroethane
GC/MS	EPA 8270E	Hexachloropropene
GC/MS	EPA 8270E	Indene
GC/MS	EPA 8270E	Indeno(1,2,3-c,d)pyrene
GC/MS	EPA 8270E	Isodrin
GC/MS	EPA 8270E	Isophorone
GC/MS	EPA 8270E	Isosafrole
GC/MS	EPA 8270E	Kepone
GC/MS	EPA 8270E	m+p-Cresol
GC/MS	EPA 8270E	m-Dinitrobenzene
GC/MS	EPA 8270E	Methyl methacrylate
GC/MS	EPA 8270E	Methyl methanesulfonate
GC/MS	EPA 8270E	Methyl parathion
GC/MS	EPA 8270E	Mirex
GC/MS	EPA 8270E	Naphthalene
GC/MS	EPA 8270E	Nitrobenzene
GC/MS	EPA 8270E	N-Nitrosodiethylamine
GC/MS	EPA 8270E	N-Nitrosodimethylamine
GC/MS	EPA 8270E	N-Nitrosodi-n-butylamine
GC/MS	EPA 8270E	n-Nitroso-di-n-propylamine
GC/MS	EPA 8270E	n-Nitrosodiphenylamine
GC/MS	EPA 8270E	N-Nitrosomethylethylamine
GC/MS	EPA 8270E	N-Nitrosomorpholine

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E	N-Nitrosopiperidine
GC/MS	EPA 8270E	N-Nitrosopyrrolidine
GC/MS	EPA 8270E	o-Cresol
GC/MS	EPA 8270E	o-Toluidine
GC/MS	EPA 8270E	p-(Dimethylamino)azobenzene
GC/MS	EPA 8270E	Parathion
GC/MS	EPA 8270E	Pentachlorobenzene
GC/MS	EPA 8270E	Pentachloroethane
GC/MS	EPA 8270E	Pentachloronitrobenzene
GC/MS	EPA 8270E	Pentachlorophenol
GC/MS	EPA 8270E	Phenacetin
GC/MS	EPA 8270E	Phenanthrene
GC/MS	EPA 8270E	Phenol
GC/MS	EPA 8270E	Phorate
GC/MS	EPA 8270E	Pronamide
GC/MS	EPA 8270E	Pyrene
GC/MS	EPA 8270E	Pyridine
GC/MS	EPA 8270E	Quinoline
GC/MS	EPA 8270E	Safrole
GC/MS	EPA 8270E	Tetraethyl dithiopyrophosphate
GC/MS	EPA 8270E	Thionazine
GC/MS	EPA 8270E	Tributyl phosphate
GC/MS	EPA 8270E	p-Phenylenediamine
GC/MS	EPA 8270E SIM	Acenaphthene
GC/MS	EPA 8270E SIM	Acenaphthylene
GC/MS	EPA 8270E SIM	Anthracene
GC/MS	EPA 8270E SIM	Benzo(a)anthracene
GC/MS	EPA 8270E SIM	Benzo(a)pyrene
GC/MS	EPA 8270E SIM	Benzo(b)fluoranthene
GC/MS	EPA 8270E SIM	Benzo(g,h,i)perylene
GC/MS	EPA 8270E SIM	Benzo(k)fluoranthene
GC/MS	EPA 8270E SIM	Chrysene
GC/MS	EPA 8270E SIM	Dibenz(a,h)anthracene
GC/MS	EPA 8270E SIM	1,4-Dioxane
GC/MS	EPA 8270E SIM	Fluoranthene
GC/MS	EPA 8270E SIM	Fluorene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E SIM	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270E SIM	1-Methylphenol
GC/MS	EPA 8270E SIM	2-Methylphenol
GC/MS	EPA 8270E SIM	Naphthalene
GC/MS	EPA 8270E SIM	Pentachlorophenol
GC/MS	EPA 8270E SIM	Phenanthrene
GC/MS	EPA 8270E SIM	Pyrene
GC/MS	EPA 625.1	Acenaphthene
GC/MS	EPA 625.1	Acenaphthylene
GC/MS	EPA 625.1	Aniline
GC/MS	EPA 625.1	Anthracene
GC/MS	EPA 625.1	Benzidine
GC/MS	EPA 625.1	Benzo(a)anthracene
GC/MS	EPA 625.1	Benzo(a)pyrene
GC/MS	EPA 625.1	Benzo(b)fluoranthene
GC/MS	EPA 625.1	Benzo(g,h,i)perylene
GC/MS	EPA 625.1	Benzo(k)fluoranthene
GC/MS	EPA 625.1	Benzoic acid
GC/MS	EPA 625.1	Benzyl alcohol
GC/MS	EPA 625.1	4-Bromophenyl phenyl ether
GC/MS	EPA 625.1	Butyl benzyl phthalate
GC/MS	EPA 625.1	Carbazole
GC/MS	EPA 625.1	bis (2-Chloro-1-methylethyl) ether
GC/MS	EPA 625.1	4-Chloro-3-methyl phenol
GC/MS	EPA 625.1	4-Chloroaniline
GC/MS	EPA 625.1	bis(2-Chloroethoxy)methane
GC/MS	EPA 625.1	bis(2-Chloroethyl)ether
GC/MS	EPA 625.1	2-Chloronaphthalene
GC/MS	EPA 625.1	2-Chlorophenol
GC/MS	EPA 625.1	4-Chlorophenyl phenyl ether
GC/MS	EPA 625.1	Chrysene
GC/MS	EPA 625.1	n-Decane
GC/MS	EPA 625.1	Dibenzo(a,h)anthracene
GC/MS	EPA 625.1	Dibenzofuran
GC/MS	EPA 625.1	2,3-Dichloroaniline
GC/MS	EPA 625.1	1,2-Dichlorobenzene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 625.1	1,3-Dichlorobenzene
GC/MS	EPA 625.1	1,4-Dichlorobenzene
GC/MS	EPA 625.1	3,3'-Dichlorobenzidine
GC/MS	EPA 625.1	2,4-Dichlorophenol
GC/MS	EPA 625.1	2,6-Dichlorophenol
GC/MS	EPA 625.1	Diethylphthalate
GC/MS	EPA 625.1	Dimethyl phthalate
GC/MS	EPA 625.1	3,3'-Dimethylbenzidine
GC/MS	EPA 625.1	2,4-Dimethylphenol
GC/MS	EPA 625.1	Di-n-butyl phthalate
GC/MS	EPA 625.1	4,6-Dinitro-2-methylphenol
GC/MS	EPA 625.1	2,4-Dinitrophenol
GC/MS	EPA 625.1	2,4-Dinitrotoluene
GC/MS	EPA 625.1	2,6-Dinitrotoluene
GC/MS	EPA 625.1	Di-n-octylphthalate
GC/MS	EPA 625.1	1,2-Diphenylhydrazine(as azobenzene)
GC/MS	EPA 625.1	bis(2-Ethylhexyl)phthalate
GC/MS	EPA 625.1	Fluoranthene
GC/MS	EPA 625.1	Fluorene
GC/MS	EPA 625.1	Hexachlorobenzene
GC/MS	EPA 625.1	Hexachlorobutadiene
GC/MS	EPA 625.1	Hexachlorocyclopentadiene
GC/MS	EPA 625.1	Hexachloroethane
GC/MS	EPA 625.1	Indeno(1,2,3-c,d)pyrene
GC/MS	EPA 625.1	Isophorone
GC/MS	EPA 625.1	1-Methylnaphthalene
GC/MS	EPA 625.1	2-Methylnaphthalene
GC/MS	EPA 625.1	2-Methylphenol
GC/MS	EPA 625.1	3+4-Methylphenol
GC/MS	EPA 625.1	N,N-Diethyl-m-toluamide (DEET)
GC/MS	EPA 625.1	Naphthalene
GC/MS	EPA 625.1	Nitrobenzene
GC/MS	EPA 625.1	2-Nitrophenol
GC/MS	EPA 625.1	4-Nitrophenol
GC/MS	EPA 625.1	N-Nitrosodimethylamine
GC/MS	EPA 625.1	N-Nitrosodi-n-propylamine

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 625.1	N-Nitrosodiphenylamine (Diphenylamine)
GC/MS	EPA 625.1	n-Octadecane
GC/MS	EPA 625.1	Pentachlorophenol
GC/MS	EPA 625.1	Phenanthrene
GC/MS	EPA 625.1	Phenol
GC/MS	EPA 625.1	Piperonyl butoxide (PIP)
GC/MS	EPA 625.1	Pyrene
GC/MS	EPA 625.1	alpha-Terpineol
GC/MS	EPA 625.1	1,2,4-Trichlorobenzene
GC/MS	EPA 625.1	2,4,6-Trichlorophenol
GC/MS	EPA 8260D	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260D	1,1,1-Trichloroethane
GC/MS	EPA 8260D	1,1,2,2-Tetrachloroethene
GC/MS	EPA 8260D	1,1,2-Trichloroethane
GC/MS	EPA 8260D	1,1-Dichloroethane
GC/MS	EPA 8260D	1,1-Dichloroethene
GC/MS	EPA 8260D	1,1-Dichloropropene
GC/MS	EPA 8260D	1,2,3-Trichlorobenzene
GC/MS	EPA 8260D	1,2,3-Trichloropropane
GC/MS	EPA 8260D	1,2,4-Trichlorobenzene
GC/MS	EPA 8260D	1,2,4-Trimethylbenzene
GC/MS	EPA 8260D	1,2-Dibromo-3-chloropropane
GC/MS	EPA 8260D	1,2-Dibromoethane (EDB)
GC/MS	EPA 8260D	1,2-Dichlorobenzene
GC/MS	EPA 8260D	1,2-Dichloroethane
GC/MS	EPA 8260D	1,2-Dichloropropane
GC/MS	EPA 8260D	1,3,5-Trimethylbenzene
GC/MS	EPA 8260D	1,3-Dichlorobenzene
GC/MS	EPA 8260D	1,3-Dichloropropane
GC/MS	EPA 8260D	1,4-Dichlorobenzene
GC/MS	EPA 8260D	1,4-Dioxane
GC/MS	EPA 8260D	2,2-Dichloropropane
GC/MS	EPA 8260D	2-Butanone (MEK)
GC/MS	EPA 8260D	2-Chloroethylvinyl ether
GC/MS	EPA 8260D	2-Chlorotoluene
GC/MS	EPA 8260D	2-Hexanone

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260D	3,3-Dimethyl-1-butanol
GC/MS	EPA 8260D	4-Chlorotoluene
GC/MS	EPA 8260D	4-Methyl-2-pentanone
GC/MS	EPA 8260D	Acetone
GC/MS	EPA 8260D	Acetonitrile
GC/MS	EPA 8260D	Acrolein
GC/MS	EPA 8260D	Acrylonitrile
GC/MS	EPA 8260D	Allyl chloride
GC/MS	EPA 8260D	Benzene
GC/MS	EPA 8260D	Benzyl chloride
GC/MS	EPA 8260D	Bromobenzene
GC/MS	EPA 8260D	Bromochloromethane
GC/MS	EPA 8260D	Bromodichloromethane
GC/MS	EPA 8260D	Bromoform
GC/MS	EPA 8260D	Bromomethane
GC/MS	EPA 8260D	Carbon disulfide
GC/MS	EPA 8260D	Carbon tetrachloride
GC/MS	EPA 8260D	Chlorobenzene
GC/MS	EPA 8260D	Chloroethane
GC/MS	EPA 8260D	Chloroform
GC/MS	EPA 8260D	Chloromethane
GC/MS	EPA 8260D	Chloroprene
GC/MS	EPA 8260D	cis-1,2-Dichloroethene
GC/MS	EPA 8260D	cis-1,3-Dichloropropene
GC/MS	EPA 8260D	Cyclohexane
GC/MS	EPA 8260D	Cyclohexanone
GC/MS	EPA 8260D	Dibromochloromethane
GC/MS	EPA 8260D	Dibromomethane
GC/MS	EPA 8260D	Dichlorodifluoromethane
GC/MS	EPA 8260D	Diisopropyl ether (IPE)
GC/MS	EPA 8260D	Ethanol
GC/MS	EPA 8260D	Ethyl ether
GC/MS	EPA 8260D	Ethyl methacrylate
GC/MS	EPA 8260D	Ethylbenzene
GC/MS	EPA 8260D	Ethyl-Tert-Butyl Ether
GC/MS	EPA 8260D	Freon 113

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260D	Hexachlorobutadiene
GC/MS	EPA 8260D	Isobutyl alcohol
GC/MS	EPA 8260D	Isopropylbenzene
GC/MS	EPA 8260D	m+p-Xylenes
GC/MS	EPA 8260D	Methacrylonitrile
GC/MS	EPA 8260D	Methyl Acetate
GC/MS	EPA 8260D	Methyl iodide
GC/MS	EPA 8260D	Methyl methacrylate
GC/MS	EPA 8260D	Methyl tertiary butyl ether (MTBE)
GC/MS	EPA 8260D	Methylcyclohexane
GC/MS	EPA 8260D	Methylene chloride
GC/MS	EPA 8260D	Naphthalene
GC/MS	EPA 8260D	n-Butylbenzene
GC/MS	EPA 8260D	n-Propylbenzene
GC/MS	EPA 8260D	o-Xylene
GC/MS	EPA 8260D	Pentachloroethane
GC/MS	EPA 8260D	p-Isopropyltoluene
GC/MS	EPA 8260D	Propionitrile
GC/MS	EPA 8260D	sec-Butylbenzene
GC/MS	EPA 8260D	Styrene
GC/MS	EPA 8260D	Tert-Amyl Alcohol (TAA)
GC/MS	EPA 8260D	Tert-Amyl Methyl Ether (TAME)
GC/MS	EPA 8260D	Tert-Butyl Alcohol (TBA)
GC/MS	EPA 8260D	Tert-Butyl Formate (TBF)
GC/MS	EPA 8260D	tert-Butylbenzene
GC/MS	EPA 8260D	Tetrachloroethene
GC/MS	EPA 8260D	Tetrahydrofuran
GC/MS	EPA 8260D	Toluene
GC/MS	EPA 8260D	Total Xylenes
GC/MS	EPA 8260D	trans-1,2-Dichloroethene
GC/MS	EPA 8260D	trans-1,3-Dichloropropene
GC/MS	EPA 8260D	trans-1,4-Dichloro-2-butene
GC/MS	EPA 8260D	Trichloroethene
GC/MS	EPA 8260D	Trichlorofluoromethane
GC/MS	EPA 8260D	Vinyl acetate
GC/MS	EPA 8260D	Vinyl chloride

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260D SIM	1,4-Dioxane
GC/MS	EPA 624.1	Acetone
GC/MS	EPA 624.1	Acetonitrile
GC/MS	EPA 624.1	Acrolein
GC/MS	EPA 624.1	Acrylonitrile
GC/MS	EPA 624.1	Benzene
GC/MS	EPA 624.1	Bromochloromethane
GC/MS	EPA 624.1	Bromodichloromethane
GC/MS	EPA 624.1	Bromoform
GC/MS	EPA 624.1	Bromomethane (Methyl bromide)
GC/MS	EPA 624.1	2-Butanone (MEK)
GC/MS	EPA 624.1	Carbon disulfide
GC/MS	EPA 624.1	Carbon tetrachloride
GC/MS	EPA 624.1	Chlorobenzene
GC/MS	EPA 624.1	Chloroethane
GC/MS	EPA 624.1	2-Chloroethylvinylether
GC/MS	EPA 624.1	Chloroform
GC/MS	EPA 624.1	Chloromethane (Methyl chloride)
GC/MS	EPA 624.1	Dibromochloromethane
GC/MS	EPA 624.1	1,2-Dibromoethane (EDB)
GC/MS	EPA 624.1	1,2-Dichlorobenzene
GC/MS	EPA 624.1	1,3-Dichlorobenzene
GC/MS	EPA 624.1	1,4-Dichlorobenzene
GC/MS	EPA 624.1	Dichlorodifluoromethane
GC/MS	EPA 624.1	1,1-Dichloroethane
GC/MS	EPA 624.1	1,2-Dichloroethane
GC/MS	EPA 624.1	1,1-Dichloroethene
GC/MS	EPA 624.1	cis-1,2-Dichloroethene
GC/MS	EPA 624.1	trans-1,2-Dichloroethene
GC/MS	EPA 624.1	1,2-Dichloropropane
GC/MS	EPA 624.1	cis-1,3-Dichloropropene
GC/MS	EPA 624.1	trans-1,3-Dichloropropene
GC/MS	EPA 624.1	Diisopropyl ether (IPE)
GC/MS	EPA 624.1	1,4-Dioxane
GC/MS	EPA 624.1	Ethylbenzene
GC/MS	EPA 624.1	Methyl methacrylate

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 624.1	Methyl tertiary butyl ether (MTBE)
GC/MS	EPA 624.1	Methylene chloride
GC/MS	EPA 624.1	Naphthalene
GC/MS	EPA 624.1	Styrene
GC/MS	EPA 624.1	1,1,2,2-Tetrachloroethane
GC/MS	EPA 624.1	Tetrachloroethene
GC/MS	EPA 624.1	Toluene
GC/MS	EPA 624.1	1,2,4-Trichlorobenzene
GC/MS	EPA 624.1	1,1,1-Trichloroethane
GC/MS	EPA 624.1	1,1,2-Trichloroethane
GC/MS	EPA 624.1	Trichloroethene
GC/MS	EPA 624.1	Trichlorofluoromethane
GC/MS	EPA 624.1	1,2,4-Trimethylbenzene
GC/MS	EPA 624.1	1,3,5-Trimethylbenzene
GC/MS	EPA 624.1	Vinyl acetate
GC/MS	EPA 624.1	Vinyl chloride
GC/MS	EPA 624.1	Xylenes (total)
GC/MS	SM 6200B-2011	Benzene
GC/MS	SM 6200B-2011	Bromobenzene
GC/MS	SM 6200B-2011	Bromochloromethane
GC/MS	SM 6200B-2011	Bromodichloromethane
GC/MS	SM 6200B-2011	Bromoform
GC/MS	SM 6200B-2011	Bromomethane (Methyl bromide)
GC/MS	SM 6200B-2011	n-Butylbenzene
GC/MS	SM 6200B-2011	sec-Butylbenzene
GC/MS	SM 6200B-2011	tert-Butylbenzene
GC/MS	SM 6200B-2011	Carbon tetrachloride
GC/MS	SM 6200B-2011	Chlorobenzene
GC/MS	SM 6200B-2011	Chloroethane
GC/MS	SM 6200B-2011	Chloroform
GC/MS	SM 6200B-2011	Chloromethane (Methyl chloride)
GC/MS	SM 6200B-2011	2-Chlorotoluene
GC/MS	SM 6200B-2011	4-Chlorotoluene
GC/MS	SM 6200B-2011	Dibromochloromethane
GC/MS	SM 6200B-2011	1,2-Dibromoethane (EDB)
GC/MS	SM 6200B-2011	Dibromomethane (Methylene bromide)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	SM 6200B-2011	1,2-Dichlorobenzene
GC/MS	SM 6200B-2011	1,3-Dichlorobenzene
GC/MS	SM 6200B-2011	1,4-Dichlorobenzene
GC/MS	SM 6200B-2011	Dichlorodifluoromethane
GC/MS	SM 6200B-2011	1,1-Dichloroethane
GC/MS	SM 6200B-2011	1,2-Dichloroethane
GC/MS	SM 6200B-2011	1,1-Dichloroethene
GC/MS	SM 6200B-2011	cis-1,2-Dichloroethene
GC/MS	SM 6200B-2011	trans-1,2-Dichloroethene
GC/MS	SM 6200B-2011	1,2-Dichloropropane
GC/MS	SM 6200B-2011	1,3-Dichloropropane
GC/MS	SM 6200B-2011	2,2-Dichloropropane
GC/MS	SM 6200B-2011	1,1-Dichloropropene
GC/MS	SM 6200B-2011	cis-1,3-Dichloropropene
GC/MS	SM 6200B-2011	trans-1,3-Dichloropropene
GC/MS	SM 6200B-2011	Diisopropyl ether (IPE)
GC/MS	SM 6200B-2011	Ethylbenzene
GC/MS	SM 6200B-2011	Hexachlorobutadiene
GC/MS	SM 6200B-2011	Isopropylbenzene (Cumene)
GC/MS	SM 6200B-2011	p-Isopropyltoluene (p-Cymene)
GC/MS	SM 6200B-2011	Methyl tertiary butyl ether (MTBE)
GC/MS	SM 6200B-2011	4-Methyl-2-pentanone
GC/MS	SM 6200B-2011	Methylene chloride
GC/MS	SM 6200B-2011	Naphthalene
GC/MS	SM 6200B-2011	n-Propylbenzene
GC/MS	SM 6200B-2011	Styrene
GC/MS	SM 6200B-2011	1,1,1,2-Tetrachloroethane
GC/MS	SM 6200B-2011	1,1,2,2-Tetrachloroethane
GC/MS	SM 6200B-2011	Tetrachloroethene
GC/MS	SM 6200B-2011	Toluene
GC/MS	SM 6200B-2011	1,1,2-Trichloro-1,2,2-Trifluoroethane
GC/MS	SM 6200B-2011	1,2,3-Trichlorobenzene
GC/MS	SM 6200B-2011	1,2,4-Trichlorobenzene
GC/MS	SM 6200B-2011	1,1,1-Trichloroethane
GC/MS	SM 6200B-2011	1,1,2-Trichloroethane
GC/MS	SM 6200B-2011	Trichloroethene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	SM 6200B-2011	Trichlorofluoromethane
GC/MS	SM 6200B-2011	1,2,3-Trichloropropane
GC/MS	SM 6200B-2011	1,3,5-Trimethylbenzene (Mesitylene)
GC/MS	SM 6200B-2011	1,2,4-Trimethylbenzene
GC/MS	SM 6200B-2011	Vinyl chloride
GC/MS	SM 6200B-2011	m+p - Xylenes
GC/MS	SM 6200B-2011	o-Xylene
GC	EPA 8081B	4,4'-DDD
GC	EPA 8081B	4,4'-DDE
GC	EPA 8081B	4,4'-DDT
GC	EPA 8081B	Aldrin
GC	EPA 8081B	alpha-BHC
GC	EPA 8081B	alpha-Chlordane
GC	EPA 8081B	beta-BHC
GC	EPA 8081B	Chlordane
GC	EPA 8081B	delta-BHC
GC	EPA 8081B	Dieldrin
GC	EPA 8081B	Endosulfan I
GC	EPA 8081B	Endosulfan II
GC	EPA 8081B	Endosulfan sulfate
GC	EPA 8081B	Endrin
GC	EPA 8081B	Endrin Aldehyde
GC	EPA 8081B	Endrin Ketone
GC	EPA 8081B	gamma-BHC (Lindane)
GC	EPA 8081B	gamma-Chlordane
GC	EPA 8081B	Heptachlor
GC	EPA 8081B	Heptachlor Epoxide
GC	EPA 8081B	Methoxychlor
GC	EPA 8081B	Mirex
GC	EPA 8081B	Toxaphene
GC	EPA 8082A	Aroclor 1016
GC	EPA 8082A	Aroclor 1221
GC	EPA 8082A	Aroclor 1232
GC	EPA 8082A	Aroclor 1242
GC	EPA 8082A	Aroclor 1248
GC	EPA 8082A	Aroclor 1254
GC	EPA 8082A	Aroclor 1260

Non-Potable Water		
Technology	**Method**	**Analyte**
GC	EPA 8082A	Aroclor 1262
GC	EPA 8082A	Aroclor 1268
GC	EPA 608.3	Aldrin
GC	EPA 608.3	gamma-BHC (Lindane)
GC	EPA 608.3	alpha-BHC
GC	EPA 608.3	beta-BHC
GC	EPA 608.3	delta-BHC
GC	EPA 608.3	Chlordane
GC	EPA 608.3	cis-Chlordane
GC	EPA 608.3	trans-Chlordane
GC	EPA 608.3	4,4'-DDD
GC	EPA 608.3	4,4'-DDE
GC	EPA 608.3	4,4'-DDT
GC	EPA 608.3	Dieldrin
GC	EPA 608.3	Endosulfan I
GC	EPA 608.3	Endosulfan II
GC	EPA 608.3	Endosulfan sulfate
GC	EPA 608.3	Endrin
GC	EPA 608.3	Endrin aldehyde
GC	EPA 608.3	Endrin ketone
GC	EPA 608.3	Heptachlor
GC	EPA 608.3	Heptachlor epoxide
GC	EPA 608.3	Methoxychlor
GC	EPA 608.3	Toxaphene
GC	EPA 608.3	Aroclor 1016
GC	EPA 608.3	Aroclor 1221
GC	EPA 608.3	Aroclor 1232
GC	EPA 608.3	Aroclor 1242
GC	EPA 608.3	Aroclor 1248
GC	EPA 608.3	Aroclor 1254
GC	EPA 608.3	Aroclor 1260
HPLC	EPA 8330A	2-Amino-4,6-dinitrotoluene
HPLC	EPA 8330A	4-Amino-2,6-dinitrotoluene
HPLC	EPA 8330A	1,3-Dinitrobenzene
HPLC	EPA 8330A	2,4-Dinitrotoluene
HPLC	EPA 8330A	2,6-Dinitrotoluene

Non-Potable Water		
Technology	**Method**	**Analyte**
HPLC	EPA 8330A	HMX
HPLC	EPA 8330A	Nitrobenzene
HPLC	EPA 8330B	Nitrocellulose
HPLC	EPA 8330B	Nitroguanidine
HPLC	EPA 8330B	Guanidine nitrate
HPLC	EPA 8330A	2-Nitrotoluene
HPLC	EPA 8330A	3-Nitrotoluene
HPLC	EPA 8330A	4-Nitrotoluene
HPLC	EPA 8330A	RDX
HPLC	EPA 8330A	Tetryl
HPLC	EPA 8330A	1,3,5-Trinitrobenzene
HPLC	EPA 8330A	2,4,6-Trinitrotoluene
HPLC	EPA 8330B	2-Amino-4,6-dinitrotoluene
HPLC	EPA 8330B	4-Amino-2,6-dinitrotoluene
HPLC	EPA 8330B	1,3-Dinitrobenzene
HPLC	EPA 8330B	2,4-Dinitrotoluene
HPLC	EPA 8330B	2,6-Dinitrotoluene
HPLC	EPA 8330B	HMX
HPLC	EPA 8330B	Nitrobenzene
HPLC	EPA 8330B	Nitroglycerin (NG)
HPLC	EPA 8330B	2-Nitrotoluene
HPLC	EPA 8330B	3-Nitrotoluene
HPLC	EPA 8330B	4-Nitrotoluene
HPLC	EPA 8330B	RDX
HPLC	EPA 8330B	Tetryl
HPLC	EPA 8330B	1,3,5-Trinitrobenzene
HPLC	EPA 8330B	2,4,6-Trinitrotoluene
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-ethylperfluoro-1-octanesulfonamidoacetic acid (EtFOSAA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Fluorotelomer sulfonate 8:2 [1H,1H,2H,2H-perfluorodecane sulfonate] (8:2 FTS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Fluorotelomer sulfonate 4:2 [1H,1H,2H,2H-perfluorohexane sulfonate] (4:2 FTS)

Non-Potable Water		
Technology	**Method**	**Analyte**
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Fluorotelomer sulfonate 6:2 [1H,1H,2H,2H-perfluorooctane sulfonate] (6:2 FTS
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	GenX (HFPO-DA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-methylperfluoro-1-octanesulfonamidoacetic acid (MeFOSAA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-1-butanesulfonate (PFBS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-n-butanoic acid (PFBA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-1-decanesulfonate (PFDS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-n-decanoic acid (PFDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-n-dodecanoic acid (PFDoA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-1-heptanesulfonate (PFHpS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-n-heptanoic acid (PFHpA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-1-hexanesulfonate (PFHxS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-n-hexanoic acid (PFHxA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-1-nonanesulfonate (PFNS)

Non-Potable Water		
Technology	**Method**	**Analyte**
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-n-nonanoic acid (PFNA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorooctanesulfonate (PFOS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-1-octanesulfonamide (PFOSA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-n-octanoic acid (PFOA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-n-pentanoic acid (PFPeA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-1-pentansulfonate (PFPeS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-n-tetradecanoic acid (PFTeDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-n-tridecanoic acid (PFTrDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-n-undecanoic acid (PFUDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-methylperfluoro-1-octanesulfonamide (MeFOSA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-ethylperfluoro-1-octanesulfonamide (EtFOSA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	1H,1H,2H,2H-perfluorododecane sulfonate (10:2FTS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-Ethyl Perfluorooctane sulfonamido ethanol (EtFOSE)

Non-Potable Water		
Technology	**Method**	**Analyte**
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-Methyl Perfluorooctane sulfonamido ethanol (MeFOSE)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Sodium dodecafluoro-3H-4, 8-dioxanonanoate (NaDONA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	9Cl-PF3ONS
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	11Cl-PF3OUDS
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorododecanesulfonic acid (PFDOS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorohexadecanoic acid (PFHxDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorooctadecanoic acid (PFODA)
GC	EPA 8015C	Diesel Range Organics
GC	EPA 8015C	WI-Diesel Range Organics
GC	EPA 8015C	Gasoline Range Organics
GC	EPA 8015C	WI-Gasoline Range Organics
GC	RSK - 175	Methane, Ethane, Ethene
GC	EPA 8011	1,2-Dibromoethane (EDB)
GC	EPA 8011	1,2-Dibromo-3-chloropropane (DBCP)
GC	FL-PRO	FL-PRO
ICP	EPA 6010D	Aluminum
ICP	EPA 6010D	Antimony
ICP	EPA 6010D	Arsenic
ICP	EPA 6010D	Barium
ICP	EPA 6010D	Beryllium
ICP	EPA 6010D	Cadmium
ICP	EPA 6010D	Calcium
ICP	EPA 6010D	Chromium
ICP	EPA 6010D	Cobalt
ICP	EPA 6010D	Copper

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP	EPA 6010D	Iron
ICP	EPA 6010D	Lead
ICP	EPA 6010D	Magnesium
ICP	EPA 6010D	Manganese
ICP	EPA 6010D	Molybdenum
ICP	EPA 6010D	Nickel
ICP	EPA 6010D	Potassium
ICP	EPA 6010D	Selenium
ICP	EPA 6010D	Silver
ICP	EPA 6010D	Sodium
ICP	EPA 6010D	Thallium
ICP	EPA 6010D	Tin
ICP	EPA 6010D	Vanadium
ICP	EPA 6010D	Zinc
ICP/MS	EPA 200.8	Aluminum
ICP/MS	EPA 200.8	Antimony
ICP/MS	EPA 200.8	Arsenic
ICP/MS	EPA 200.8	Barium
ICP/MS	EPA 200.8	Beryllium
ICP/MS	EPA 200.8	Boron
ICP/MS	EPA 200.8	Cadmium
ICP/MS	EPA 200.8	Calcium
ICP/MS	EPA 200.8	Chromium
ICP/MS	EPA 200.8	Cobalt
ICP/MS	EPA 200.8	Copper
ICP/MS	EPA 200.8	Iron
ICP/MS	EPA 200.8	Lead
ICP/MS	EPA 200.8	Magnesium
ICP/MS	EPA 200.8	Manganese
ICP/MS	EPA 200.8	Molybdenum
ICP/MS	EPA 200.8	Nickel
ICP/MS	EPA 200.8	Potassium
ICP/MS	EPA 200.8	Selenium
ICP/MS	EPA 200.8	Silicon
ICP/MS	EPA 200.8	Silver
ICP/MS	EPA 200.8	Sodium

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP/MS	EPA 200.8	Thallium
ICP/MS	EPA 200.8	Tin
ICP/MS	EPA 200.8	Titanium
ICP/MS	EPA 200.8	Vanadium
ICP/MS	EPA 200.8	Zinc
ICP/MS	EPA 6020B	Aluminum
ICP/MS	EPA 6020B	Antimony
ICP/MS	EPA 6020B	Arsenic
ICP/MS	EPA 6020B	Barium
ICP/MS	EPA 6020B	Beryllium
ICP/MS	EPA 6020B	Cadmium
ICP/MS	EPA 6020B	Calcium
ICP/MS	EPA 6020B	Chromium
ICP/MS	EPA 6020B	Cobalt
ICP/MS	EPA 6020B	Copper
ICP/MS	EPA 6020B	Iron
ICP/MS	EPA 6020B	Lead
ICP/MS	EPA 6020B	Magnesium
ICP/MS	EPA 6020B	Manganese
ICP/MS	EPA 6020B	Nickel
ICP/MS	EPA 6020B	Potassium
ICP/MS	EPA 6020B	Selenium
ICP/MS	EPA 6020B	Silver
ICP/MS	EPA 6020B	Sodium
ICP/MS	EPA 6020B	Thallium
ICP/MS	EPA 6020B	Tin
ICP/MS	EPA 6020B	Vanadium
ICP/MS	EPA 6020B	Zinc
CVAA	EPA 1631E	Low Level Mercury
CVAA	EPA 7470A / EPA 245.1	Mercury
Gravimetric	EPA 1664B	Oil & Grease
Titration	SM 2320B-2011	Alkalinity
Calculation	SM 2320B-2011	Bicarbonate Alkalinity
Calculation	SM 2320B-2011	Carbonate Alkalinity
Calculation	SM 2320B-2011	Hydroxide Alkalinity
Calculation	SM 4500-CO2 D	Carbon Dioxide (CO2)

Non-Potable Water		
Technology	**Method**	**Analyte**
Photometric	SM 2120F-2011	Color
Photometric	SM 2120B-2011	Platinum Cobalt Color
UV/VIS	SM 3500-Fe B-2011	Ferrous Iron
Titration	SM 4500-S2 F-2011	Sulfide
Wet Oxidation	EPA 9060A	TOC
Wet Oxidation	SM 5310C-2011	TOC
Titration	SM 2340C-2011	Total Hardness
Filtration	SM 2540C-2011	Total Dissolved Solids (TDS)
Filtration	SM 2540D-2011	Total Suspended Solids (TSS)
UV/VIS	Kelada-01	Total Cyanide
UV/VIS	SM 4500-CN E-2011	Total Cyanide
UV/VIS	EPA 9012B	Total Cyanide
UV/VIS	EPA 9065	Phenolics
UV/VIS	EPA 9066	Phenolics
IC	EPA 9056A	Fluoride
IC	EPA 9056A	Chloride
IC	EPA 9056A	Sulfate
IC	EPA 9056A	Bromide
IC	EPA 9056A	Nitrate
IC	EPA 9056A	Nitrite
IC	EPA 300.0	Fluoride
IC	EPA 300.0	Chloride
IC	EPA 300.0	Sulfate
IC	EPA 300.0	Bromide
IC	EPA 300.0	Nitrate
IC	EPA 300.0	Nitrite
IC	EPA 218.6	Hexavalent chromium
IC	EPA 7199	Hexavalent chromium
UV/VIS	EPA 7196A	Hexavalent chromium
Discrete Analyzer	SM 3500-Cr B-2011	Hexavalent chromium
Pensky-Martens	EPA 1010A	Ignitability
Electrode	EPA 9040C	Corrosivity
Electrode	SM 4500-H B-2011	Corrosivity

Non-Potable Water		
Technology	**Method**	**Analyte**
Electrode	EPA 120.1	Specific Conductance
Electrode	EPA 180.1	Turbidity
Photometric	Sec. 7.3.3 SW-846	Reactive Cyanide
Titration	Sec. 7.3.4 SW-846	Reactive Sulfide
UV/VIS	EPA 353.2	Nitrate
UV/VIS	EPA 353.2	Nitrite
UV/VIS	EPA 353.2	Nitrate-Nitrite
UV/VIS	EPA 365.1	Phosphorus/ Orthophosphate
Gas Diffusion / UV/VIS	EPA 350.1	Ammonia - N
UV/VIS	EPA 351.2	TKN
UV/VIS	SM 5220D-2011	COD
GC	MADEP-EPH-MOD	Extractable Petroleum Hydrocarbons (EPH) Modified
GC	MADEP-VPH-MOD	Volatile Petroleum Hydrocarbons (VPH) Modified
Preparation	**Method**	**Type**
Organic Preparation	EPA 3520C	Organic Prep. of Water by Continuous Liquid-Liquid
Organic Preparation	EPA 3535A	Solid-Phase Extraction (SPE)
Organic Cleanup	EPA 3620B	Florisil Cleanup Procedure
Organic Cleanup	EPA 3660B	Sulfur Cleanup Procedure
Organic Cleanup	EPA 3665A	Sulfuric Acid Cleanup Procedure
Waste Dilution	EPA 3580A	Waste Dilution
Volatile Organic Preparation	EPA 5030B	Purge-and-Trap for Aqueous Samples
Volatile Organic Preparation	EPA 5035	Closed-System Purge-and-Trap and extraction for Volatile Organics in Soil and Waste Samples
Volatile Organic Preparation	EPA 3585	Waste Dilution for Volatile Organics
Inorganic Preparation	EPA 3005A	Preparation of Waters by Hotblock
Inorganic Preparation	EPA 1311	Toxicity Characteristic Leaching Procedure (TCLP)
Inorganic Metals Preparation	EPA 3010A	Acid Digestion of Aqueous Samples and TCLP/SPLP Extracts for ICP analysis
Inorganic Metals Preparation	EPA 3030C	Acid Digestion of Aqueous Samples for ICP Spectroscopy
Organic Cleanup	EPA 3640	GPC Cleanup Procedure

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC	EPA 8151A	Dalapon
GC	EPA 8151A	Dicamba
GC	EPA 8151A	Dichloroprop
GC	EPA 8151A	MCPP
GC	EPA 8151A	MCPA
GC	EPA 8151A	2,4D
GC	EPA 8151A	Silvex(2,4,5 TP)
GC	EPA 8151A	2,4,5-T
GC	EPA 8151A	2,4DB
GC	EPA 8151A	Dinoseb
GC/MS	EPA 8270E	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270E	1,2,4-Trichlorobenzene
GC/MS	EPA 8270E	1,2-Dichlorobenzene
GC/MS	EPA 8270E	1,3-Dichlorobenzene
GC/MS	EPA 8270E	1,4-Dichlorobenzene
GC/MS	EPA 8270E	1-Methylnaphthalene
GC/MS	EPA 8270E	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270E	2,4,5-Trichlorophenol
GC/MS	EPA 8270E	2,4,6-Trichlorophenol
GC/MS	EPA 8270E	2,4-Dichlorophenol
GC/MS	EPA 8270E	2,4-Dimethylphenol
GC/MS	EPA 8270E	2,4-Dinitrophenol
GC/MS	EPA 8270E	2,4-Dinitrotoluene
GC/MS	EPA 8270E	2,6-Dichlorophenol
GC/MS	EPA 8270E	2,6-Dinitrotoluene
GC/MS	EPA 8270E	2-Chloronaphthalene
GC/MS	EPA 8270E	2-Chlorophenol
GC/MS	EPA 8270E	2-Methylnaphthalene
GC/MS	EPA 8270E	2-Methylphenol
GC/MS	EPA 8270E	2-Nitroaniline
GC/MS	EPA 8270E	2-Nitrophenol
GC/MS	EPA 8270E	3,3'-Dichlorobenzidine
GC/MS	EPA 8270E	3,3'-Dimethylbenzidine
GC/MS	EPA 8270E	3-Nitroaniline
GC/MS	EPA 8270E	4,6-Dinitro-2-methylphenol
GC/MS	EPA 8270E	4-Bromophenylphenylether

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E	4-Chloro-3-methylphenol
GC/MS	EPA 8270E	4-Chloroaniline
GC/MS	EPA 8270E	4-Chlorophenylphenylether
GC/MS	EPA 8270E	4-Nitroaniline
GC/MS	EPA 8270E	4-Nitrophenol
GC/MS	EPA 8270E	Acenaphthene
GC/MS	EPA 8270E	Acenaphthylene
GC/MS	EPA 8270E	Acetophenone
GC/MS	EPA 8270E	Anthracene
GC/MS	EPA 8270E	Atrazine
GC/MS	EPA 8270E	Azobenzene
GC/MS	EPA 8270E	Benzaldhyde
GC/MS	EPA 8270E	Benzidine
GC/MS	EPA 8270E	Benzo(a)Anthracene
GC/MS	EPA 8270E	Benzo(a)pyrene
GC/MS	EPA 8270E	Benzo(b)fluoranthene
GC/MS	EPA 8270E	Benzo(g,h,i)perylene
GC/MS	EPA 8270E	Benzo(k)fluoranthene
GC/MS	EPA 8270E	Benzoic acid
GC/MS	EPA 8270E	Benzyl alcohol
GC/MS	EPA 8270E	Biphenyl
GC/MS	EPA 8270E	bis(2-Chloroethoxy)methane
GC/MS	EPA 8270E	bis(2-Chloroethyl)ether
GCMS	EPA 8270E	Bis (2-Chloro-1-methylethyl) ether
GC/MS	EPA 8270E	bis(2-Ethylhexyl)phthalate
GC/MS	EPA 8270E	Butylbenzylphthalate
GC/MS	EPA 8270E	Caprolactam
GC/MS	EPA 8270E	Carbazole
GC/MS	EPA 8270E	Chrysene
GC/MS	EPA 8270E	DEET
GC/MS	EPA 8270E	Dibenzo(a,h)anthracene
GC/MS	EPA 8270E	Dibenzofuran
GC/MS	EPA 8270E	Diethylphthalate
GC/MS	EPA 8270E	Dimethylphthalate
GC/MS	EPA 8270E	Di-n-butylphthalate
GC/MS	EPA 8270E	Di-n-octylphthalate

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E	Fluoranthene
GC/MS	EPA 8270E	Fluorene
GC/MS	EPA 8270E	Hexachlorobenzene
GC/MS	EPA 8270E	Hexachlorobutadiene
GC/MS	EPA 8270E	Hexachlorocyclopentadiene
GC/MS	EPA 8270E	Hexachloroethane
GC/MS	EPA 8270E	Indeno(1,2,3-c,d)pyrene
GC/MS	EPA 8270E	Isophorone
GC/MS	EPA 8270E	m+p-Cresol
GC/MS	EPA 8270E	Naphthalene
GC/MS	EPA 8270E	Nitrobenzene
GC/MS	EPA 8270E	N-Nitrosodimethylamine
GC/MS	EPA 8270E	n-Nitroso-di-n-propylamine
GC/MS	EPA 8270E	n-Nitrosodiphenylamine
GC/MS	EPA 8270E	N-Nitrosopyrrolidine
GC/MS	EPA 8270E	o-Cresol
GC/MS	EPA 8270E	Pentachlorophenol
GC/MS	EPA 8270E	Phenanthrene
GC/MS	EPA 8270E	Phenol
GC/MS	EPA 8270E	Pyrene
GC/MS	EPA 8270E	Pyridine
GC/MS	EPA 8270E	n-Nitrosopiperidine
GC/MS	EPA 8270E	n-Nitrosomethylethylamine
GC/MS	EPA 8270E	p-Phenylenediamine
GC/MS	EPA 8270E	2-Picoline
GC/MS	EPA 8270E	1-Naphthylamine
GC/MS	EPA 8270E	n-Nitrosodiethylamine
GC/MS	EPA 8270E	n-Nitrosomorpholine
GC/MS	EPA 8270E	p-(Dimethylamino)azobenzene
GC/MS	EPA 8270E	Phenacetin
GC/MS	EPA 8270E	Pentachloronitrobenzene (Quintozene)
GC/MS	EPA 8270E	2-Naphthylamine
GC/MS	EPA 8270E	n-Nitroso-di-n-butylamine
GC/MS	EPA 8270E	4-Aminobiphenyl
GC/MS	EPA 8270E	o-Toluidine
GC/MS	EPA 8270E	5-Nitro-o-toluidine

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E SIM	Acenaphthene
GC/MS	EPA 8270E SIM	Acenaphthylene
GC/MS	EPA 8270E SIM	Anthracene
GC/MS	EPA 8270E SIM	Benzo(a)anthracene
GC/MS	EPA 8270E SIM	Benzo(a)pyrene
GC/MS	EPA 8270E SIM	Benzo(b)fluoranthene
GC/MS	EPA 8270E SIM	Benzo(g,h,i)perylene
GC/MS	EPA 8270E SIM	Benzo(k)fluoranthene
GC/MS	EPA 8270E SIM	Chrysene
GC/MS	EPA 8270E SIM	Dibenz(a,h)anthracene
GC/MS	EPA 8270E SIM	1,4-Dioxane
GC/MS	EPA 8270E SIM	Fluoranthene
GC/MS	EPA 8270E SIM	Fluorene
GC/MS	EPA 8270E SIM	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270E SIM	1-Methylphenol
GC/MS	EPA 8270E SIM	2-Methylphenol
GC/MS	EPA 8270E SIM	Naphthalene
GC/MS	EPA 8270E SIM	Pentachlorophenol
GC/MS	EPA 8270E SIM	Phenanthrene
GC/MS	EPA 8270E SIM	Pyrene
GC/MS	EPA 8260D	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260D	1,1,1-Trichloroethane
GC/MS	EPA 8260D	1,1,2,2-Tetrachloroethene
GC/MS	EPA 8260D	1,1,2-Trichloroethane
GC/MS	EPA 8260D	1,1-Dichloroethane
GC/MS	EPA 8260D	1,1-Dichloroethene
GC/MS	EPA 8260D	1,1-Dichloropropene
GC/MS	EPA 8260D	1,2,3-Trichlorobenzene
GC/MS	EPA 8260D	1,2,3-Trichloropropane
GC/MS	EPA 8260D	1,2,4-Trichlorobenzene
GC/MS	EPA 8260D	1,2,4-Trimethylbenzene
GC/MS	EPA 8260D	1,2-Dibromo-3-chloropropane
GC/MS	EPA 8260D	1,2-Dibromoethane (EDB)
GC/MS	EPA 8260D	1,2-Dichlorobenzene
GC/MS	EPA 8260D	1,2-Dichloroethane
GC/MS	EPA 8260D	1,2-Dichloropropane

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260D	1,3,5-Trimethylbenzene
GC/MS	EPA 8260D	1,3-Dichlorobenzene
GC/MS	EPA 8260D	1,3-Dichloropropane
GC/MS	EPA 8260D	1,4-Dichlorobenzene
GC/MS	EPA 8260D	1,4-Dioxane
GC/MS	EPA 8260D	2,2-Dichloropropane
GC/MS	EPA 8260D	2-Butanone (MEK)
GC/MS	EPA 8260D	2-Chloroethylvinyl ether
GC/MS	EPA 8260D	2-Chlorotoluene
GC/MS	EPA 8260D	2-Hexanone
GC/MS	EPA 8260D	4-Chlorotoluene
GC/MS	EPA 8260D	4-Methyl-2-pentanone
GC/MS	EPA 8260D	Acetone
GC/MS	EPA 8260D	Acetonitrile
GC/MS	EPA 8260D	Acrolein
GC/MS	EPA 8260D	Acrylonitrile
GC/MS	EPA 8260D	Allyl chloride
GC/MS	EPA 8260D	Benzene
GC/MS	EPA 8260D	Benzyl chloride
GC/MS	EPA 8260D	Bromobenzene
GC/MS	EPA 8260D	Bromochloromethane
GC/MS	EPA 8260D	Bromodichloromethane
GC/MS	EPA 8260D	Bromoform
GC/MS	EPA 8260D	Bromomethane
GC/MS	EPA 8260D	Carbon disulfide
GC/MS	EPA 8260D	Carbon tetrachloride
GC/MS	EPA 8260D	Chlorobenzene
GC/MS	EPA 8260D	Chloroethane
GC/MS	EPA 8260D	Chloroform
GC/MS	EPA 8260D	Chloromethane
GC/MS	EPA 8260D	Chloroprene
GC/MS	EPA 8260D	cis-1,2-Dichloroethene
GC/MS	EPA 8260D	cis-1,3-Dichloropropene
GC/MS	EPA 8260D	Cyclohexane
GC/MS	EPA 8260D	Cyclohexanone
GC/MS	EPA 8260D	Dibromochloromethane

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260D	Dibromomethane
GC/MS	EPA 8260D	Dichlorodifluoromethane
GC/MS	EPA 8260D	Diisopropyl ether (IPE)
GC/MS	EPA 8260D	Ethyl ether
GC/MS	EPA 8260D	Ethyl methacrylate
GC/MS	EPA 8260D	Ethylbenzene
GC/MS	EPA 8260D	Freon 113
GC/MS	EPA 8260D	Hexachlorobutadiene
GC/MS	EPA 8260D	Isobutyl alcohol
GC/MS	EPA 8260D	Isopropylbenzene
GC/MS	EPA 8260D	m+p-Xylenes
GC/MS	EPA 8260D	Methacrylonitrile
GC/MS	EPA 8260D	Methyl Acetate
GC/MS	EPA 8260D	Methyl iodide
GC/MS	EPA 8260D	Methyl methacrylate
GC/MS	EPA 8260D	Methyl tertiary butyl ether (MTBE)
GC/MS	EPA 8260D	Methylcyclohexane
GC/MS	EPA 8260D	Methylene chloride
GC/MS	EPA 8260D	Naphthalene
GC/MS	EPA 8260D	n-Butylbenzene
GC/MS	EPA 8260D	n-Propylbenzene
GC/MS	EPA 8260D	o-Xylene
GC/MS	EPA 8260D	Pentachloroethane
GC/MS	EPA 8260D	p-Isopropyltoluene
GC/MS	EPA 8260D	Propionitrile
GC/MS	EPA 8260D	sec-Butylbenzene
GC/MS	EPA 8260D	Styrene
GC/MS	EPA 8260D	tert-Butylbenzene
GC/MS	EPA 8260D	Tetrachloroethene
GC/MS	EPA 8260D	Tetrahydrofuran
GC/MS	EPA 8260D	Toluene
GC/MS	EPA 8260D	Total Xylenes
GC/MS	EPA 8260D	trans-1,2-Dichloroethene
GC/MS	EPA 8260D	trans-1,3-Dichloropropene
GC/MS	EPA 8260D	trans-1,4-Dichloro-2-butene
GC/MS	EPA 8260D	Trichloroethene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260D	Trichlorofluoromethane
GC/MS	EPA 8260D	Vinyl acetate
GC/MS	EPA 8260D	Vinyl chloride
GC/MS	EPA 8260C SIM	1,4-Dioxane
GC	EPA 8081B	4,4'-DDD
GC	EPA 8081B	4,4'-DDE
GC	EPA 8081B	4,4'-DDT
GC	EPA 8081B	Aldrin
GC	EPA 8081B	alpha-BHC
GC	EPA 8081B	alpha-Chlordane
GC	EPA 8081B	beta-BHC
GC	EPA 8081B	Chlordane
GC	EPA 8081B	delta-BHC
GC	EPA 8081B	Dieldrin
GC	EPA 8081B	Endosulfan I
GC	EPA 8081B	Endosulfan II
GC	EPA 8081B	Endosulfan sulfate
GC	EPA 8081B	Endrin
GC	EPA 8081B	Endrin Aldehyde
GC	EPA 8081B	Endrin Ketone
GC	EPA 8081B	gamma-BHC (Lindane)
GC	EPA 8081B	gamma-Chlordane
GC	EPA 8081B	Heptachlor
GC	EPA 8081B	Heptachlor Epoxide
GC	EPA 8081B	Methoxychlor
GC	EPA 8081B	Mirex
GC	EPA 8081B	Toxaphene
GC	EPA 8082A	Aroclor 1016
GC	EPA 8082A	Aroclor 1221
GC	EPA 8082A	Aroclor 1232
GC	EPA 8082A	Aroclor 1242
GC	EPA 8082A	Aroclor 1248
GC	EPA 8082A	Aroclor 1254
GC	EPA 8082A	Aroclor 1260
GC	EPA 8082A	Aroclor 1262
GC	EPA 8082A	Aroclor 1268

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
HPLC	EPA 8330A	2-Amino-4,6-dinitrotoluene
HPLC	EPA 8330A	4-Amino-2,6-dinitrotoluene
HPLC	EPA 8330A	1,3-Dinitrobenzene
HPLC	EPA 8330A	2,4-Dinitrotoluene
HPLC	EPA 8330A	2,6-Dinitrotoluene
HPLC	EPA 8330A	HMX
HPLC	EPA 8330A	Nitrobenzene
HPLC	EPA 8330A	2-Nitrotoluene
HPLC	EPA 8330A	3-Nitrotoluene
HPLC	EPA 8330A	4-Nitrotoluene
HPLC	EPA 8330A	RDX
HPLC	EPA 8330A	Tetryl
HPLC	EPA 8330A	1,3,5-Trinitrobenzene
HPLC	EPA 8330A	2,4,6-Trinitrotoluene
GC	EPA 8015C	Diesel Range Organics
GC	EPA 8015C	WI-Diesel Range Organics
GC	EPA 8015C	Gasoline Range Organics
GC	EPA 8015C	WI-Gasoline Range Organics
GC	FL-PRO	FL-PRO
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-ethylperfluoro-1-octanesulfonamidoacetic acid (EtFOSAA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Fluorotelomer sulfonate 8:2 [1H,1H,2H,2H-perfluorodecane sulfonate] (8:2 FTS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Fluorotelomer sulfonate 4:2 [1H,1H,2H,2H-perfluorohexane sulfonate] (4:2 FTS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Fluorotelomer sulfonate 6:2 [1H,1H,2H,2H-perfluorooctane sulfonate] (6:2 FTS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	GenX (HFPO-DA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-methylperfluoro-1-octanesulfonamidoacetic acid (MeFOSAA)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-1-butanesulfonate (PFBS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-n-butanoic acid (PFBA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-1-decanesulfonate (PFDS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-n-decanoic acid (PFDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-n-dodecanoic acid (PFDoA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-1-heptanesulfonate (PFHpS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-n-heptanoic acid (PFHpA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-1-hexanesulfonate (PFHxS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-n-hexanoic acid (PFHxA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-1-nonanesulfonate (PFNS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-n-nonanoic acid (PFNA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorooctanesulfonate (PFOS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-1-octanesulfonamide (PFOSA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-n-octanoic acid (PFOA)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-n-pentanoic acid (PFPeA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-1-pentansulfonate (PFPeS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-n-tetradecanoic acid (PFTeDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-n-tridecanoic acid (PFTrDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-n-undecanoic acid (PFUDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-methylperfluoro-1-octanesulfonamide (MeFOSA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-ethylperfluoro-1-octanesulfonamide (EtFOSA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	1H,1H,2H,2H-perfluorododecane sulfonate (10:2FTS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-Ethyl Perfluorooctane sulfonamido ethanol (EtFOSE)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-Methyl Perfluorooctane sulfonamido ethanol (MeFOSE)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Sodium dodecafluoro-3H-4, 8-dioxanonanoate (NaDONA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	9Cl-PF3ONS
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	11Cl-PF3OUDS
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorododecanesulfonic acid (PFDOS)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorohexadecanoic acid (PFHxDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorooctadecanoic acid (PFODA)
HPLC	EPA 8330B MOD	2-Amino-4,6-dinitrotoluene
HPLC	EPA 8330B MOD	4-Amino-2,6-dinitrotoluene
HPLC	EPA 8330B MOD	1,3-Dinitrobenzene
HPLC	EPA 8330B MOD	2,4-Dinitrotoluene
HPLC	EPA 8330B MOD	2,6-Dinitrotoluene
HPLC	EPA 8330B MOD	HMX
HPLC	EPA 8330B MOD	Nitrobenzene
HPLC	EPA 8330B MOD	Nitrocellulose
HPLC	EPA 8330B MOD	Nitroguanidine
HPLC	EPA 8330B MOD	Guanidine nitrate
HPLC	EPA 8330B MOD	Nitroglycerin (NG)
HPLC	EPA 8330B MOD	2-Nitrotoluene
HPLC	EPA 8330B MOD	3-Nitrotoluene
HPLC	EPA 8330B MOD	4-Nitrotoluene
HPLC	EPA 8330B MOD	RDX
HPLC	EPA 8330B MOD	Tetryl
HPLC	EPA 8330B MOD	1,3,5-Trinitrobenzene
HPLC	EPA 8330B MOD	2,4,6-Trinitrotoluene
ICP	EPA 6010D	Aluminum
ICP	EPA 6010D	Antimony
ICP	EPA 6010D	Arsenic
ICP	EPA 6010D	Barium
ICP	EPA 6010D	Beryllium
ICP	EPA 6010D	Cadmium
ICP	EPA 6010D	Calcium
ICP	EPA 6010D	Chromium
ICP	EPA 6010D	Cobalt
ICP	EPA 6010D	Copper
ICP	EPA 6010D	Iron
ICP	EPA 6010D	Lead
ICP	EPA 6010D	Magnesium

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP	EPA 6010D	Manganese
ICP	EPA 6010D	Molybdenum
ICP	EPA 6010D	Nickel
ICP	EPA 6010D	Potassium
ICP	EPA 6010D	Selenium
ICP	EPA 6010D	Silver
ICP	EPA 6010D	Sodium
ICP	EPA 6010D	Thallium
ICP	EPA 6010D	Tin
ICP	EPA 6010D	Vanadium
ICP	EPA 6010D	Zinc
ICP/MS	EPA 6020B	Aluminum
ICP/MS	EPA 6020B	Antimony
ICP/MS	EPA 6020B	Arsenic
ICP/MS	EPA 6020B	Barium
ICP/MS	EPA 6020B	Beryllium
ICP/MS	EPA 6020B	Cadmium
ICP/MS	EPA 6020B	Calcium
ICP/MS	EPA 6020B	Chromium
ICP/MS	EPA 6020B	Cobalt
ICP/MS	EPA 6020B	Copper
ICP/MS	EPA 6020B	Iron
ICP/MS	EPA 6020B	Lead
ICP/MS	EPA 6020B	Magnesium
ICP/MS	EPA 6020B	Manganese
ICP/MS	EPA 6020B	Nickel
ICP/MS	EPA 6020B	Potassium
ICP/MS	EPA 6020B	Selenium
ICP/MS	EPA 6020B	Silver
ICP/MS	EPA 6020B	Sodium
ICP/MS	EPA 6020B	Thallium
ICP/MS	EPA 6020B	Vanadium
ICP/MS	EPA 6020B	Zinc
CVAA	EPA 7471B	Mercury
Titration	Walkley-Black	TOC
UV/VIS	EPA 9012B	Total Cyanide

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
UV/VIS	EPA 9065	Phenolics
UV/VIS	EPA 9066	Phenolics
IC	EPA 9056A	Bromide
IC	EPA 9056A	Chloride
IC	EPA 9056A	Fluoride
IC	EPA 9056A	Sulfate
IC	EPA 9056A	Nitrate
IC	EPA 9056A	Nitrite
IC	EPA 300.0	Fluoride
IC	EPA 300.0	Chloride
IC	EPA 300.0	Sulfate
IC	EPA 300.0	Bromide
IC	EPA 300.0	Nitrate
IC	EPA 300.0	Nitrite
IC	EPA 7199 / EPA 3060A	Hexavalent chromium
UV/VIS	EPA 7196A EPA 3060A	Hexavalent chromium
Pensky-Martens	EPA 1010A	Ignitability
Electrode	EPA 9045D	Corrosivity
Photometric	Sec. 7.3.3 SW-846	Reactive Cyanide
Titration	Sec. 7.3.4 SW-846	Reactive Sulfide
Filtration	EPA 9095B	Paint Filter Test
UV/VIS	EPA 365.1 MOD	Phosphorus
UV/VIS	EPA 353.2 MOD	Nitrate
UV/VIS	EPA 353.2 MOD	Nitrite
UV/VIS	EPA 353.2 MOD	Nitrate-Nitrite
UV/VIS	EPA 350.1	Ammonia
Gas Diffusion / UV/VIS	EPA 350.1	Ammonia - N
UV/VIS	EPA 351.2 MOD	TKN
GC	MADEP-EPH MOD	Extractable Petroleum Hydrocarbons (EPH) Modified
GC	MADEP-VPH-MOD	Volatile Petroleum Hydrocarbons (VPH) Modified
Preparation	**Method**	**Type**
Organic Preparation	EPA 3550C	Preparation of Soil by Sonication
Organic Preparation	EPA 3546	Microwave Extraction
Organic Cleanup	EPA 3620B	Florisil Cleanup Procedure
Organic Cleanup	EPA 3660B	Sulfur Cleanup Procedure

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Organic Cleanup	EPA 3665A	Sulfuric Acid Cleanup Procedure
Organic Cleanup	EPA 3640C	GPC Cleanup Procedure
Volatile Organic Preparation	EPA 5035	Closed-System Purge-and-Trap and extraction for Volatile Organics in Soil and Waste Samples
Volatile Organic Preparation	EPA 3585	Waste Dilution for Volatile Organics
Waste Dilution	EPA 3580A	Waste Dilution
Inorganic Preparation	EPA 3050B	Preparation of Soils by Hotblock
Inorganic Preparation	EPA 1311	Toxicity Characteristic Leaching Procedure (TCLP)
Inorganic Preparation	EPA 1312	Synthetic Precipitation Leaching Procedure (SPLP)
Inorganic Metals Preparation	EPA 3010A	Acid Digestion of Aqueous Samples and TCLP/SPLP Extracts

Biological Tissue		
Technology	**Method**	**Analyte**
GC	EPA 8081B	4,4'-DDE
GC	EPA 8081B	4,4'-DDT
GC	EPA 8081B	Aldrin
GC	EPA 8081B	alpha-BHC
GC	EPA 8081B	alpha-Chlordane
GC	EPA 8081B	beta-BHC
GC	EPA 8081B	Chlordane
GC	EPA 8081B	delta-BHC
GC	EPA 8081B	Dieldrin
GC	EPA 8081B	Endosulfan I
GC	EPA 8081B	Endosulfan II
GC	EPA 8081B	Endosulfan sulfate
GC	EPA 8081B	Endrin
GC	EPA 8081B	Endrin Aldehyde
GC	EPA 8081B	Endrin Ketone
GC	EPA 8081B	gamma-BHC (Lindane)
GC	EPA 8081B	gamma-Chlordane
GC	EPA 8081B	Heptachlor
GC	EPA 8081B	Heptachlor Epoxide
GC	EPA 8081B	Methoxychlor
GC	EPA 8081B	Toxaphene
GC	EPA 8082A	Aroclor 1016
GC	EPA 8082A	Aroclor 1221

Biological Tissue		
Technology	**Method**	**Analyte**
GC	EPA 8082A	Aroclor 1232
GC	EPA 8082A	Aroclor 1242
GC	EPA 8082A	Aroclor 1248
GC	EPA 8082A	Aroclor 1254
GC	EPA 8082A	Aroclor 1260
GC	EPA 8082A	Aroclor 1262
GC	EPA 8082A	Aroclor 1268
GC/MS	EPA 8270E	1,1'-Biphenyl
GC/MS	EPA 8270E	1-Methylnaphthalene
GC/MS	EPA 8270E	2-Methylnaphthalene
GC/MS	EPA 8270E	Acenaphthene
GC/MS	EPA 8270E	Acenaphthylene
GC/MS	EPA 8270E	Anthracene
GC/MS	EPA 8270E	Atrazine
GC/MS	EPA 8270E	Benzo(a)anthracene
GC/MS	EPA 8270E	Benzo(b)fluoranthene
GC/MS	EPA 8270E	Benzo(g,h,i)perylene
GC/MS	EPA 8270E	Benzo(k)fluoranthene
GC/MS	EPA 8270E	Chrysene
GC/MS	EPA 8270E	Dibenzo(a,h)anthracene
GC/MS	EPA 8270E	Dibenzofuran
GC/MS	EPA 8270E	Dimethoate
GC/MS	EPA 8270E	Disulfoton
GC/MS	EPA 8270E	Fluoranthene
GC/MS	EPA 8270E	Fluorene
GC/MS	EPA 8270E	Hexachlorobenzene
GC/MS	EPA 8270E	Indeno(1,2,3-c,d)pyrene
GC/MS	EPA 8270E	Methyl parathion
GC/MS	EPA 8270E	Naphthalene
GC/MS	EPA 8270E	Phenanthrene
GC/MS	EPA 8270E	Phorate
GC/MS	EPA 8270E	Pronamide
GC/MS	EPA 8270E	Pyrene
Preparation	**Method**	**Type**
Organic Preparation	EPA 3540C	Soxhlet Extraction

Drinking water		
Technology	**Method**	**Analyte**
GC/MS	EPA 524.2	1,1,1,2-Tetrachloroethane
GC/MS	EPA 524.2	1,1,1-Trichloroethane
GC/MS	EPA 524.2	1,1,2,2-Tetrachloroethane
GC/MS	EPA 524.2	1,1,2-Trichloroethane
GC/MS	EPA 524.2	1,1-Dichloroethane
GC/MS	EPA 524.2	1,1-Dichloroethene
GC/MS	EPA 524.2	1,1-Dichloropropene
GC/MS	EPA 524.2	1,2,3-Trichlorobenzene
GC/MS	EPA 524.2	1,2,3-Trichloropropane
GC/MS	EPA 524.2	1,2,4-Trichlorobenzene
GC/MS	EPA 524.2	1,2,4-Trimethylbenzene
GC/MS	EPA 524.2	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	EPA 524.2	1,2-Dibromoethane (EDB)
GC/MS	EPA 524.2	1,2-Dichlorobenzene
GC/MS	EPA 524.2	1,2-Dichloroethane
GC/MS	EPA 524.2	1,2-Dichloropropane
GC/MS	EPA 524.2	1,3,5-Trimethylbenzene
GC/MS	EPA 524.2	1,3-Dichlorobenzene
GC/MS	EPA 524.2	1,3-Dichloropropane
GC/MS	EPA 524.2	1,4-Dichlorobenzene
GC/MS	EPA 524.2	2,2-Dichloropropane
GC/MS	EPA 524.2	2-Butanone (MEK)
GC/MS	EPA 524.2	2-Chlorotoluene
GC/MS	EPA 524.2	2-Hexanone
GC/MS	EPA 524.2	2-Nitropropane
GC/MS	EPA 524.2	3-Chloropropene (Allyl chloride)
GC/MS	EPA 524.2	4-Chlorotoluene
GC/MS	EPA 524.2	4-Methyl-2-pentanone
GC/MS	EPA 524.2	Acetone
GC/MS	EPA 524.2	Acrylonitrile
GC/MS	EPA 524.2	Benzene
GC/MS	EPA 524.2	Bromobenzene
GC/MS	EPA 524.2	Bromochloromethane
GC/MS	EPA 524.2	Bromodichloromethane
GC/MS	EPA 524.2	Bromoform
GC/MS	EPA 524.2	Bromomethane (Methyl bromide)
GC/MS	EPA 524.2	Carbon disulfide
GC/MS	EPA 524.2	Carbon tetrachloride

Drinking water		
Technology	**Method**	**Analyte**
GC/MS	EPA 524.2	Chlorobenzene
GC/MS	EPA 524.2	Chloroethane
GC/MS	EPA 524.2	Chloroform
GC/MS	EPA 524.2	Chloromethane (Methyl chloride)
GC/MS	EPA 524.2	cis-1,2-Dichloroethene
GC/MS	EPA 524.2	cis-1,3-Dichloropropene
GC/MS	EPA 524.2	Cyclohexane
GC/MS	EPA 524.2	Dibromochloromethane
GC/MS	EPA 524.2	Dibromomethane (Methylene bromide)
GC/MS	EPA 524.2	Dichlorodifluoromethane
GC/MS	EPA 524.2	Ethyl ether
GC/MS	EPA 524.2	Ethyl methacrylate
GC/MS	EPA 524.2	Ethylbenzene
GC/MS	EPA 524.2	Hexachlorobutadiene
GC/MS	EPA 524.2	Isopropylbenzene
GC/MS	EPA 524.2	m+p - Xylenes
GC/MS	EPA 524.2	Methacrylonitrile
GC/MS	EPA 524.2	Methyl iodide (Iodomethane)
GC/MS	EPA 524.2	Methyl methacrylate
GC/MS	EPA 524.2	Methyl tertiary butyl ether (MTBE)
GC/MS	EPA 524.2	Methylene chloride
GC/MS	EPA 524.2	Naphthalene
GC/MS	EPA 524.2	n-Butylbenzene
GC/MS	EPA 524.2	n-Propylbenzene
GC/MS	EPA 524.2	o - Xylenes
GC/MS	EPA 524.2	Pentachloroethane
GC/MS	EPA 524.2	p-Isopropyltoluene
GC/MS	EPA 524.2	Propionitrile (Ethyl cyanide)
GC/MS	EPA 524.2	sec-Butylbenzene
GC/MS	EPA 524.2	Styrene
GC/MS	EPA 524.2	tert-Butylbenzene
GC/MS	EPA 524.2	Tetrachloroethene
GC/MS	EPA 524.2	Toluene
GC/MS	EPA 524.2	trans-1,2-Dichloroethene
GC/MS	EPA 524.2	trans-1,3-Dichloropropene
GC/MS	EPA 524.2	trans-1,4-Dichloro-2-butene

Drinking water		
Technology	**Method**	**Analyte**
GC/MS	EPA 524.2	Trichloroethene
GC/MS	EPA 524.2	Trichlorofluoromethane
GC/MS	EPA 524.2	Vinyl chloride
GC/MS	EPA 524.2	Xylenes (total)
LC/MS/MS	EPA 537.1	N-ethylperfluoro-1-octanesulfonamidoacetic acid (EtFOSAA)
LC/MS/MS	EPA 537.1	N-methylperfluoro-1-octanesulfonamidoacetic acid (MeFOSAA)
LC/MS/MS	EPA 537.1	Perfluoro-1-butanesulfonate (PFBS)
LC/MS/MS	EPA 537.1	Perfluoro-n-decanoic acid (PFDA)
LC/MS/MS	EPA 537.1	Perfluoro-n-dodecanoic acid (PFDoA)
LC/MS/MS	EPA 537.1	Perfluoro-n-heptanoic acid (PFHpA)
LC/MS/MS	EPA 537.1	Perfluoro-1-hexanesulfonate (PFHxS)
LC/MS/MS	EPA 537.1	Perfluoro-n-hexanoic acid (PFHxA)
LC/MS/MS	EPA 537.1	Perfluoro-n-nonanoic acid (PFNA)
LC/MS/MS	EPA 537.1	Perfluorooctanesulfonate (PFOS)
LC/MS/MS	EPA 537.1	Perfluoro-n-octanoic acid (PFOA)
LC/MS/MS	EPA 537.1	Perfluoro-n-tetradecanoic acid (PFTeDA)
LC/MS/MS	EPA 537.1	Perfluoro-n-tridecanoic acid (PFTrDA)
LC/MS/MS	EPA 537.1	Perfluoro-n-undecanoic acid (PFUDA)
LC/MS/MS	EPA 537.1	NaDONA
LC/MS/MS	EPA 537.1	GenX
LC/MS/MS	EPA 537.1	9CL-PF3ONS
LC/MS/MS	EPA 537.1	11CL-PF3OUDS
LC/MS/MS	EPA 533	9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS)
LC/MS/MS	EPA 533	11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)
LC/MS/MS	EPA 533	1H, 1H, 2H, 2H-perfluorodecane sulfonic acid (8:2 FTS)
LC/MS/MS	EPA 533	1H, 1H, 2H, 2H-perfluorooctane sulfonic acid (6:2 FTS)
LC/MS/MS	EPA 533	1H, 1H, 2H, 2H-perfluorohexane sulfonic acid (4:2 FTS)
LC/MS/MS	EPA 533	Hexafluoropropylene oxide dimer acid (GenX)
LC/MS/MS	EPA 533	4,8-dioxa-3H-perfluorononanoic acid (ADONA)
LC/MS/MS	EPA 533	Nonafluoro-3,6-dioxaheptanoic acid (NFDHA)
LC/MS/MS	EPA 533	Perfluoro(2-ehtoxyethane)sulfonic acid (PFEESA)
LC/MS/MS	EPA 533	Perfluorobutanesulfonic acid (PFBS)
LC/MS/MS	EPA 533	Perfluoroheptanesulfonic acid (PFHpS)

Drinking water		
Technology	**Method**	**Analyte**
LC/MS/MS	EPA 533	Perfluoropentanesulfonic acid (PFPeS)
LC/MS/MS	EPA 533	Perfluoro-3-methoxypropanoic acid (PFMPA)
LC/MS/MS	EPA 533	Perfluoro-4-methoxybutanoic acid (PFMBA)
LC/MS/MS	EPA 533	Perfluorohexanesulfonic acid (PFHxS)
LC/MS/MS	EPA 533	Perfluorobutanoic acid (PFBA)
LC/MS/MS	EPA 533	Perfluorodecanoic acid (PFDA)
LC/MS/MS	EPA 533	Perfluorododecanoic acid (PFDoA)
LC/MS/MS	EPA 533	Perfluoroheptanoic acid (PFHpA)
LC/MS/MS	EPA 533	Perfluorohexanoic acid (PFHxA)
LC/MS/MS	EPA 533	Perfluorononanoic acid (PFNA)
LC/MS/MS	EPA 533	Perfluorooctanoic acid (PFOA)
LC/MS/MS	EPA 533	Perfluoropentanoic acid (PFPeA)
LC/MS/MS	EPA 533	Perfluoroundecanoic acid (PFUdA)
LC/MS/MS	EPA 533	Perfluorooctanesulfonic acid (PFOS)

Note:

1. This scope is formatted as part of a single document including Certificate of Accreditation No. L2224.

R. Douglas Leonard Jr., VP, PILR SBU

APPENDIX C

LABORATORY STANDARD OPERATING PROCEDURES

Document Information

Document Number: ME0012X **Revision: -22**

Document Title: GC/MS Volatiles Analysis

Department(s): |Volatiles|

Date Information

Effective Date: Thursday, September 23, 2021

Notes

Document Notes:

All Dates and Times are in Eastern Standard Time Zone.

Signature Manifest

Document Number: ME0012X **Revision:** -22

Title: GC/MS Volatiles Analysis

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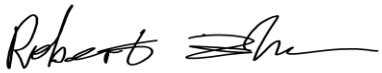
ME0012X-22

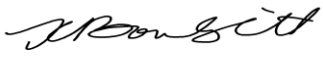
9/21/2021 2:51:40 PM
Daniel J. Wright
General Manager 1

9/21/2021 9:16:13 AM
Kelly M. Nance
Quality Manager



9/22/2021 1:50:41 PM
Robert Zhu
Technical Specialist

9/22/2021 2:08:11 PM
Bradley E. Belding
Operations Manager



9/21/2021 9:17:05 AM
Kristina P. Bouknight
Environmental Health and Radiation Safety Officer

9/20/2021 5:51:05 PM
Erin C. Boyd
Supervisor

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1.0 Scope and Application

This standard operating procedure (SOP) describes the laboratory procedure for the determination of volatile organic compounds by Gas Chromatography/Mass Spectrometry.

This method is based upon SW-846 8260B. In addition, this method meets the requirements outlined in SW-846 8260C, SW-846 8260D, Standard Methods 6200 B-2011 and for analysis of wastewater following EPA Method 624.1. Refer to the appendices at the end of the SOP for 624.1, SM6200 B-2011,8260C and 8260D requirements. Refer to Table A-I for the list of compounds applicable for this method. Additional compounds may be amenable to this method. If non-standard analytes are required, they must be validated by the procedures described in Section 11.4.1.2 before sample analysis.

M-xylene cannot be separated from p-xylene by the conditions specified in this method.

Note: Numerous reference methods are covered under this SOP, the current practice will be in most cases to follow the most stringent criteria.

1.1 Target Analyte List and Limits of Quantitation (LOQ)

The limit of quantitation (LOQ) for determining an individual compound is approximately 5.0 µg/Kg (wet weight) for low level soil/sediment samples, 250 µg/kg for high level soil/sediment, 2500 µg/kg for wastes (dependent of matrix), and 5.0 µg/L for aqueous samples. Some compounds have higher reporting limits. For projects requiring lower LOQs, Shealy can supply a lower calibration that will reach as low as 0.5 µg/L for most compounds. The target analytes and the standarde LOQ that can be achieved with this procedure are provided in Table A-I, Appendix A.

LOQ are established in accordance with Pace policy and SOPs for method validation and for the determination of detection limits (DL) and quantitation limits (LOQ). DL and LOQ are routinely verified and updated when needed. The current LOQ for each target analyte that can be determined by this SOP as of the effective date of this SOP is provided in Table 1, Appendix A.

The reporting limit (RL) is the value to which analytes are reported as detected or not detected in the final report. When the RL is less than the lower limit of quantitation (LLOQ), all detects and non-detects at the RL are qualitative. The LLOQ is the lowest point of the calibration curve used for each target analyte.

DL, LOQ, and RL are always adjusted to account for actual amounts used and for dilution.

1.2 Applicable Matrices

This method is applicable to the determination of the concentration of volatile organic compounds in solid, non-aqueous, and aqueous matrices

2.0 Summary of Method

Volatile compounds are purged by helium gas from aqueous and soil/sediment samples and concentrated onto a trap. The trap is then heated rapidly, and all the volatile compounds are transferred into GC inlet and analyzed by GC/MS. Qualitative identification of the target compounds is performed using the retention time and the relative abundance of characteristic ions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

3.0 Interferences

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. If interference is detected, it is necessary to determine if the source of interference is in the preparation and/or cleanup of the samples; then take corrective action to eliminate the problem.

3.2 Volatile preparation and analysis should be physically separated from the laboratory area where target solvents are used. Air supply for the volatiles should provide positive pressure relative to other laboratory areas.

3.3 Samples can be contaminated by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during shipment and storage. Protect samples from sources of volatiles during collection, shipment, and storage. A reagent water field blank carried through sampling and analysis can serve as a check on such contamination.

3.4 The use of high purity reagents, solvents, and gases helps to minimize interference problems.

3.5 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample.

3.6 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sampling system and the concentrator must be cleaned thoroughly between samples. Whenever an unusually concentrated sample is encountered, it should be followed, if possible, by the analysis of a blank to check for cross contamination. The sample immediately following the unusually concentrated sample may also be used to check for cross-contamination.

3.7 Acetone and methylene chloride contamination is commonly observed in this analysis and its occurrence must be carefully evaluated as an indicator of a contamination problem in the sample preparation step of the analysis.

4.0 Definitions

Refer to the *Laboratory Quality Manual* [QAMP ME0012K] for a glossary of common lab terms and definitions.

4.1 Non-conformance Memo (NCM) - A form used to document a non-conforming event. An analyst must document a non-conformance memo when a non-conforming event occurs. A non-conforming event may include the reporting of analytical data outside of method or SOP criteria, or when there is a deviation from a written policy or procedure. Information in an NCM may be used by project managers to flag data in the report narrative, or by the quality department to track trends and initiate corrective actions, where applicable. Additional information on the NCM policy and procedure is located in the *Complaints and Nonconformances* SOP [QA SOP ME001BO].

4.2 Calibration Check Compounds (CCC) – A subset of target compounds used to evaluate the calibration stability of the GC/MS system. A maximum percent deviation of the CCC's is specified for calibration acceptance.

4.3 System Performance Check Compounds (SPCC) – Target compounds designated to monitor chromatographic performance, sensitivity, and compound instability or degradation on active sites. Minimum response factors are specified for acceptable performance.

4.4 Marginal Exceedance (ME) – A marginal exceedance is defined as being beyond the LCS control limit (method defined or three standard deviations), but within the ME limits. ME limits are between three and four standards deviations around the mean.

5.0 Health and Safety

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of

solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

6.0 Sample Collection, Preservation, Holding Time, and Storage

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory performs samples collection for samples to be analyzed by this SOP in accordance with the *Field Services* SOP [FS SOP ME001BS]. Refer to this SOP for these instructions.

The laboratory will provide containers for the collection of samples upon client request for analytical services. Bottle kits are prepared in accordance with the *Sample Container Shipping* SOP [AD SOP ME001DS].

Requirements for container type, preservation, and field quality control (QC) for the common list of test methods offered by Pace are listed in the *Pace-WCOL Analytical Methods List* [ME002BS].

General Requirements

Aqueous Sample Collection and Preservation

Aqueous samples are collected in three 40-mL volatile sample vials preserved with hydrochloric acid ($Na_2S_2O_3$ if chlorinated) and stored at 4 ± 2°C.

If 2-Chloroethyl vinyl ether is an analyte of interest, another set of three 40-mL volatile vials must be collected without preservative and stored at 4 ± 2°C. The compound 2-Chloroethyl vinyl ether breaks down in the presence of acid, and therefore, cannot be reported from a preserved sample.

If acrolein and acrylonitrile are analytes of interest, samples must be analyzed from an unpreserved via and stored at 4 ± 2°C.

Samples must be checked for presence of air bubbles. The size of any bubble caused by degassing upon cooling the sample must not exceed 5 – 6 mm. When a bubble is present, observe the cap and septum to ensure that a proper seal was made at the time of sampling. Document presence of air bubbles (> 5-6 mm) using an NCM.

Samples requiring 1,4-Dioxane by method 624.1 must be submitted unpreserved.

Solid Sample Collection and Preservation

Solid samples collected using Terra Core® **or equivalent soil kit:**

Use the Terra Core® or equivalent sampling kit in to collect approximately 5 grams of soil sample into 2 pre-weighed 40-mL vials each containing reagent water and a stir bar, 1 pre-weighed 40-mL vial containing 5 mL of methanol preservative, and 1 empty (un-preserved) pre-weighed 40-mL vial. Tightly

cap all the vials. In addition, solid samples are collected in a 2 oz glass container for dry weight determination. The 40-mL vials containing solid samples and reagent water must be stored in a freezer in which temperature is less than -10°C within 48 hours of collection for preservation. To avoid potential vial breakage, store the vials horizontally in freezer until frozen, at which time vials may be placed upright in boxes. After samples are frozen, boxes may be stacked on top of each other to conserve space in the freezer. The solid samples preserved in methanol, in unpreserved vials, and those in the 2 oz containers must be stored under 4 ± 2°C.

Solid Samples Collected in En Core® Sampler:

Two En Core® samplers should be collected for low level analysis. An additional sample is collected for potential high-level analysis. The samplers must be sealed immediately after collection. The samples must be preserved in the laboratory as follows within 48 hours of collection.

Weigh two 40-mL VOC vials each containing 5.0 mL of reagent water and a magnetic stir bar to the nearest 0.01 gram. Open the sampler and carefully transfer the solid sample into the vial. Cap the vial. Re-weigh the vial. Record the weight to the nearest 0.01 g. The difference between the second and first weight is the sample weight. Record the weight in a logbook. The samples must be analyzed within 48 hours of preservation or the vials must be stored horizontally in a freezer within 48 hours of collection. Note: Care must be taken to keep vial threads clean in order to achieve a hermetic seal. Internal standard recoveries of less than 50% are typical when vial is not properly sealed.

An additional sample is collected for potential high-level analysis in a 2 oz glass container or a third En Core® sampler. Within 48 hours of collection, approximately 5 grams of the solid sample from the 2 oz jar or the contents of the sampler must be preserved in 5.0 mLs of methanol.

Compositing Low Level 5035 VOA vials (soils):

Samples must be collected in En Core® samplers. At least 3 total En Core® samplers (5 En Core® samplers if MS/MSD is required) must be collected to ensure that the laboratory has adequate mass for homogenization (e.g. if 3 discrete samples collect 1 En Core® sampler each – if 2 discrete samples collect 2 En Core®samplers each).

The laboratory will extrude the En Core® samplers into a clean beaker that is cooled on ice and quickly (30-60 seconds) mix the soil together to reduce any potential loss of volatiles.

Once mixed a TerraCore sampler will be used to collect 2 aliquots (4 aliquots if MS/MSD is required) of the homogenized soil into pre-weighed 40-mL volatile vials with water and stir bar.

Aqueous & Solid Sample Holding Times

The maximum holding time for all samples is 14 days from collection, unless otherwise stated below.

If 2-chloroethyl vinyl ether is an analyte of interest, the holding time is 7 days in an unpreserved vial. The compound 2-Chloroethyl vinyl ether breaks down in the presence of acid and therefore, cannot be reported from a preserved sample.

EPA SW-846 Method 8260 - If acrolein and/or acrylonitrile is an analyte of interest, the maximum holding time is 7 days. Samples must be preserved to a pH of 4-5 SU and stored at –4 ± 2°C.

EPA Method 624.1 - If acrolein and/or acrylonitrile is an analyte of interest, the holding time is 3 days and must be analyzed from an unpreserved vial. The holding time may be extended to 14 days if sample pH is adjusted to 4-5 SU.

Field / Matrix QC

Trip blanks require two 40-mL volatile vials with the applicable preservative. If required, equipment blanks and field duplicates require an additional set of 40-mL volatile vials with the applicable preservative. If requested, MS/MSD require two additional sets of 40-mL volatile vials with the applicable preservative.

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with the *Sample Receiving* SOP [AD SOP ME0013H]. Chemical preservation is checked and recorded by the laboratory after sample analysis.

After receipt, samples are stored at 4 ± 2°C until sample preparation. Prepared samples (extracts, digestates, distillates, other) are stored at 4 ± 2°C until sample analysis.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 28 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

7.0 Equipment and Supplies

NOTE: Refer to the *Major Operational Equipment List* [QA Control Log ME001PM] for specific details regarding the equipment utilized during this procedure.

7.1 Equipment

7.1.1 Gas Chromatograph/Mass Spectrometer System (GC/MS) – An analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

7.1.2 Column – Silicon-coated fused-silica capillary columns. DB-624 30m x 0.25mm x 1.4 µm, DB-624 20m x 0.18mm x 1.0 µm, Rxi-624Sil MS 30m x 0.25mmIDx 1.4umdf or equivalent.

7.1.3 Mass Spectrometer – Capable of scanning from mass/charge (m/z) 35 to 300 every two seconds or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for 4-bromofluorobenzene (BFB) which meets all of the criteria in Table A-IV when 50 ng of the GC/MS tuning standard are injected through the GC.

7.1.4 GC/MS Interface – Any GC-to-MS interface that gives acceptable calibration points and achieves acceptable tuning performance criteria may be used.

7.1.5 Data System – A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as the Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIH Mass Spectral Library is recommended.

7.1.6 Purge and Trap – Eclipse model 4660 purge and trap coupled to an OI 4551-A or OI model 4100 autosampler.

7.1.7 Trap – OI Trap #10 or equivalent

7.1.8 Syringes –Hamilton Laboratory grade syringes or equivalent.

7.1.9 Laboratory glassware - Class A Volumetric flasks, and other laboratory glassware required for adequate preparation and storage of any standards and reagents.

7.1.10 Analytical balance - capable of accurately weighing to the nearest 0.01g.

7.2 Supplies

7.2.1 Soil sample collection kit (Terra Core® or equivalent) – Two 40-ml VOC vials each with a magnetic stir bar and 5.0 mL of reagent water weighed to nearest 0.01 gram. One 40-mLVOC vial containing 5.0 mL methanol preservative, weighed to the nearest 0.01 gram. One unpreserved 40-mL VOC vial weighed to the nearest 0.01 gram. One 2 oz glass container, at client request for percent solid analysis. One 5.0g plastic disposable Terra Core® Sampler or equivalent (En Novative Technologies, Inc., 1795 Industrial Drive, Green Bay, WI 54302, www.ennovativetech.com) The plastic sampler or T-handle has a barrel smaller than the neck of the soil vial and is not an approved storage device. One sampler is needed for each sample to be collected.

7.2.2 En Core® sampler – (En Novative Technologies, Inc., 1795 Industrial Drive, Green Bay, WI 54302, www.ennovativetech.com), or equivalent. Soil sample can be stored in the inert composite polymer En Core® sampler for up to 48 hours without preservative. 5-gram samplers are used for each sample.

7.2.3 Ottawa sand – for matrix match.

7.2.4 pH test strips, wide range, Fisher Scientific 13640516, or equivalent

7.2.5 pH test strips, 0-3 SU, Fisher Scientific 13-640-511, or equivalent

7.2.6 chlorine test strips, Hach Aquachek TRC test strips 2745050, or equivalent

8.0 Reagents and Standards

NOTE: Other volumes of standards and reagents may be prepared to account for expected usage. As long as all ratios are kept constant, this is not considered a deviation from the approved procedure.

NOTE: All stored reagents and standards must be labeled as required by the Preparation and *Documentation of Laboratory Standards and Reagents* SOP [QA SOP ME001HG], the *Contingency and Emergency Preparedness Plan* [HS SOP ME0012D], the *Safety Manual* [Corp Manual COR-MAN-HSE], and the *Laboratory Quality Manual* [QAMP ME0012K].

8.1 Reagents

8.1.1 Methanol (CH3OH) – Purge-and-trap grade or equivalent, demonstrated to be free from interferences for the compounds of interest at their lower limit of quantitation (LLOQ). This solvent must be stored apart from other solvents to avoid contamination. Commercially available from an approved vendor; follow manufacture expiration date.

8.1.2 Reagent water – A series of in-house deionized (DI) tanks is employed to purify the incoming water. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks. See the *Deionized Water System* SOP [QA SOP ME0012S] for further information.

8.1.3 Carrier gas – Ultra high purity helium.

8.1.4 Antifoaming agent - Antifoam B Silicone, part number JTB531-5, or equivalent.

8.1.5 Sodium Thiosulfate.

8.2 Standards

8.2.1 Primary standards - All standards used directly out of the flame-sealed ampules are considered to be primary standards. All primary standards expire six months after the open date (or manufacturer's expiration date, whichever has the stricter criteria). After the ampule is opened, the remaining standard is stored in a an amber 1mL autosampler vial. The standard must be refrigerated at ≤ -10 °C when not in use.

8.2.2 Working standards - All standards that are diluted from the manufacturer's ampule before analysis are considered working standards. The recipe for prepping the working standards can be found in Appendix F. All working standards expire one month after their prep date (or manufacturer's expiration date, whichever has the stricter criteria). Gas standards expire after one week. The standard must be refrigerated at ≤ -10 °C when not in use.

8.2.2.1 Daily working standards expire 12 hours after prep when not refrigerated and 24 hours after prep if refrigerated at 4 ± 2°C.

8.2.2.2 The gas working standards expire one week after the open date. The standard must be refrigerated at ≤-10° C when not in use.

8.2.2.3 Standards for the permanent gases must be monitored frequently by comparison to the initial calibration curve. A fresh ampule or fresh standard must be prepared if the gas compounds fail CCV criteria on more than one instrument.

8.2.2.4 Standards for the non-gases must be monitored frequently by comparison to the initial calibration. Fresh standards must be prepared is a noticeable drift is observed.

8.2.3 Calibration standards - A minimum five-point calibration curve is prepared. The low point must be at or below the reporting limit. Refer to Table A-X for typical calibration levels for all analytes. Other calibration levels may be used, depending on instrument capability, but the low standard must support the reporting limit and the high standard defines the upper range of the calibration.

8.2.4 Initial Calibration Verification (ICV) standard - An ICV is prepared from a different source of standards used in the preparation for the initial calibration. The concentration of the ICV must be at or near the middle of the calibration range. Refer to Table A-XII for ICV compounds and acceptance criteria.

8.2.4.1 Dichlorodifluoromethane is a common poor performer due to its high volatility. It is common for this compound to not pass ICV criteria, proper correction action will be taken.

8.2.5 Internal Standard (IS) solution - An IS solution is prepared. Compounds in the IS Mix are: pentafluorobenzene, 1,4-difluorobenzene, chlorobenzene-d5, 1,4-dichlorobenzene-d4.

8.2.5.1 Internal Standards are added to all standards and samples to result in 25-50 µg/L on-column value.

8.2.5.2 Internal Standards expire six months after the open date (or manufacturer's expiration date, whichever has the stricter criteria).

8.2.6 Surrogate standard spiking solution – Prepared at 125 or 250 µg/mL concentration. Due to variances in autosampler loop volume, on column concentration will vary. Surrogate compounds are listed in Table A-IX. Surrogates expire six months after the open date (or manufacturer's expiration date, whichever has the stricter criteria).

8.2.7 GC/MS tuning standard - A methanol solution containing 25 µg/mL of 4-bromofluorobenzene (BFB) is prepared.

8.2.8 Laboratory Control Sample (LCS) spiking solution – Prepared at 100 µg/ml concentration. LCS compounds are listed in Table A-VII.

8.2.9 Matrix Spike (MS) solution – Same as LCS spiking solution in Section 8.2.8 above.

9.0 Procedure

9.1 Equipment Preparation

9.1.1 Support Equipment

9.1.1.1 Incubators, water baths, refrigerator units, freezer units, bottle top dispensers, pipettes, thermometers, and ovens are maintained and verified as required by the *Equipment and Instrumentation* SOP [QA SOP ME002JT].

9.1.1.2 The balance is verified at the beginning of each analytical day using a certified weight set. Refer to the *Equipment and Instrumentation* SOP [QA SOP ME002JT] for balance verification procedures and acceptance criteria.

9.1.2 Instrument

9.1.2.1 Routine Instrument Operating Conditions – Refer to Table A-III, Appendix A for recommended instrument conditions.

9.1.2.2 Instrument Tuning – At the beginning of every twelve-hour shift when analyses are to be performed, the GC/MS system must be checked to see if acceptable performance criteria (Table A-IV) has been achieved for BFB (4-Bromofluorobenzene).

9.1.2.2.1 Inject 50 ng (2 µL) of the GC/MS tuning standard (Section 8.2.7) into the GC/MS system. Obtain a background-subtracted mass spectrum of BFB and confirm that all the key m/z criteria in Table A-IV are achieved. If all criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

9.1.2.2.2 A purged BFB using the surrogate standard (Section 8.2.6) can also be used to evaluate instrument tuning. In this case 25ng of BFB are introduced to the GC/MS system.

9.2 Initial Calibration

9.2.1 **Calibration Design**

9.2.1.1 The instrument is tuned for 4-bromofluorobenzene (BFB), calibrated initially with a five-point calibration curve, and verified each 12-hour shift with one or more continuing calibration standard(s). Recommended instrument conditions are listed in Table A-III. Daily run conditions are documented on forms saved in the source method file for each instrument.

9.2.1.2 All standards and samples are allowed to warm to room temperature before injecting.

9.2.1.3 Internal Standard Calibration Procedure – Internal standards are listed in Table A-VI. Use the base peak m/z as the primary m/z for quantitation of the standards. If interferences are noted, use one of the next two most intense masses for quantitation.

9.2.1.4 Compounds must be assigned to the Internal Standard (IS) with the closest retention time. See Table A-VI for IS assignments.

9.2.1.5 Prepare calibration standards at a minimum of five concentration levels for each parameter of interest. Additional levels can be analyzed and use the lower five for most analytes and the upper five for analytes that have poor response. Add the internal standard mixture to result in a concentration of 50 µg/L in the solution.

9.2.1.6 Analyze each calibration standard and tabulate the area of the primary characteristic m/z against concentration for each compound and internal standard. Calculate response factors (RF), average response factors, and the percent RSD (relative percent difference) of the response factors for each compound using the equations in Section 10.3.1. The average RF and %RSD must meet the criteria listed in Sections 9.2.3.2 through 9.2.3.6 The ICAL cannot be forced through zero. No sample analysis may be performed unless these criteria are met.

9.2.2 **Calibration Sequence**

9.2.2.1 Follow outlined procedure below to prepare at least a five-point calibration curve at concentrations of 1.0, 5.0, 20.0, 50.0, 100.0, 200.0 parts per billion (ppb). A 0.5 ppb standard may also be made if needed. All dilutions are made in class A volumetric flasks.

9.2.2.2 Using the primary working standards found in Appendix F make the following dilutions:

For Standard (ppb or µg/L)	Dilute (µL)	*into* Water (mL)
5.0	5.0	100
20	20.0	100
50	50.0	100
100	100.0	100
200	200.0	100

9.2.2.3 Using second preparation of the 50ppb standard above, make the following dilutions:

For Standard (ppb or µg/L)	Dilute (mL)	*into* Water (mL)
0.5	1.0	100
1.0	2.0	100

9.2.3 **ICAL Evaluation**

9.2.3.1 Weighing of Data Points

9.2.3.1.1 In linear fits, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason, it is preferable to increase the weighting of the lower concentration points. 1/Concentrations weighting (often called 1/ X2 weighting) will improve accuracy at the low end of the curve and should be used.

9.2.3.2 System Performance Check Compounds (SPCCs - 8260B only) - The minimum average RF for volatile SPCCs is listed below. If the minimum response factors are not met, the system must be evaluated and corrective action must be taken before sample analysis begins. Some possible problems are standard mixture degradation, injection port contamination, contamination of the front end of the analytical column, active sites in the column or chromatographic system and gas leaks or restrictions. This check must be met before analysis begins.

SPCC Compounds:	Minimum RF	Example Problem
Chloromethane	0.100	Purge flow too fast
1,1-Dichloroethane	0.100	Contaminated transfer lines in PT systems and/or active sites in trapping materials.
Bromoform	0.100	Purge flow too slow. Cold spots, active sites in the transfer lines
1,1,2,2-Tetrachloroethane	0.300	Same as 1
Chlorobenzene	0.300	Same as 1

9.2.3.3 Calibration Check Compounds (CCCs – 8260B only) - The %RSD of the response factors for each CCC in the initial calibration must be ≤ 30% for the initial calibration to be considered valid. This criterion must be met before sample analysis begins. Problems similar to those listed under SPCCs could affect this criterion.

9.2.3.4 CCC Compounds:
1,1-Dichloroethene
Chloroform
2-Dichloropropane
Toluene
Ethylbenzene
Vinyl Chloride

9.2.3.5 The average response factor is used when the %RSD of a compound is ≤ 15%.

9.2.3.6 For compounds that have % RSD >15% linear regression should be used. The correlation coefficient must be ≥ 0.995. The Target® software calculates the square of correlation coefficient which must be ≥ 0.99.

9.2.3.7 It is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria, unless the points are the highest or lowest of the curve AND the reporting limit and/or linear range is adjusted accordingly. In any event, at least 5 points must be included in the calibration curve.

9.2.3.8 A level may be removed from the calibration if the reason can be clearly documented, for example a poor purge. A minimum of five levels must remain in the calibration.

9.2.3.9 A level may be reanalyzed within 12 hours of the start of the calibration sequence, if no sample has been processed with the new calibration.

9.2.3.10 As soon as a new initial calibration curve is analyzed, all previous calibration curves are invalid whether the new calibration curve passes or not. The analysts cannot use an old curve to analyze sample.

9.2.3.11 If time remains in the 12-hour period initiated by the BFB injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration.

9.2.3.12 Quantitation is performed using average response factor from the initial curve, not the continuing calibration.

9.2.3.13 Relative Error

9.2.3.13.1 Either of the two methods described below may be used to determine calibration function acceptability for linear and non-linear curves. Both procedures refit the calibration data back to the calibration model and evaluates the difference between the measured and the true amounts or concentrations used to create the model.

9.2.3.13.2 % Error:

$$\%\ \text{Error} = \frac{X_i - X'_i}{X_i} \times 100$$

Where:
X'i = Measured amount of analyte at calibration level i, in mass or concentration units.
Xi = True amount of analyte at calibration level i, in mass or concentration units.

Percent error between the calculated and expected amounts of an analyte must be ≤ 30% for all standards, except the lowest calibration standard which must be ≤ 50%.

9.2.3.13.3 Relative Standard Error (RSE):

$$RSE = \sqrt{\sum_{i=1}^{n}\left[\frac{x'_i - x_i}{x_i}\right]^2 \Big/ (n-p)}$$

Where:
x'i = Measured amount of analyte at calibration level i, in mass or concentration units.
xi = True amount of analyte at calibration level i, in mass or concentration units.
p = Number of terms in fitting equation (average = 1, linear = 2)
n= Number of calibration points.

The RSE acceptance limit criterion for the calibration model is the same as the RSD limit (Section 9.2.3).

9.2.4 **Initial Calibration Verification (ICV)** / Second Source Calibration Verification - A standard that is prepared from a source independent of standards of the initial calibration. The ICV contains all Target compounds and its concentration must be at or near the mid-level of the initial calibration. The concentration of the ICV, determined from the analysis, is compared to the known value of the standard to determine the accuracy of the ICAL. Recovery must be within 70-130% before sample analysis to begin. The ICV must be analyzed immediately after the ICAL.

9.2.5 **Continuing Calibration Verification (CCV)** (also known as the Instrument Performance Check)

9.2.5.1 At the start of each 12-hour period, the GC/MS tuning standard must be analyzed. A ≤50 ng injection of 4-bromofluorobenzene (BFB) must result in a mass spectrum for BFB which meets the criteria given in Table A-IV.

9.2.5.2 Following a successful BFB analysis, the continuing calibration verification (CCV) standard(s) are analyzed. The standards must contain all volatile analytes, including all required surrogates. A mid-level calibration standard is used for the continuing calibration.

9.2.5.3 Continuing Calibration Verification (CCV) technical acceptance criteria:

9.2.5.3.1 The response factor for the SPCC compounds must meet the criteria listed in Section 9.2.3.2.

9.2.5.3.2 The percent difference of the CCC compounds from the initial calibration must be ± 20% difference (8260B only). See Section 10 for calculation. In addition, the percent difference of all other target analytes must meet the criteria in Table A-XIII with the following exceptions:

9.2.5.3.2.1. One or more CCC's may fail if the CCC is not a target analyte and all target analytes meet the 20% difference or drift criterion.

9.2.5.3.2.2. If the CCC's are not included in the list of analytes for a project, and therefore not included in the calibration standards, then all analytes must meet the 20% difference or drift criterion.

9.2.5.4 The CCV % difference criteria for 8260C/D is 20% for all compounds.

9.2.5.4.1 The internal standard response must be within 50-200% of the response in the mid-level of the initial calibration.

9.2.5.4.2 The internal standard retention times must be within ± 30 seconds of the retention times in the mid-level of the initial calibration.

9.2.5.5 Once the above criteria have been met, sample analysis may begin. Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs. Analysis may proceed until 12 hours from the injection of the BFB have passed. (A sample injected less than 12 hours after the BFB is acceptable.)

9.2.5.6 In any case, the criteria for SPCC compounds must be met for 8260B.

9.2.5.7 Corrective Action for CCV Failure

9.2.5.7.1 Check all calculations and integrations. Inspect the GC/MS system for malfunction. Try to determine the cause of the failure. If it appears that the analytical system is in proper working order but the standard has been improperly prepared, degraded, or compromised, a new standard may be prepared and reanalyzed. If it is suspected that the instrument is unstable or malfunctioning then maintenance and/or repair must be performed before running another CCV.

9.2.5.7.1.1. If the 2nd consecutive CCV fails to pass acceptance criteria, the analyst must perform corrective action and demonstrate that the system is stable and in proper working order with two consecutive passing CCV's or an initial calibration (ICAL) must be analyzed before analysis of samples can begin.

9.2.5.7.1.2. An LCS can be used as the second passing CCV if it is run directly after the initial CCV and if it passes all criteria.

9.2.5.7.2 If the system appears to be in working order and standards are prepared correctly corrective action is not required for the following exceptions:

9.2.5.7.2.1. If ≤10% of the analytes exceed the calibration verification criteria, then the initial calibration may still be used for non-detect samples. Any detected analytes exceeding the limit must be reported as estimated.

9.2.5.7.2.2. Non-detected analytes may be reported if the calibration verification for that specific analyte exceeds the upper acceptance criteria (e.g., > +20%).

9.2.5.7.2.3. In order to report non-detected analytes that exceeds the lower acceptance criteria (e.g., < -20%), a sensitivity verification standard at or below the LOQ must be analyzed in the analytical batch. The analyte must be detected in the LOQ standard and meet all of the qualitative identification criteria. (8260C/D only)

9.2.5.7.2.4. If more than 10% of analytes are exceeding the calibration verification criteria for both detected and non-detected analyte corrective action is required.

9.2.5.7.3 If >10% of the analytes exceed the calibration verification criteria, and instrument maintenance does not correct the problem, then a new ICAL must be analyzed before sample analysis may begin.

9.3 Sample Preparation

9.3.1 High level soil sample preparation procedures are in Appendix D.

9.4 Analysis

9.4.1 Calibrate the instrument as described in Section 9.2. Depending on the target compounds required by the client, it may be necessary to use more than one calibration standard.

9.4.2 All samples must be analyzed using the same instrument conditions as the preceding continuing calibration standard.

9.4.3 The weight for soil sample in water must be determined before analysis can begin. Weigh the vial to the nearest 0.01 gram. Document the sample weights in the VOA Extraction Batch 5035 High Level / Waste Dilution [VOA Forms ME001CY] logbook or in directly into LIMS 4.

9.4.3.1 To account for the weight of the label placed on the sample container during sample receipt, the balance must be tared using a sample label. After taring, remove the label to obtain sample weights.

Note: The presence of excessive amounts of tape or labeling requires documentation in an NCM to ensure this information is provided in the case narrative of the final report.

9.4.4 The frozen soil sample must be warmed to ambient temperature before analysis can begin.

9.4.5 Add internal standard and surrogate standard to the sample to result in 25-50 µg/L concentration in the aqueous solution.

9.4.6 Load the autosampler and begin the sequence. The procedure for the samples and the standards must be the same.

9.4.7 The data system will determine the concentration of each analyte in the extract using calculations equivalent to those in Section 10. Quantitation is based on the initial calibration, not the continuing calibration.

9.4.8 Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst or automatically by the data system.

9.4.9 Target compounds identified by the data system are evaluated using the criteria listed in Section 10.1.

9.4.10 Library searches of peaks present in the chromatogram that are not target compounds (Tentatively Identified Compounds, TIC) may be performed if required by the client. They are evaluated using the criteria in Section 10.1.2.

9.4.11 Samples with matrices that foam should be treated with antifoaming agent. Antifoam is diluted 20X in deionized water. From this diluted antifoam solution, inject 20 ul into the septa of the vial until the foaming is controlled.

9.4.12 When using antifoam, either a method blank or instrument blank needs to be injected with the largest amount of antifoam used in the samples. The blank should be analyzed prior to the samples in order to confirm there is no contamination in the antifoam solution. An LCS must also be prepared with the largest amount of antifoam used in the samples to verify the antifoam does not contribute any interference. All related QC criteria apply.

9.5 Dilutions

9.5.1 Dilutions must be performed so that the resulting dilution factor is an integer.

9.5.2 For aqueous, high level soil, and waste samples, if the response for any compound exceeds the working range of the GC/MS system, a dilution of the sample or extract is prepared and analyzed. An appropriate dilution should target the CCV (mid-range of the ICAL). Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or its highest hits are below 25% of the calibration range and the matrix allows for analysis at a lesser dilution, the sample must be reanalyzed at a dilution targeted to bring the largest hit above the CCV (mid-range of the ICAL).

9.5.3 For low level soil samples, if the response for any compound exceeds the working range of the GC/MS system, a high-level soil analysis must be performed. See Appendix D-4 for details.

9.5.4 *Guidance for Dilutions Due to Matrix* – If the sample is initially run at a dilution and the baseline rise is less than the height of the internal standards, or if individual non-target

peaks are less than two times the height of the internal standards, the sample should be reanalyzed at a less concentrated dilution. This requirement is approximate and subject to analyst judgment. For example, samples containing petroleum fuel may need to be analyzed at a higher dilution to avoid contaminating the purge-and-trap system.

9.5.5 *Reporting Dilutions* – The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

9.5.6 When preparing manual dilutions syringes of 1 mL or less flush 3 times with methanol and syringes greater than 1 mL flush at least 1 time with methanol and then 3 times with DI water between dilutions.

9.6 Internal Standard Criteria for Samples

9.6.1 If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

9.6.2 If the retention time of any internal standard in any sample varies by more than 0.1 minute from the preceding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows. Refer to Section 10 for further evaluation of co-eluting pairs.

9.6.3 The internal standard response must be within 50 – 200% of the response in the last continuing calibration standard.

9.7 pH and Chlorine Verifications

9.7.1 All aqueous samples must be checked for pH immediately after analysis. The pH must be taken from the parent sample in the case of a dilution. The pH verification is documented on the Chemstation sequence printout. If the pH is not <2 an NCM must be completed to ensure the anomaly is communicated to the client and reported in the case narrative unless the sample is in an unpreserved vial. If the sample is in an unpreserved vial and the pH <2, this must be noted on the Chemstation sequence printout.

9.7.1.1 EPA 624.1 aqueous samples must be checked for the presence of Chlorine immediately after sample analysis. The Chlorine must be taken from the parent sample in the case of a dilution. The Chlorine verification is documented on the Chemstation sequence printout. If Chlorine is present an NCM is documented and issued to the project manager. The project manager notifies the client of the anomaly in the report narrative

9.8 Percent Moisture

9.8.1 Analytical results may be reported as dry or wet weight, as required by the client. Percent moisture must be determined if results will be reported as dry weight.

9.8.2 Refer to *the Percent Solid and Percent Moisture in Solids and Semi-Solids* SOP [Wet Chem SOP ME0013F] for percent moisture determination.

9.9 Troubleshooting Guide

9.9.1 Instrument Maintenance – In addition to the checks listed in the instrument maintenance schedule in the Shealy QAMP, the following maintenance activities must be monitored daily and performed if necessary:

- Injection port inspection/maintenance
- Clip column
- Install new or cleaned injection port liner and gold seal
- Install new septum
- Change trap every 6 month or as necessary.
- Perform mass calibration as necessary.

9.9.2 Major Maintenance – A new calibration is necessary following major maintenance. Major maintenance includes changing the column, cleaning the ion electron source, replacing the multiplier, and changing the trap to a new lot number. Refer to the manufacturer's manual for specific guidance. All maintenance must be recorded in the instrument maintenance log with the date and initials.

9.10 Sufficient records must be maintained to allow for the historical reconstruction of testing procedures. Refer to the *Logbook and Data Recording* SOP [QA SOP ME0012T] for details regarding documentation requirements. The following quality documents are included in the technical record for this analysis:

- *Working Standard Prep Log* – Volatiles [VOA form ME001KC]
- *Working Standard Prep Log* – AQ CLP [VOA form ME001KD]
- *VOA Extraction Batch 5035 High Level / Waste Dilution* [VOA Forms ME001CY]

10.0 DATA ANALYSIS AND CALCULATIONS

10.1 Qualitative Identification

10.1.1 An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NIST02 library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions. (Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.)

- The sample component retention time must compare to within ± 0.06 RRT units of the RRT of the standard component. For reference, the standard must be run within the same twelve hours as the sample.

- The characteristic ions of a compound must maximize in the same scan or within one scan of each other.

- The relative intensities of ions must agree to within ± 30% for target analytes and ± 20% for TICs (between the standard and sample spectra). (Example: A target ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20% and 80%).

10.1.1.1 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst the identification is correct, the analyst shall report that identification and proceed with quantitation. GC/MS analysts are trained to carefully evaluate data for compounds that have close retention times and share common ions.

10.1.1.1.1 If an alternate compound was selected by the analyst, the compound will be flagged with an “H” by Target®. The triple plots are generated to document that the correct compound was selected.

10.1.1.1.2 If a compound fails the ratio test, the compound will be flagged with a “Q” by Target®. The triple plot must be printed to document that the correct compound was selected.

10.1.1.1.2.1. The following compounds may display the “Q” flag at concentrations > half the calibration range due to shared ions:

Close Eluting Compounds	
Ethylbenzene	m&p-Xylenes
o–Xylene	Styrene
Allyl chloride	Acetonitrile
Allyl chloride	Carbon disulfide
Dichlorobenzenes	Dichlorobenzenes
Vinyl acetate	Diisopropyl ether
trans-1,4-Dichloro-2-butene	1,2,3-Trichloropropane
1,2,4-Trimethylbenzene	1,3,5-Trimethylbenzene

10.1.1.2 Due to the type of column used, there can be considerable breakdown of benzene into 1,2-dichloroethane (1,2-DCA) resulting in possible false positive detection of 1,2-DCA. Anytime a large benzene peak is present, special attention to the spectra must be taken. Ion 98 is only present in 1,2-DCA and not present in benzene so that this ion must be present for 1,2-DCA to be detected. Consult the

group leader and/or the technical director for further guidance when this issue occurs

10.1.2 Tentatively Identified Compounds (TICS) - For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. Computer generated library search routines must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample spectra with the nearest library searches shall the mass spectral interpretation specialist assign a tentative identification. Guidelines for making tentative identification are:

- Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) must be present in the sample spectrum.
- The relative intensities of the major ions must 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%)
- Molecular ions present in the reference spectrum must be present in the sample spectrum.
- Ions present in the sample spectrum, but not in the reference spectrum, must be reviewed for possible background contamination or presence of co-eluting compounds.
- Ions present in the reference spectrum, but not in the sample spectrum, must 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.
- Automatic background subtraction can severely distort spectra from samples with unresolved hydrocarbons.

10.1.3 Manual Integration

10.1.3.1 Manual changes to automated integration is called manual integration. Manual integration is sometimes necessary to correct inaccurate automated integrations but must never be used to meet QC criteria or to substitute for proper instrument maintenance and/or method set-up. To assure that all manual integrations are performed consistently and are ethically justified, all manual integrations must be performed, reviewed, and recorded in accordance with corporate SOP ENV-SOP-CORQ-0006, Manual Integration.

10.2 Quantitative Identification

10.3 Calculations

See the *Laboratory Quality Assurance Manual* [QAMP ME0012K] for equations for common calculations.

10.3.1 Percent Relative Standard Deviation (RSD) for Initial Calibration

$$\% \text{ RSD} = \frac{\text{SD}}{\overline{\text{RF}}} \text{ x } 100$$

Where:

RF = Mean RFs from initial calibration for a compound
SD = Standard deviation of RFs from initial calibration for a compound

$$\text{SD} = \sqrt{\sum_{i=1}^{N} \frac{\left(\text{RF}_i - \overline{\text{RF}}\right)^2}{\text{N} - 1}}$$

Where:

RF_i = RF for each of the calibration levels.
N = Number of RF values.

10.3.2 Continuing Calibration (CCV) Percent Difference and Percent Drift

$$\% \text{ Difference} = \frac{\text{RF} - \overline{\text{RF}}}{\overline{\text{RF}}} \text{ x } 100$$

Where:

RF = Compound's response factor in the CCV.
$\overline{\text{RF}}$ = Compound's average response factor in the initial calibration.

$$\% \text{ Drift} = \frac{\text{Calc. Conc.} - \text{Theoretical Conc.}}{\text{Theoretical Conc.}} \text{ X } 100$$

10.3.3 Concentration in the Sample – The concentration of each identified analyte and surrogate in the sample is from the average RF of the initial calibratioin.

10.3.3.1 On-Column Concentration (µg/L)

$$C = \frac{A_x \times C_{is}}{A_{is} \times \overline{RF}}$$

10.3.3.2 Aqueous Sample (µg/L)

Concentration = C x DF

Where:

A_x= Response for analyte, in area count of the characteristic ion.
A_{is} = Response for internal standard.
C_{is} = Concentration of internal standard.
DF = Dilution factor.

10.3.3.3 Sediment/Soil, Sludge (on a dry-weight basis) and Waste (normally on a wet-weight basis)

$$\text{Concentration, µg/kg} = \frac{C \times V_0 \times DF}{W \times D}$$

Where:
V_0 = Volume of sample extract in mLs.
W = Weight of sample extracted or diluted in grams.
D = (100 - % moisture in sample)/100, for a dry weight basis or 1 for a wet weight basis.

10.3.4 MS/MSD Percent Recovery Calculation

$$\text{Matrix Spike Recovery} = \frac{S_{SR} - S_R}{S_A} \times 100\%$$

Where:

S_{SR} = Spike sample result.
S_R = Sample result.
S_A = Spike added.

10.3.5 Relative % Difference Calculation for the MS/MSD

$$RPD = \frac{MS_R - MSD_R}{½ (MS_R + MSD_R)} \times 100$$

Where:
RPD = Relative percent difference.
MS_R = Matrix spike result.
MSD_R = Matrix spike duplicate result.

10.3.6 Relative Response Factor Calculation

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:
A_x= Area of the characteristic ion for the compound being measured.
A_{is}= Area of the characteristic ion for the specific internal standard.
C_x = Concentration of the compound being measured (µg/L).
C_{is} = Concentration of the specific internal standard (µg/L).

10.3.7 Calculation of TICs – The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

A_x = Area of the total ion chromatogram for the compound being measured.
A_{is} = Area of the total ion chromatogram for the nearest internal standard without interference.
RF = 1.

11.0 Quality Control and Method Performance

11.1 Quality Control

The following QC samples are prepared and analyzed with each batch of samples.

QC Item	Frequency
Method Blank (MB)	1 per batch
Laboratory Control Sample (LCS)	1 per batch
Laboratory Control Sample Duplicate (LCSD)	As needed
Matrix Spike (MS)	1 per batch
Matrix Spike Duplicate (MSD)	1 per batch
Sample Duplicate	As needed

11.2 Instrument QC

The following Instrument QC checks are performed.

QC Item	Frequency
Initial Calibration	When CCVs do not meet criteria
Initial Calibration Verification	After calibration.
Tuning standard	At the start of each 12 hour period
BFB	After tuning
Continuing Calibration Verification	After BFB
LOQ Verification	Quarterly, or when significant changes are made to prep or analysis procedures
NC LOQ Verification Standard	Each day NC samples are analyzed

11.3 Acceptance Criteria and Required Corrective Action

11.3.1 Initial and Continuing Demonstration of Capability (IDOC and CDOC) – To establish the ability of an analyst to generate acceptable accuracy, each analyst must make a satisfactory initial demonstration of capability prior to using any method and at any time there is a change instrument type or method. Thereafter, a continuing demonstration of capability is required annually. Refer to the *Demonstration of Capability* SOP [QA SOP ME001F2] for additional information.

11.3.2 Control Limits – In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS/LCSD). These limits are reviewed regularly and updated as needed. Acceptance limits are calculated by using the mean recovery ± 3 standard deviations. Refer to the LCS section below (Section 11.3.4) for maximum limit ranges.

11.3.2.1 All surrogate, LCS/LCSD, and MS/MSD recoveries must be entered into the Shealy LIMS (when available) or other database so that accurate historical control limits can be generated. Surrogate and matrix spike percent recoveries will be reported for all dilutions.

11.3.2.2 Refer to the Quality Assurance Management Plan [QAMP ME0012K] or the Trend Analysis of *Data Using Control Charts* policy [QA Policy ME001IW] for further details on control limits.

11.3.3 Method Blank (MB) / Laboratory Reagent Blank – A method blank is prepared and analyzed with each batch of samples. The method blank consists of a quality system matrix that is similar to the associated samples (organic free reagent water or Ottawa sand), free of the analytes of interest, and is taken through the entire preparation and analytical procedures. The method blank is used to identify any systemic and process interferences or contamination that may lead to the reporting of elevated concentrations of false positive data. Surrogates and internal standards are added and the method blank is carried through the entire analytical procedure.

11.3.3.1 The method blank must not contain any analyte of interest above 1/2 the LOQ, or project-specific requirements. If the method blank contains an analyte of interest above this limit, the method blank and associated samples must be re-analyzed with a method blank, which passes acceptance criteria. Corrective action may not be required if the samples are not affected. Where permitted by the program area or client, the following exceptions apply. Any method blank that does not meet acceptance criteria, is flagged on the data report with a B flag.

11.3.3.1.1 The sample was non-detect for the contamination.

11.3.3.1.2 The sample concentration was ≥10x the blank concentration.

11.3.3.2 The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, sample analysis must stop immediately. Corrective

action must be taken. A new method blank must be analyzed and meet the surrogate criteria before sample analysis can begin.

11.3.3.3 Sample results are NOT blank subtracted.

11.3.4 Laboratory Control Sample (LCS) - A laboratory control sample (LCS) is prepared from a source independent of the initial calibration standards (8260B only) and analyzed with every batch of samples. The LCS is spiked with the compounds listed in Table A-VII, unless specified by a client or agency. The LCS sample for low level soil (5035) must contain approximately 5 grams of Ottawa sand. The acceptable control limits for LCS/LCSD are statistically derived. However, if the lower control limits are < 70% or the upper control limits are >130%, then the default limits of 70-130% will apply to the compounds in Table A-VII. The only exceptions are the * compounds found in Tables A-XIV and A-XV. These compounds have default control limits of 60-140% (if not statistically more stringent). Refer to Tables A-XIV and A-XV for the entire lists of aqueous and soil LCS recovery limits. Refer to Table DoD-III for DoD control limits. Refer to Table A-VII and Table A-XV for a list of spiked LCS compounds and their spiking concentrations.

11.3.4.1 For 8260D, the LCS may be prepared with standards used for calibration.

11.3.4.2 Marginal Exceedances: When there are a large number of analytes in the LCS or matrix spike, there is a high statistical probability that a few analytes will recover outside of control limits. This may not indicate that the system is out of control, therefore corrective action may not be necessary. The number of allowable marginal exceedances (ME) is based on the number of analytes in the LCS. If more analytes exceed the LCS control limits than is allowed, corrective action is necessary. The number of allowable marginal exceedances is as follows:

Number of Analytes in LCS	Number Allowed as Marginal Exceedances
>90	5
71-90	4
51-70	3
31-50	2
11-30	1
<11	0

11.3.4.2.1 Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systemic problem. The source of the error must be located and corrective action taken.

Note: Marginal exceedances may not be allowed for some projects (not allowed for South Carolina compliance samples, or DoD without project specific approval). If any compounds fail to meet LCS limits, refer to actions outlined in Section 11.3.4.3.

11.3.4.3 Corrective Action for LCS failure - If an LCS fails to meet the ME criteria for a target compound, an attempt must be made to determine the source of error and find a solution. A target compound is any compound being reported for that sample. All findings and corrective actions should be documented in the maintenance logbook. The corrective action applied shall be based on professional judgment in the review of other QC measures (i.e. surrogates). If analyte falls outside a second time or if there is not sufficient sample to be reanalyzed, an NCM must be filled out documenting the failure and corrective actions that were applied.

11.3.4.4 If the LCS is failing high and the sample is non-detect an NCM can be generated and reanalysis is not required.

11.3.5 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) – For each QC batch, analyze a matrix spike and a sample duplicate or an MS/MSD if client requested. Spiking compound and levels are given in the appendices. When an MS/MSD is analyzed compare the percent recovery and relative percent difference (RPD) to those in the laboratory specific historically generated limits. MS/MSD failures are flagged in the report.

11.3.5.1 If any individual recovery or RPD falls outside the acceptable range ≤ 30%, corrective action must occur. The initial corrective action will be to check all calculations. If the calculations are correct, check the recovery of that analyte in the Laboratory Control Sample (LCS). If the recovery of the analyte in the LCS is within limits, then matrix interference has been demonstrated and the laboratory operation may proceed.

11.3.5.2 If the recovery for any component is outside QC limits for the matrix spike, sample duplicate, or matrix spike/spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will include re-preparation and reanalysis of the batch. An NCM must be generated to document this occurrence.

11.3.5.3 Every effort is made to ensure that a Matrix spike and a sample duplicate are included in every batch. On the rare occasion that one is not possible (sample bottle was broken, etc.) then an LCS duplicate must be analyzed or a MS/MSD pair can be analyzed at the client's request.

11.3.5.4 The matrix spike/ matrix spike duplicate must initially be analyzed at the same dilution as the parent sample (or the un-spiked sample). If the sample requires reanalysis at a dilution and objective evidence of matrix interference exists, re-analysis may not be required.

11.3.5.5 The sample duplicate must be analyzed at the same dilution as the parent sample. If the parent sample requires a dilution, the sample duplicate must be re-analyzed at the same dilution if sufficient sample volume is available.

11.3.6 Surrogates - Every sample, blank, and QC sample is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits. The compounds routinely included in the surrogate spiking solution, along with recommended standard concentrations, are listed in Table A-IX.

11.3.6.1 If any surrogates are outside limits, the following corrective actions must take place. For high level soil samples in methanol and/or non-aqueous samples with dilutions ≥ 5x, all corrective actions are applicable with the exception of sample reanalysis. If the corrective actions do not fix the problem, an NCM must be written.

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze the sample if either of the above checks reveals a problem.
- It is only necessary to reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect. A demonstrated matrix effect must be documented on an NCM.

11.3.7 LOQ Verification – A verification of the LOQ is performed quarterly, and whenever significant changes are made to the preparation and/or analytical procedure.

11.3.7.1 The verification is performed by the extraction and analysis of reagent water spiked at 0.5-2 times the established LOQ.

11.3.7.2 The LOQ check is carried through the same preparation and analytical procedures as environmental samples and other QC samples.

11.3.7.3 The LOQ verification is performed after the initial calibration and must be performed on every instrument where data is reported annually.

11.3.7.4 Recovery of the target analytes in the LOQ verification must be within ± 20 % of the LCS criteria, until the laboratory has enough data to establish acceptance limits for the LOQ. This practice acknowledges the potential for greater uncertainty at the low end of the calibration curve.

11.3.7.5 When reporting concentrations below the LOQ, the results are qualified as estimated with a J flag on the analytical report.

11.3.8 North Carolina LOQ Verification Standard – A standard at or below the LOQ must be analyzed each day North Carolina samples are analyzed. To be acceptable, all analytes of interest must be detected. If not, reanalyze the batch. If reanalysis is not possible, generate an NCM.

11.3.9 Trip Blank (TB) – The purpose of the TB is to detect possible volatiles organic contamination of samples to be analyzed for volatile compounds. TBs are prepared by

filling volatile sampling vials with deionized water. TBs accompany volatile sample collection vials to the sample site, back to the laboratory, and are stored with the collected samples before analysis. TB are treated just like any other sample within the analysis batch, but must not be used as matrix spikes or matrix spike duplicates.

11.3.9.1 Trip blanks shall only be re-run when QC suggests the re-run is needed.

Note: Refer to appendices for state and/or program specific method performance criteria, which superseded and/or supplement the general method performance criteria prescribed in this SOP.

11.4 Method Performance

11.4.1 Method Validation

11.4.1.1 Detection Limits

Detection limits (DL) and limits of quantitation (LOQ) are established at initial method setup and verified on an on-going basis thereafter. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 Method Validation and Instrument Verification and to the Method Validation SOP [QA Policy ME003BF] for these procedures.

11.4.1.2 Non-Standard Analytes

For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

11.4.1.3 Data Quality Objectives (DQO)

Refer to project-specific Quality Assurance plans for DQO information.

11.5 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to the *Demonstration of Capability* SOP [QA SOP ME001F2] for more information.

12.0 Data Review and Corrective Action

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical

record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employee's complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to the *Data Review* SOP [QA SOP ME003LP] for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

Refer to Appendix B for a complete summary of QC, acceptance criteria, and recommended corrective actions for QC associated with this test method.

13.0 Pollution Prevention

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

14.0 Modifications

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 Method Validation and Instrument Verification for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

For compliance samples, all procedures outlined in this SOP must be followed without exception. In the event that a deviation from this SOP cannot be avoided (i.e., demonstrated and uncorrectable matrix interference, non-compatible matrix, insufficient sample amount due to client or laboratory error) it is imperative that an NCM is completed that clearly documents the anomaly. This anomaly must be communicated to the client such that corrective action activity including sample recollection can be performed. Based upon the type and severity of anomaly, the results may not be appropriate for compliance reporting. Discussions between the client, the appropriate regulatory authority, and the laboratory will be necessary to resolve certain anomalies. In any case, it is essential that any anomaly be documented on an NCM.

14.1 The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

14.2 Directions for analysis of Method 624.1 are in Appendix C.

14.3 The reporting limit for total xylene is 5.0 µg/L. If m/p xylenes and o-xylene are detected at less than 5 µg/L each, but the sum is more than 5 µg/L, the total value will be reported.

14.4 All Department of Defense (DOD) / Department of Energy (DOE) requirements are found in Appendix DoD.

14.5 Historically the surrogate compounds have been included in the multi-point initial calibration at variable concentrations in order to evaluate the linear response as with any target analyte. However, with improvements in instrumentation and more reliance on the autosampler, an option is available depending on the project-specific data quality requirements for allowing the autosampler (or using a manual technique) to spike the initial calibration standards with surrogates in the same manner as the samples are spiked. With this option the surrogate

standards in the initial calibration can be averaged to develop a response factor and an effective one-point calibration with the sole purpose to measure the surrogate recovery using the same concentration for each sample analysis. For this calibration option the surrogate linear response is less important, since multiple concentrations of surrogates are not being measured. Instead, the surrogate concentration remains constant throughout and the recovery of this known concentration can easily be attained without demonstrating if the response is linear.

14.6 Directions for analysis of Method 8260B/C/D and 624.1 for 1,4-Dioxane by Selected Ion Monitoring can be found in Appendix E.

15.0 Responsibilities

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 Attachments

16.1 Appendix A: Tables

16.1.1 Table A-1: CAS Numbers and Reporting Limits

16.1.2 Table A-II: Reportable Analytes for TCLP

16.1.3 Table A-III: Recommended Instrument Conditions

16.1.4 Table A-IV: BFB Key Ions and Ion Abundance Criteria

16.1.5 Table A-V: Characteristic Ions

16.1.6 Table A-VI: VOA Target Compounds and Surrogates with Corresponding Internal Standards for Quantiation

16.1.7 Table A-VII: 8260B/C/D LCS/MS/MSD Compounds

16.1.8 Table A-VIII: TCLP LCS/MS/MSD Compounds

16.1.9 Table A-VIX: 8260B/C/D Surrogate Compounds

16.1.10 Table A-X: Calibration Levels, Aqueous (µg/L) and Solid (µg/kg), Regular Level

16.1.11 Table A-XI: Low Level Calibration Levels, Aqueous (µg/L)

16.1.12 Table A-XII: ICV Acceptance Criteria

16.6.12 Table F-XII: 8260 Secondary Pentachloroethane Standard (Total volume 2mL in Methanol)

16.7 Appendix G: SW-846 8260C Requirements

16.7.1 Table G-I: Recommended Minimum Relative Response Factor Criteria for Initial and Continuing Calibration Verification

16.8 Appendix H: Changes to 8260D Rev 4 Compared to 8260C Rev 3 (as pertains to this SOP)

16.9 Appendix DoD: DoD/DOE Method Specific Quality Control Requirements

16.9.1 Table DoD-I: Volatile Organic Compounds and Standard Reporting Limits

16.9.2 Table DoD-II: Volatile Organic Surrogate Compounds and Control Limits

16.9.3 Table DoD-III: LCS/MS Control Limits for Volatile Organic Compounds

16.9.4 Table DoD-IV: Characteristic Ions for Volatile Organic Compounds

17.0 References

Note: Where reference exclude a date or edition, the latest edition of the referenced document adopted/recognized by the laboratory's accreditation bodies applies. Refer to the *Laboratory Quality Manual* [QAMP ME0012K] for details.

17.1 SW-846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, December 1996, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Method 8260B.

17.2 SW-846, Test Methods for Evaluating Solid Waste, Revision 4, Update V, July 2014, Determinative Chromatographic Separations, Method 8000D.

17.3 SW-846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, December 1996, Purge-and-Trap for Aqueous Samples, Method 5030B.

17.4 SW-846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, December 1996, Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Method 5035, Revision 0.

17.5 SW-846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, December 1996, Sample Preparation for Organic Volatile Compounds, Method 5000B.

17.6 SW-846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, December 1996, Sample Preparation for Organic Volatile Compounds, Method 3585.

17.7 Consolidated Quality System Manual (QSM) for Environmental Laboratories. Department of Defense (DoD) / Department of Energy (DoE).

17.8 Standard Methods for the Examination of Water and Wastewater, 22nd Edition, 2011 – Purge and Trap Capillary-Column Gas Chromatographic/Mass Spectrometric Method, Method 6200 B-2011.

17.9 South Carolina Department of Health and Environmental Control (DHEC). Certification of the Oxygenate Compounds, February 2017.

17.10 SW-846, Test Methods for Evaluating Solid Waste, August 2006, Revision 3, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Method 8260C.

17.11 SW-846, Test Methods for Evaluating Solid Waste, June 2018, Revision 4, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry, Method 8260D.

17.12 Laboratory Accreditation Standards. TNI Standard. The NELAC Institute.

17.13 General Requirements for the Competence of Testing and Calibration Laboratories. International Standard ISO/IEC 17025.

18.0 Revision History

Revision #	Revision Date	Section Modified	Modification	Reason for Modification
-22	**09/23/2021**	Signature Page	Updated to QM to Kelly Nance and added Kristina Bouknight as EHSO	Personnel update
		All	Re-wrote and re-formatted entire document	Compliance with Pace policy
		7	Moved Ottawa sand from Reagents and Standards to Equipment and Supplies	Clarification
		7.2	Added pH and chlorine test strips	SC DHEC audit finding #18
		9.8.2	Added reference to % Solids SOP	SC DHEC audit finding #18
		Table A-XIII	Updated to 8260D acceptance criteria	SC DHEC audit finding #18
		Appendix C C.1.1	Added that 'X' and 's' criteria in Table C-II must be met for the EPA 624.1 initial demonstration of capability (IDOC).	SC DHEC audit finding #18

Appendix A: Tables

TABLE A-I: CAS Numbers and Reporting Limits

Analytes	CAS Number	Standard Reporting Limits				
		Trace Aqueous µg/L	Low Level Aqueous µg/L	Aqueous µg/L	Low Soil µg/kg	High Soil µg/kg
Acetone	67-64-1	N/A	N/A	20	20	1000
Acetonitrile	75-05-8	N/A	N/A	10	10	500
Acrolein	107-02-8	N/A	N/A	50	50	2500
Acrylonitrile	107-13-1	N/A	N/A	50	50	2500
Benzene	71-43-2	0.5	1	5	5	250
Benzyl chloride	100-44-7	N/A	N/A	5	5	250
Bromobenzene	108-86-1	0.5	1	5	5	250
Bromochloromethane	74-97-5	0.5	1	5	5	250
Bromodichloromethane	75-27-4	0.5	1	5	5	250
Bromoform	75-25-2	0.5	1	5	5	250
Bromomethane (Methyl bromide)	74-83-9	0.5	1	5	5	250
2-Butanone (MEK)	78-93-3	N/A	N/A	10	10	500
n-Butylbenzene	104-51-8	0.5	1	5	5	250
tert-Butylbenzene	98-06-6	0.5	1	5	5	250
sec-Butylbenzene	135-98-8	0.5	1	5	5	250
Carbon disulfide	75-15-0	0.5	1	5	5	250
Carbon tetrachloride	56-23-5	0.5	1	5	5	250
Chlorobenzene	108-90-7	0.5	1	5	5	250
2-Chloro-1,3-Butadiene (Chloroprene)	126-99-8	N/A	N/A	5	5	250
Chloroethane	75-00-3	0.5	1	5	5	250
2-Chloroethyl vinyl ether	110-75-8	N/A	N/A	10	10	500
Chloroform	67-66-3	0.5	1	5	5	250
Chloromethane (Methyl chloride)	74-87-3	0.5	1	5	5	250
3-Chloropropene (Allyl chloride)	107-05-1	N/A	N/A	10	10	500
2-Chlorotoluene	95-49-8	0.5	1	5	5	250
4-Chlorotoluene	106-43-4	0.5	1	5	5	250
Cyclohexane	110-82-7	N/A	1	5	5	250
Cyclohexanone	108-94-1	N/A	N/A	50	50	2500
Dibromochloromethane	124-48-1	0.5	1	5	5	250
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	0.5	1	5	5	250
1,2-Dibromoethane (EDB)	106-93-4	0.5	1	5	5	250
Dibromomethane (Methylene bromide)	74-95-3	0.5	1	5	5	250
1,2-Dichlorobenzene	95-50-1	0.5	1	5	5	250
1,3-Dichlorobenzene	541-73-1	0.5	1	5	5	250

TABLE A-I: CAS Numbers and Reporting Limits (continued)

Analytes	CAS Number	Standard Reporting Limits				
		Trace Aqueous µg/L	Low Level Aqueous µg/L	Aqueous µg/L	Low Soil µg/kg	High Soil µg/kg
1,4-Dichlorobenzene	106-46-7	0.5	1	5	5	250
trans-1,4-Dichloro-2-butene	110-57-6	N/A	N/A	10	10	500
Dichlorodifluoromethane	75-71-8	0.5	1	5	5	250
1,1-Dichloroethane	75-34-3	0.5	1	5	5	250
1,2-Dichloroethane	107-06-2	0.5	1	5	5	250
cis-1,2-Dichloroethene	156-59-2	0.5	1	5	5	250
trans-1,2-Dichloroethene	156-60-5	0.5	1	5	5	250
1,1-Dichloroethene	75-35-4	0.5	1	5	5	250
1,3-Dichloropropane	142-28-9	0.5	1	5	5	250
2,2-Dichloropropane	594-20-7	0.5	1	5	5	250
1,1-Dichloropropene	563-58-6	0.5	1	5	5	250
cis-1,3-Dichloropropene	10061-01-5	0.5	1	5	5	250
trans-1,3-Dichloropropene	10061-02-6	0.5	1	5	5	250
1,2-Dichloropropane	78-87-5	0.5	1	5	5	250
Diisopropyl ether (IPE)	108-20-3	0.5	1	5	5	250
3,3-dimethyl-1-butanol	624-95-3	N/A	N/A	100	N/A	N/A
1,4-Dioxane	123-91-1	5	10	250	250	12,000
Ethyl acetate	141-78-6	N/A	N/A	5	5	250
Ethanol	64-17-5	N/A	N/A	100	N/A	N/A
Ethyl ether	60-29-7	0.5	1	5	5	250
Ethylbenzene	100-41-4	0.5	1	5	5	250
Ethyl methacrylate	97-63-2	N/A	N/A	5	5	250
Ethyl-tert-butyl ether	637-92-3	N/A	N/A	1.0	N/A	N/A
Hexachlorobutadiene	87-68-3	N/A	N/A	5	5	250
Hexane	110-54-3	N/A	0.5	2.5	2.5	125
2-Hexanone	591-78-6	N/A	N/A	10	10	500
Isopropyl acetate	108-21-4	N/A	N/A	2.5	N/A	N/A
Isopropylbenzene	98-82-8	0.5	1	5	5	250
p-Isopropyltoluene	99-87-6	0.5	1	5	5	250
Isobutyl alcohol	78-83-1	N/A	N/A	50	50	2500
Methacrylonitrile	126-98-7	N/A	N/A	5	5	250
Methyl acetate	79-20-9	N/A	N/A	5	5	250
Methylcyclohexane	108-87-2	N/A	N/A	5	5	250
Methyl iodide (Iodomethane)	74-88-4	N/A	N/A	5	5	250
Methyl methacrylate	80-62-6	N/A	N/A	5	5	250
4-Methyl-2-Pentanone	108-10-1	N/A	N/A	10	10	500
Methyl tert butyl ether (MTBE)	1634-04-4	0.5	1	5	5	250
Methylene chloride	75-09-2	0.5	1	5	5	250

TABLE A-I: CAS Numbers and Reporting Limits (continued)

Analytes	CAS Number	Standard Reporting Limits				
		Trace Aqueous µg/L	Low Level Aqueous µg/L	Aqueous µg/L	Low Soil µg/kg	High Soil µg/kg
Naphthalene	91-20-3	0.5	1	5	5	250
2-Nitropropane	79-46-9	N/A	N/A	5	N/A	N/A
n-Butyl acetate	123-86-4	N/A	2	10	N/A	N/A
n-Propylbenzene	103-65-1	0.5	1	5	5	250
Propionitrile (Ethyl cyanide)	107-12-0	NA	NA	50	50	2500
Styrene	100-42-5	0.5	1	5	5	250
tert-amyl Alcohol	75-85-4	N/A	N/A	20	N/A	N/A
tert-amyl-methyl ether	994-05-8	N/A	N/A	10	N/A	N/A
tert-Butyl Alcohol	75-65-0	N/A	N/A	20	N/A	N/A
tert-Buytl Formate	762-75-4	N/A	N/A	5.0	N/A	N/A
1,1,1,2-Tetrachloroethane	630-20-6	0.5	1	5	5	250
1,1,2,2-Tetrachloroethane	79-34-5	0.5	1	5	5	250
Tetrachloroethene	127-18-4	0.5	1	5	5	250
Tetrahydrofuran	109-99-9	NA	NA	10	10	500
Toluene	108-88-3	0.5	1	5	5	250
1,2,4-Trichlorobenzene	120-82-1	0.5	1	5	5	250
1,2,3-Trichlorobenzene	87-61-6	0.5	1	5	5	250
1,1,1-Trichloroethane	71-55-6	0.5	1	5	5	250
1,1,2-Trichloroethane	79-00-5	0.5	1	5	5	250
Trichloroethene	79-01-6	0.5	1	5	5	250
Trichlorofluoromethane	75-69-4	0.5	1	5	5	250
1,1,2-Trichloro-1,2,2-Trifluoroethane (Freon 113)	76-13-1	0.5	1	5	5	250
1,2,3-Trichloropropane	96-18-4	0.5	1	5	5	250
1,2,4-Trimethylbenzene	95-63-6	0.5	1	5	5	250
1,3,5-Trimethylbenzene	108-67-8	0.5	1	5	5	250
Vinyl acetate	108-05-4	0.5	1	5	5	250
Vinyl Chloride	75-01-4	0.5	1	2	10	500
Xylenes (total)	1330-20-7	0.5	1	5	5	250
m/p-Xylenes	108-38-3	0.5	1	5	5	250
o-Xylenes	95-47-6	0.5	1	5	5	250

Note: The reporting limits for non-aqueous waste samples which are prepared by waste dilution are 10x the high soil limits.

TABLE A-II: Reportable Analytes for TCLP

Analytes	CAS Number
Benzene	71-43-2
2-Butanone (MEK)	78-93-9
Carbon tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chloroform	75-00-3
1,2-Dichloroethane	107-06-2
1,1-Dichloroethene	75-35-4
Tetrachloroethene	127-18-4
Trichloroethene	79-01-6
Vinyl Chloride	75-01-4

TABLE A-III: Recommended Instrument Conditions

Mass range	35-300 amu
Scan time	£ 1 second/scan
Initial column temperature / Hold time	45°C for 3.50 minutes
Column temperature program	10 °C/min to 100°C, 25 °C/min to 225 °C
Final column temperature / Hold time	225 °C for 3 minutes
Injector temperature	220°C
MS interface transfer line temperature	225 °C
Source temperature	According to manufacturer's specifications
Carrier gas	Helium at 0.5-1ml/min, constant flow
Trap	OI #10, #11 or #7
Purge gas	Helium at 40ml/min
Purge time	11 min
Sample heater	50 °C soils, ambient or heated for waters
Dry purge	0 min
Desorb preheat	180°C
Desorb	200°C for 1 min
Bake	210°C for 5-7 min
Transfer line/Valve temperature	110ºC-140ºC

TABLE A-IV: BFB Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15 – 40% of mass 95
75	30 – 60% of mass 95
95	Base peak, 100% relative abundance
96	5 – 9% of mass 95
173	Less than 2% of mass 174
174	Greater than 50 % of mass 95
175	5 – 9% of mass 174
176	Greater than 95% but less than 101% of mass 174
177	5 – 9 % of mass 176

TABLE A-V: Characteristic Ions

Analyte	Primary	Secondary	Tertiary
Acetone	43	58	
Acetonitrile	40	39	41
Acrolein	56	55	
Acrylonitrile	53	52	51
Benzene	78		
Benzyl chloride	91	126	
Bromobenzene	156	77	158
Bromochloromethane	128	49	130
Bromodichloromethane	83	85	
Bromoform	173	175	
Bromomethane (Methyl bromide)	94	96	
2-Butanone (MEK)	72	43	57
n-Butyl acetate	43	56	
n-Butylbenzene	91	92	134
tert-Butylbenzene	119	91	134
sec-Butylbenzene	105	134	
Carbon disulfide	76	78	
Carbon tetrachloride	119	117	121
Chlorobenzene	112	77	114
2-Chloro-1,3-Butadiene (Chloroprene)	53	88	51
Chloroethane	64	66	
2-Chloroethyl vinyl ether	63	65	106
Chloroform	83	85	
Chloromethane (Methyl chloride)	50	52	
3-Chloropropene (Allyl chloride)	76	78	41
4-Chlorotoluene	126	91	
Cyclohexane	41	56	
Cyclohexanone	55	69	98
Dibromochloromethane	129	127	
1,2-Dibromo-3-chloropropane (DBCP)	75	155	157
1,2-Dibromoethane (EDB)	107	109	
Dibromomethane (Methylene bromide)	93	174	95
2-Chlorotoluene	91	126	

TABLE A-V: Characteristic Ions (continued)

Analyte	Primary	Secondary	Tertiary
1,2-Dichlorobenzene	146	148	111
1,3-Dichlorobenzene	146	148	111
1,4-Dichlorobenzene	146	148	111
trans-1,4-Dichloro-2-butene	53	88	
Dichlorodifluoromethane	85	87	
1,1-Dichloroethane	63	65	
1,2-Dichloroethane	62	64	
cis-1,2-Dichloroethene	96	61	
trans-1,2-Dichloroethene	96	61	98
1,1-Dichloroethene	96	61	63
1,2-Dichloropropane	63	76	
1,3-Dichloropropane	76	78	
2,2-Dichloropropane	77	79	
1,1-Dichloropropene	75	110	77
cis-1,3-Dichloropropene	75	77	
trans-1,3-Dichloropropene	75	77	
Diisopropyl ether (IPE)	87	59	69
3,3-dimethyl-1-butanol	57	69	
1,4-Dioxane	88	58	
Ethyl acetate	43	88	
Ethanol	45	46	
Ethyl ether	59	45	74
Ethylbenzene	106	91	
Ethyl methacrylate	69	41	
Ethyl-tert-butyl ether	59	87	
Hexachlorobutadiene	225	223	227
Hexane	57	43	86
2-Hexanone	43	58	
Isopropyl acetate	43	61	87
Isopropylbenzene	105	120	
p-Isopropyltoluene	119	134	
Isobutyl alcohol	42	43	41
Methacrylonitrile	67	41	39
Methyl acetate	43	74	
Methyl iodide (Iodomethane)	142	127	
Methyl methacrylate	41	69	
4-Methyl-2-Pentanone	43	58	
Methyl tertiary butyl ether (MTBE)	73	57	

TABLE A-V: Characteristic Ions (continued)

Analyte	Primary	Secondary	Tertiary
Methylcyclohexane	83	55	98
Methylene chloride	84	49	51
Naphthalene	128		
2-Nitropropane	43	41	
n-Propylbenzene	91	120	
Propionitrile (Ethyl cyanide)	54	52	55
Styrene	104	78	
tert-amyl Alcohol	59	73	55
tert-amyl-methyl ether	87	73	
tert-Butyl Alcohol	59	57	
tert-Butyl Formate	59	57	
1,1,1,2-Tetrachloroethane	131	133	
1,1,2,2-Tetrachloroethane	83	85	
Tetrachloroethene	164	129	131
Tetrahydrofuran	42	72	
Toluene	92	91	
1,2,4-Trichlorobenzene	180	182	145
1,2,3-Trichlorobenzene	180	182	145
1,1,1-Trichloroethane	97	101	
1,1,2-Trichloroethane	97	83	85
Trichloroethene	130	95	97
Trichlorofluoromethane	101	103	
1,1,2-Trichloro-1,2,2-Trifluoroethane (Freon 113)	101	103	151
1,2,3-Trichloropropane	110	75	77
1,2,4-Trimethylbenzene	105	120	
1,3,5-Trimethylbenzene	105	120	
Vinyl Acetate	86	44	
Vinyl Chloride	62	64	
m/p-Xylenes	106	91	
o-Xylenes	106	91	
Xylenes (total)	Summation	Summation	
*Pentafluorobenzene (IS)	168		
*1,4-Difluorobenzene (IS)	114		
*Chlorobenzene-d5 (IS)	117	82	119
*1,4-Dichlorobenzene-d4 (IS)	152	150	
$1,2-Dichloroethane-d4 (SS)	65	102	
$Toluene-d8 (SS)	98		
$4-Bromofluorobenzene (SS)	95	174	176

TABLE A-VI: VOA Target Compounds and Surrogates with Corresponding Internal Standards for Quantitation

Group 1: Pentafluorobenzene		
Dichlorodifluoromethane	Allyl Chloride	Propionitrile
Chloromethane	Methylene Chloride	Ethyl acetate
Vinyl Chloride	Tert-Butyl Alcohol	Methacrylonitrile
Bromomethane	Acrylonitrile	Bromochloromethane
Chloroethane	trans-1,2-Dichloroethene	Tetrahydrofuran
Trichlorofluoromethane	tert-Butyl methyl ether (MTBE)	Chloroform
Ethanol	Hexane	Tert-Butyl Formate
Ethyl ether	1,1-Dichloroethane	1,1,1-Trichloroethane
Acrolein	Vinyl acetate	Cyclohexane
Acetone	Diisopropyl ether (IPE)	1,1-Dichloropropene
1,1-Dichloroethene	Chloroprene	Carbon Tetrachloride
1,1,2-Trichloro-1,2,2-Trifluoroethane (Freon 113)	Ethyl-Tert-Butyl Ether	Isobutyl Alcohol
	cis-1,2-Dichloroethene	Tert-Amyl Alcohol
Methyl Iodide	2-Butanone (MEK)	Dibromofluoromethane (Surrogate, DOD only)
Carbon Disulfide	2,2-Dichloropropane	
Acetonitrile		
Methyl acetate		
Group 2: 1,4-Difluorobenzene		
Benzene	1,2-Dichloropropane	2-Chloroethylvinyl ether
Isopropyl Acetate	Dibromomethane	cis-1,3-Dichloropropene
1,2-Dichloroethane	Methyl Methacrylate	4-Methyl-2-pentanone
Tert-Amyl Methyl Ether	1,4-Dioxane	Toluene
Trichloroethene	Bromodichloromethane	1,2-Dichloroethane-d4 (Surrogate)
Methylcyclohexane	2-nitropropane	
Group 3: Chlorobenzene-d5		
trans-1,3-Dichloropropene	3,3-Dimethyl-1-butanol	Bromoform
Ethyl Methacrylate	1,2-Dibromoethane (EDB)	Isopropylbenzene
1,1,2-Trichloroethane	Chlorobenzene	Cyclohexanone
Tetrachloroethene	1,1,1,2-Tetrachloroethane	Toluene-d8 (Surrogate)
1,3-Dichloropropane	Ethylbenzene	Bromofluorobenzene (Surrogate)
2-Hexanone	m+p-Xylene	
Dibromochloromethane	o-Xylene	
n-Butyl acetate	Styrene	
Group 4: 1,4-Dichlorobenzene-d4		
Isopropylbenzene	1,3,5-Trimethylbenzene	Benzyl Chloride
Cyclohexanone	4-Chlorotoluene	n-Butybenzene
Bromobenzene	tert-Butylbenzene	1,2-Dichlorobenzene
1,1,2,2-Tetrachloroethane	1,2,4-Trimethylbenzene	1,2-Dibromo-3-chloropropane
1,2,3-Trichloropropane	sec-Butylbenzene	1,2,4-Trichlorobenzene
trans-1,4-Dichloro-2-butene	1,3-Dichlorobenzene	Hexachlorobutadiene
n-Propylbenzene	1-Isopropyltoluene	Naphthalene
2-Chlorotoluene	1,4-Dichlorobenzene	1,2,3-Trichlorobenzene

TABLE A-VII: 8260B/C/D LCS/MS/MSD Compounds

LCS Compounds	Spiking Level in Extract (mg/L)
Acetone	100
Acetonitrile	500
Acrolein	500
Acrylonitrile	100
Benzene	50
Benzyl chloride	50
Bromobenzene	50
Bromochloromethane	50
Bromodichloromethane	50
Bromoform	50
Bromomethane (Methyl bromide)	50
2-Butanone (MEK)	100
n-Butyl Acetate	50
n-Butylbenzene	50
tert-Butylbenzene	50
sec-Butylbenzene	50
Carbon disulfide	50
Carbon tetrachloride	50
Chlorobenzene	50
2-Chloro-1,3-Butadiene (Chloroprene)	50
Chloroethane	50
2-Chloroethyl vinyl ether	50
Chloroform	50
Chloromethane (Methyl chloride)	50
3-Chloropropene (Allyl chloride)	50
2-Chlorotoluene	50
4-Chlorotoluene	50
Cyclohexane	50
Cyclohexanone	500
Dibromochloromethane	50
1,2-Dibromo-3-chloropropane (DBCP)	50
1,2-Dibromoethane (EDB)	50
Dibromomethane (Methylene bromide)	50
1,2-Dichlorobenzene	50
1,3-Dichlorobenzene	50
1,4-Dichlorobenzene	50
trans-1,4-Dichloro-2-butene	50
Dichlorodifluoromethane	50
1,1-Dichloroethane	50
1,2-Dichloroethane	50
cis-1,2-Dichloroethene	50
trans-1,2-Dichloroethene	50

TABLE A-VII: 8260B/C/D LCS/MS/MSD Compounds (continued)

LCS Compounds	Spiking Level in Extract (mg/L)
1,1-Dichloroethene	50
1,3-Dichloropropane	50
2,2-Dichloropropane	50
1,1-Dichloropropene	50
cis-1,3-Dichloropropene	50
trans-1,3-Dichloropropene	50
1,2-Dichloropropane	50
Diisopropyl ether (IPE)	50
3,3-dimethyl-1-butanol	1000
1,4-Dioxane	500
Ethylbenzene	50
Ethanol	5000
Ethyl acetate	50
Ethyl ether	50
Ethyl methacrylate	50
Ethyl-tert-butyl ether	50
Hexachlorobutadiene	50
Hexane	50
2-Hexanone	100
Isopropyl acetate	50
Isopropylbenzene	50
p-Isopropyltoluene	50
Isobutyl alcohol	500
Methacrylonitrile	250
Methyl acetate	50
Methylcyclohexane	50
Methyl iodide (Iodomethane)	50
Methyl methacrylate	50
4-Methyl-2-Pentanone	100
Methyl tertiary butyl ether (MTBE)	50
Methylene chloride	50
Naphthalene	50
2-Nitropropane	50
n-Propylbenzene	50
Propionitrile (Ethyl cyanide)	500
Styrene	50
Tert-amyl Alcohol	1000
Tert-amyl-methyl ether	50
Tert-Butyl Alcohol	1000
Tert-Butyl Formate	250
1,1,1,2-Tetrachloroethane	50
1,1,2,2-Tetrachloroethane	50

TABLE A-VII: 8260B/C/D LCS/MS/MSD Compounds (continued)

LCS Compounds	Spiking Level in Extract (mg/L)
Tetrachloroethene	50
Tetrahydrofuran	50
Toluene	50
1,2,4-Trichlorobenzene	50
1,2,3-Trichlorobenzene	50
1,1,1-Trichloroethane	50
1,1,2-Trichloroethane	50
Trichloroethene	50
Trichlorofluoromethane	50
1,1,2-Trichloro-1,2,2-Trifluoroethane (Freon 113)	50
1,2,3-Trichloropropane	50
1,2,4-Trimethylbenzene	50
1,3,5-Trimethylbenzene	50
Vinyl acetate	50
Vinyl Chloride	50
m/p-Xylenes	50
o-Xylenes	50
Xylenes (total)	100

TABLE A-VIII: TCLP LCS/MS/MSD Compounds

LCS Compounds	Spiking Level in Sample (mg/L)
Benzene	50
2-Butanone (MEK)	100
Carbon tetrachloride	50
Chlorobenzene	50
Chloroform	50
1,2-Dichloroethane	50
1,1-Dichloroethene	50
Tetrachloroethene	50
Trichloroethene	50
Vinyl chloride	50

Recovery limits for the LCS and for matrix spikes are generated from historical data. Refer to Appendix C and D for recovery limits.

TABLE A-IX: 8260B/C/D Surrogate Compounds

Surrogate Compounds	Spiking Level in extract (mg/L or mg/kg)
1,2-Dichloroethane-d4	50
Toluene-d8	50
4-Bromofluorobenzene	50

TABLE A-X: Calibration Levels, Aqueous (µg/L) and Solid (µg/kg), Regular Level

Aqueous Analyte	Level 1	Level 2	Level 3	Level 4	Level 5
Acetone	10	40	100	200	400
Acetonitrile	50	200	500	1000	2000
Acrolein	50	200	500	1000	2000
Acrylonitrile	10	40	100	200	400
Benzene	5.0	20	50	100	200
Benzyl chloride	5.0	20	50	100	200
Bromobenzene	5.0	20	50	100	200
Bromochloromethane	5.0	20	50	100	200
Bromodichloromethane	5.0	20	50	100	200
Bromoform	5.0	20	50	100	200
Bromomethane (Methyl bromide)	5.0	20	50	100	200
2-Butanone (MEK)	10	40	100	200	400
n-Butyl acetate	5.0	20	50	100	200
n-Butylbenzene	5.0	20	50	100	200
tert-Butylbenzene	5.0	20	50	100	200
sec-Butylbenzene	5.0	20	50	100	200
Carbon disulfide	5.0	20	50	100	200
Carbon tetrachloride	5.0	20	50	100	200
Chlorobenzene	5.0	20	50	100	200
2-Chloro-1,3-Butadiene (Chloroprene)	5.0	20	50	100	200
Chloroethane	5.0	20	50	100	200
2-Chloroethyl vinyl ether	5.0	20	50	100	200
Chloroform	5.0	20	50	100	200
Chloromethane (Methyl chloride)	5.0	20	50	100	200
3-Chloropropene (Allyl chloride)	5.0	20	50	100	200
2-Chlorotoluene	5.0	20	50	100	200
4-Chlorotoluene	5.0	20	50	100	200
Cyclohexane	5.0	20	50	100	200
Cyclohexanone	50	200	500	1000	2000
Dibromochloromethane	5.0	20	50	100	200
1,2-Dibromo-3-chloropropane (DBCP)	5.0	20	50	100	200
1,2-Dibromoethane (EDB)	5.0	20	50	100	200
Dibromomethane (Methylene bromide)	5.0	20	50	100	200
1,2-Dichlorobenzene	5.0	20	50	100	200
1,3-Dichlorobenzene	5.0	20	50	100	200
1,4-Dichlorobenzene	5.0	20	50	100	200
trans-1,4-Dichloro-2-butene	5.0	20	50	100	200
Dichlorodifluoromethane	5.0	20	50	100	200

TABLE A-X: Calibration Levels, Aqueous (µg/L) and Solid (µg/kg), Regular Level (continued)

Aqueous Analyte	Level 1	Level 2	Level 3	Level 4	Level 5
1,1-Dichloroethane	5.0	20	50	100	200
1,2-Dichloroethane	5.0	20	50	100	200
cis-1,2-Dichloroethene	5.0	20	50	100	200
trans-1,2-Dichloroethene	5.0	20	50	100	200
1,1-Dichloroethene	5.0	20	50	100	200
1,3-Dichloropropane	5.0	20	50	100	200
2,2-Dichloropropane	5.0	20	50	100	200
1,1-Dichloropropene	5.0	20	50	100	200
cis-1,3-Dichloropropene	5.0	20	50	100	200
trans-1,3-Dichloropropene	5.0	20	50	100	200
1,2-Dichloropropane	5.0	20	50	100	200
Diisopropyl ether (IPE)	5.0	20	50	100	200
3,3-dimethyl-1-butanol	100	400	1000	2000	4000
1,4-Dioxane	50	200	500	1000	2000
Ethanol	500	2000	5000	10000	20000
Ethyl acetate	5.0	20	50	100	200
Ethyl ether	5.0	20	50	100	200
Ethylbenzene	5.0	20	50	100	200
Ethyl methacrylate	5.0	20	50	100	200
Ethyl-tert-butyl ether	5.0	20	50	100	200
Hexachlorobutadiene	5.0	20	50	100	200
Hexane	5.0	20	50	100	200
2-Hexanone	10	40	100	200	400
Isopropyl acetate	5.0	20	50	100	200
Isopropylbenzene	5.0	20	50	100	200
p-Isopropyltoluene	5.0	20	50	100	200
Isobutyl alcohol	50	200	500	1000	2000
Methacrylonitrile	25.0	100	250	500	1000
Methyl acetate	5.0	20	50	100	200
Methylcyclohexane	5.0	20	50	100	200
Methyl iodide (Iodomethane)	5.0	20	50	100	200
Methyl methacrylate	5.0	20	50	100	200
4-Methyl-2-Pentanone	10	40	100	200	400
Methyl tertiary butyl ether (MTBE)	5.0	20	50	100	200
Methylene chloride	5.0	20	50	100	200
Naphthalene	5.0	20	50	100	200
2-Nitropropane	5.0	20	50	100	200
n-Propylbenzene	5.0	20	50	100	200
Propionitrile (Ethyl cyanide)	50	200	500	1000	2000

TABLE A-X: Calibration Levels, Aqueous (µg/L) and Solid (µg/kg), Regular Level (continued)

Aqueous Analyte	Level 1	Level 2	Level 3	Level 4	Level 5
Styrene	5.0	20	50	100	200
Tert-amyl Alcohol	100	400	1000	2000	4000
Tert-amyl-methyl ether	5.0	20	50	100	200
Tert-Butyl Alcohol	100	400	1000	2000	4000
Tert-Butyl Formate	25	100	250	500	1000
Tetrahydrofuran	5.0	20	50	100	200
Toluene	5.0	20	50	100	200
1,2,4-Trichlorobenzene	5.0	20	50	100	200
1,2,3-Trichlorobenzene	5.0	20	50	100	200
1,1,1-Trichloroethane	5.0	20	50	100	200
1,1,2-Trichloroethane	5.0	20	50	100	200
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	5.0	20	50	100	200
Trichloroethene	5.0	20	50	100	200
Trichlorofluoromethane	5.0	20	50	100	200
1,2,3-Trichloropropane	5.0	20	50	100	200
1,2,4-Trimethylbenzene	5.0	20	50	100	200
1,3,5-Trimethylbenzene	5.0	20	50	100	200
Vinyl acetate	5.0	20	50	100	200
Vinyl Chloride	5.0	20	50	100	200
Xylenes (total)	10	40	100	200	400
m/p-Xylenes	5.0	20	50	100	200
o-Xylenes	5.0	20	50	100	200

TABLE A-XI: Low Level Calibration Levels, Aqueous (µg/L)

Low Level Aqueous Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Acetone	1.0	2.0	10	40	100	200	400
Acetonitrile	5.0	10	50	200	500	1000	2000
Acrolein	5.0	10	50	200	500	1000	2000
Acrylonitrile	1.0	2.0	10	40	100	200	400
Benzene	0.5	1.0	5.0	20	50	100	200
Benzyl chloride	0.5	1.0	5.0	20	50	100	200
Bromobenzene	0.5	1.0	5.0	20	50	100	200
Bromochloromethane	0.5	1.0	5.0	20	50	100	200
Bromodichloromethane	0.5	1.0	5.0	20	50	100	200
Bromoform	0.5	1.0	5.0	20	50	100	200
Bromomethane (Methyl bromide)	0.5	1.0	5.0	20	50	100	200
2-Butanone (MEK)	1.0	2.0	10	40	100	200	400
n-Butyl acetate	0.5	1.0	5.0	20	50	100	200
n-Butylbenzene	0.5	1.0	5.0	20	50	100	200
tert-Butylbenzene	0.5	1.0	5.0	20	50	100	200
sec-Butylbenzene	0.5	1.0	5.0	20	50	100	200
Carbon disulfide	0.5	1.0	5.0	20	50	100	200
Carbon tetrachloride	0.5	1.0	5.0	20	50	100	200
Chlorobenzene	0.5	1.0	5.0	20	50	100	200
2-Chloro-1,3-Butadiene (Chloroprene)	0.5	1.0	5.0	20	50	100	200
Chloroethane	0.5	1.0	5.0	20	50	100	200
2-Chloroethyl vinyl ether	0.5	1.0	5.0	20	50	100	200
Chloroform	0.5	1.0	5.0	20	50	100	200
Chloromethane (Methyl chloride)	0.5	1.0	5.0	20	50	100	200
3-Chloropropene (Allyl chloride)	0.5	1.0	5.0	20	50	100	200
2-Chlorotoluene	0.5	1.0	5.0	20	50	100	200
4-Chlorotoluene	0.5	1.0	5.0	20	50	100	200
Cyclohexane	0.5	1.0	5.0	20	50	100	200
Cyclohexanone	5	10	50	200	500	1000	2000
Dibromochloromethane	0.5	1.0	5.0	20	50	100	200
1,2-Dibromo-3-chloropropane (DBCP)	0.5	1.0	5.0	20	50	100	200
1,2-Dibromoethane (EDB)	0.5	1.0	5.0	20	50	100	200
Dibromomethane (Methylene bromide)	0.5	1.0	5.0	20	50	100	200
1,2-Dichlorobenzene	0.5	1.0	5.0	20	50	100	200
1,3-Dichlorobenzene	0.5	1.0	5.0	20	50	100	200
1,4-Diclorobenzene	0.5	1.0	5.0	20	50	100	200

TABLE A-XI: Low Level Calibration Levels, Aqueous (µg/L) (continued)

Low Level Aqueous Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
trans-1,4-Dichloro-2-butene	0.5	1.0	5.0	20	50	100	200
Dichlorodifluoromethane	0.5	1.0	5.0	20	50	100	200
1,1-Dichloroethane	0.5	1.0	5.0	20	50	100	200
1,2-Dichloroethane	0.5	1.0	5.0	20	50	100	200
cis-1,2-Dichloroethene	0.5	1.0	5.0	20	50	100	200
trans-1,2-Dichloroethene	0.5	1.0	5.0	20	50	100	200
1,1-Dichloroethene	0.5	1.0	5.0	20	50	100	200
1,3-Dichloropropane	0.5	1.0	5.0	20	50	100	200
2,2-Dichloropropane	0.5	1.0	5.0	20	50	100	200
1,1-Dichloropropene	0.5	1.0	5.0	20	50	100	200
cis-1,3-Dichloropropene	0.5	1.0	5.0	20	50	100	200
trans-1,3-Dichloropropene	0.5	1.0	5.0	20	50	100	200
1,2-Dichloropropane	0.5	1.0	5.0	20	50	100	200
Diisopropyl ether (IPE)	0.5	1.0	5.0	20	50	100	200
3,3-dimethyl-1-butanol	NA	20	100	400	1000	2000	4000
1,4-Dioxane	5.0	10	50	200	500	1000	2000
Ethanol	NA	100	500	2000	5000	10000	20000
Ethyl acetate	0.5	1.0	5.0	20	50	100	200
Ethyl ether	0.5	1.0	5.0	20	50	100	200
Ethylbenzene	0.5	1.0	5.0	20	50	100	200
Ethyl methacrylate	0.5	1.0	5.0	20	50	100	200
Ethyl-tert-butyl ether	NA	1.0	5.0	20	50	100	200
Hexachlorobutadiene	0.5	1.0	5.0	20	50	100	200
2-Hexanone	1.0	2.0	10	40	100	200	400
Isopropylbenzene	0.5	1.0	5.0	20	50	100	200
p-Isopropyltoluene	0.5	1.0	5.0	20	50	100	200
Isobutyl alcohol	5.0	10	50	200	500	1000	2000
Methacrylonitrile	2.5	5.0	25	100	250	500	1000
Methyl acetate	0.5	1.0	5.0	20	50	100	200
Methylcyclohexane	0.5	1.0	5.0	20	50	100	200
Methyl iodide (Iodomethane)	0.5	1.0	5.0	20	50	100	200
Methyl methacrylate	0.5	1.0	5.0	20	50	100	200
4-Methyl-2-Pentanone	1.0	2.0	10	40	100	200	400
Methyl tertiary butyl ether (MTBE)	0.5	1.0	5.0	20	50	100	200
Methylene chloride	0.5	1.0	5.0	20	50	100	200
Naphthalene	0.5	1.0	5.0	20	50	100	200
n-Propylbenzene	0.5	1.0	5.0	20	50	100	200
Propionitrile (Ethyl cyanide)	5.0	10	50	200	500	1000	2000
Styrene	0.5	1.0	5.0	20	50	100	200
Tert-amyl Alcohol	NA	20	100	400	1000	2000	4000
Tert-amyl-methyl ether	NA	1.0	5.0	20	50	100	200

TABLE A-XI: Low Level Calibration Levels, Aqueous (µg/L) (continued)

Low Level Aqueous Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Tert-Butyl Alcohol	NA	20	100	400	1000	2000	4000
Tert-Buytl Formate	NA	5.0	25	100	250	500	1000
1,1,1,2-Tetrachloroethane	0.5	1.0	5.0	20	50	100	200
1,1,2,2-Tetrachloroethane	0.5	1.0	5.0	20	50	100	200
Tetrachloroethene	0.5	1.0	5.0	20	50	100	200
Tetrahydrofuran	0.5	1.0	5.0	20	50	100	200
Toluene	0.5	1.0	5.0	20	50	100	200
1,2,4-Trichlorobenzene	0.5	1.0	5.0	20	50	100	200
1,2,3-Trichlorobenzene	0.5	1.0	5.0	20	50	100	200
1,1,1-Trichloroethane	0.5	1.0	5.0	20	50	100	200
1,1,2-Trichloroethane	0.5	1.0	5.0	20	50	100	200
Trichloroethene	0.5	1.0	5.0	20	50	100	200
Trichlorofluoromethane	0.5	1.0	5.0	20	50	100	200
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	0.5	1.0	5.0	20	50	100	200
1,2,3-Trichloropropane	0.5	1.0	5.0	20	50	100	200
1,2,4-Trimethylbenzene	0.5	1.0	5.0	20	50	100	200
1,3,5-Trimethylbenzene	0.5	1.0	5.0	20	50	100	200
Vinyl acetate	0.5	1.0	5.0	20	50	100	200
Vinyl Chloride	0.5	1.0	5.0	20	50	100	200
m/p-Xylenes	0.5	1.0	5.0	20	50	100	200
o-Xylenes	0.5	1.0	5.0	20	50	100	200
Xylenes (total)	1.0	2.0	10	40	100	200	400

TABLE A-XII: ICV Acceptance Criteria

Analyte	%Difference or %Drift
Acetone	± 30
Acetonitrile	± 30
Acrolein	± 30
Acrylonitrile	± 30
Benzene	± 30
Benzyl chloride	± 30
Bromobenzene	± 30
Bromochloromethane	± 30
Bromodichloromethane	± 30
Bromoform	± 30
Bromomethane (Methyl bromide)	± 30
2-Butanone (MEK)	± 30
n-Butyl acetate	± 30
n-Butylbenzene	± 30
tert-Butylbenzene	± 30
sec-Butylbenzene	± 30
Carbon disulfide	± 30
Carbon tetrachloride	± 30
Chlorobenzene	± 30
2-Chloro-1,3-Butadiene (Chloroprene)	± 30
Chloroethane	± 30
2-Chloroethyl vinyl ether	± 30
Chloroform	± 30
Chloromethane (Methyl chloride)	± 30
3-Chloropropene (Allyl chloride)	± 30
2-Chlorotoluene	± 30
4-Chlorotoluene	± 30
Cyclohexane	± 30
Cyclohexanone	± 40
Dibromochloromethane	± 30
1,2-Dibromo-3-chloropropane (DBCP)	± 30
1,2-Dibromoethane (EDB)	± 30
Dibromomethane (Methylene bromide)	± 30
1,2-Dichlorobenzene	± 30
1,3-Dichlorobenzene	± 30
1,4-Dichlorobenzene	± 30
trans-1,4-Dichloro-2-butene	± 30
Dichlorodifluoromethane	± 30
1,1-Dichloroethane	± 30
1,2-Dichloroethane	± 30

TABLE A-XII: ICV Acceptance Criteria (continued)

Analyte	%Difference or %Drift
cis-1,2-Dichloroethene	± 30
trans-1,2-Dichloroethene	± 30
1,1-Dichloroethene	± 30
1,3-Dichloropropane	± 30
2,2-Dichloropropane	± 30
1,1-Dichloropropene	± 30
cis-1,3-Dichloropropene	± 30
trans-1,3-Dichloropropene	± 30
1,2-Dichloropropane	± 30
Diisopropyl ether (IPE)	± 30
3,3-dimethyl-1-butanol	± 30
1,4-Dioxane	± 30
Ethanol	± 30
Ethyl acetate	± 30
Ethyl ether	± 30
Ethylbenzene	± 30
Ethyl methacrylate	± 30
Ethyl-tert-butyl ether	± 30
Hexachlorobutadiene	± 30
Hexane	± 30
2-Hexanone	± 30
Isopropyl acetate	± 30
Isopropylbenzene	± 30
p-Isopropyltoluene	± 30
Isobutyl alcohol	± 30
Methacrylonitrile	± 30
Methyl acetate	± 30
Methylcyclohexane	± 30
Methyl iodide (Iodomethane)	± 30
Methyl methacrylate	± 30
4-Methyl-2-Pentanone	± 30
Methyl tertiary butyl ether (MTBE)	± 30
Methylene chloride	± 30
Naphthalene	± 30
2-Nitropropane	± 30
n-Propylbenzene	± 30
Propionitrile (Ethyl cyanide)	± 30
Styrene	± 30
Tert-amyl Alcohol	± 30
Tert-amyl-methyl ether	± 30
Tert-Butyl Alcohol	± 30
Tert-Butyl Formate	± 30

TABLE A-XII: ICV Acceptance Criteria (continued)

Analyte	%Difference or %Drift
1,1,1,2-Tetrachloroethane	± 30
Tetrachloroethene	± 30
Tetrahydrofuran	± 30
Toluene	± 30
1,2,4-Trichlorobenzene	± 30
1,2,3-Trichlorobenzene	± 30
1,1,1-Trichloroethane	± 30
1,1,2-Trichloroethane	± 30
Trichloroethene	± 30
Trichlorofluoromethane	± 30
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	± 30
1,2,3-Trichloropropane	± 30
1,2,4-Trimethylbenzene	± 30
1,3,5-Trimethylbenzene	± 30
Vinyl acetate	± 30
Vinyl Chloride	± 30
Xylenes (total)	± 30
m/p-Xylenes	± 30
o-Xylenes	± 30

TABLE A-XIII: CCV Acceptance Criteria

Analyte	%Difference or %Drift
Acetone	20
Acetonitrile	20
Acrolein	20
Acrylonitrile	20
Benzene	20
Benzyl chloride	20
Bromobenzene	20
Bromochloromethane	20
Bromodichloromethane	20
Bromoform	20
Bromomethane (Methyl bromide)	20
2-Butanone (MEK)	20
n-Butyl acetate	20
n-Butylbenzene	20
tert-Butylbenzene	20
sec-Butylbenzene	20
Carbon disulfide	20
Carbon tetrachloride	20
Chlorobenzene	20
2-Chloro-1,3-Butadiene (Chloroprene)	20
Chloroethane	20
2-Chloroethyl vinyl ether	20
Chloroform	20
Chloromethane (Methyl chloride)	20
3-Chloropropene (Allyl chloride)	20
2-Chlorotoluene	20
4-Chlorotoluene	20
Cyclohexane	20
Cyclohexanone	20
Dibromochloromethane	20
1,2-Dibromo-3-chloropropane (DBCP)	20
1,2-Dibromoethane (EDB)	20
Dibromomethane (Methylene bromide)	20
1,2-Dichlorobenzene	20
1,3-Dichlorobenzene	20
1,4-Dichlorobenzene	20
trans-1,4-Dichloro-2-butene	20
Dichlorodifluoromethane	20
1,1-Dichloroethane	20
1,2-Dichloroethane	20
cis-1,2-Dichloroethene	20

TABLE A-XIII: CCV Acceptance Criteria (continued)

Analyte	%Difference or %Drift
trans-1,2-Dichloroethene	20
1,1-Dichloroethene	20
1,3-Dichloropropane	20
2,2-Dichloropropane	20
1,1-Dichloropropene	20
cis-1,3-Dichloropropene	20
trans-1,3-Dichloropropene	20
1,2-Dichloropropane	20
Diisopropyl ether (IPE)	20
3,3-dimethyl-1-butanol	20
1,4-Dioxane	20
Ethanol	20
Ethyl acetate	20
Ethyl ether	20
Ethylbenzene	20
Ethyl methacrylate	20
Ethyl-tert-butyl ether	20
Hexachlorobutadiene	20
Hexane	20
2-Hexanone	20
Isopropyl acetate	20
Isopropylbenzene	20
p-Isopropyltoluene	20
Isobutyl alcohol	20
Methacrylonitrile	20
Methyl acetate	20
Methylcyclohexane	20
Methyl iodide (Iodomethane)	20
Methyl methacrylate	20
4-Methyl-2-Pentanone	20
Methyl tertiary butyl ether (MTBE)	20
Methylene chloride	20
Naphthalene	20
2-Nitropropane	20
n-Propylbenzene	20
Propionitrile (Ethyl cyanide)	20
Styrene	20
Tert-amyl Alcohol (TAA)	20
Tert-amyl-methyl ether (TAME)	20
Tert-Butyl Alcohol (TBA)	20
Tert-Butyl Formate (TBF)	20
1,1,1,2-Tetrachloroethane	20

TABLE A-XIII: CCV Acceptance Criteria (continued)

Analyte	%Difference or %Drift
1,1,2,2-Tetrachloroethane	20
Tetrachloroethene	20
Tetrahydrofuran	20
Toluene	20
1,1,1-Trichloroethane	20
1,1,2-Trichloroethane	20
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	20
Trichloroethene	20
Trichlorofluoromethane	20
1,2,3-Trichloropropane	20
1,2,4-Trimethylbenzene	20
1,3,5-Trimethylbenzene	20
Vinyl acetate	20
Vinyl Chloride	20
Xylenes (total)	20
m/p-Xylenes	20
o-Xylenes	20

TABLE A-XIV: Aqueous LCS Recovery Limits

Aqueous LCS/D Analyte	Lower % Recovery Limit	Upper % Recovery Limit
Acetone	*60	*140
Acetonitrile	*60	*140
Acrolein	*60	*140
Acrylonitrile	70	130
Benzene	70	130
Benzyl chloride	*60	*140
Bromobenzene	70	130
Bromochloromethane	70	130
Bromodichloromethane	70	130
Bromoform	70	130
Bromomethane (Methyl bromide)	70	130
2-Butanone (MEK)	70	130
n-Butyl acetate	70	130
n-Butylbenzene	70	130
tert-Butylbenzene	70	130
sec-Butylbenzene	70	130
Carbon disulfide	70	130
Carbon tetrachloride	70	130
Chlorobenzene	70	130
2-Chloro-1,3-Butadiene (Chloroprene)	70	130
Chloroethane	70	130
2-Chloroethyl vinyl ether	70	130
Chloroform	70	130
Chloromethane (Methyl chloride)	*60	*140
3-Chloropropene (Allyl chloride)	70	130
2-Chlorotoluene	70	130
4-Chlorotoluene	70	130
Cyclohexane	70	130
Cyclohexanone	70	130
Dibromochloromethane	70	130
1,2-Dibromo-3-chloropropane (DBCP)	70	130
1,2-Dibromoethane (EDB)	70	130
Dibromomethane (Methylene bromide)	70	130
1,2-Dichlorobenzene	70	130
1,3-Dichlorobenzene	70	130
1,4-Dichlorobenzene	70	130
trans-1,4-Dichloro-2-butene	70	130
Dichlorodifluoromethane	*60	*140
1,1-Dichloroethane	70	130
1,2-Dichloroethane	70	130
cis-1,2-Dichloroethene	70	130
trans-1,2-Dichloroethene	70	130

TABLE A-XIV: Aqueous LCS Recovery Limits (continued)

Aqueous LCS/LCSD Analyte	Lower % Recovery Limit	Upper % Recovery Limit
1,1-Dichloroethene	70	130
1,3-Dichloropropane	70	130
2,2-Dichloropropane	70	130
1,1-Dichloropropene	70	130
cis-1,3-Dichloropropene	70	130
trans-1,3-Dichloropropene	70	130
1,2-Dichloropropane	70	130
Diisopropyl ether (IPE)	70	130
3,3-dimethyl-1-butanol	70	130
1,4-Dioxane	*60	*140
Ethanol	70	130
Ethyl acetate	70	130
Ethyl ether	70	130
Ethylbenzene	70	130
Ethyl methacrylate	70	130
Ethyl tert butyl ether	70	130
Hexachlorobutadiene	*60	*140
Hexane	70	130
2-Hexanone	70	130
Isopropyl acetate	70	130
Isopropylbenzene	70	130
p-Isopropyltoluene	70	130
Isobutyl alcohol	*60	*140
Methacrylonitrile	70	130
Methyl acetate	70	130
Methylcyclohexane	70	130
Methyl iodide (Iodomethane)	70	130
Methyl methacrylate	70	130
4-Methyl-2-Pentanone	70	130
Methyl tertiary butyl ether (MTBE)	70	130
Methylene chloride	70	130
Naphthalene	70	130
2-Nitropropane	70	130
n-Propylbenzene	70	130
Propionitrile (Ethyl cyanide)	70	130
Styrene	70	130
Tert-amyl Alcohol (TAA)	70	130
Tert-amyl-methyl-ether (TAME)	70	130
Tert-Butyl Alcohol (TBA)	70	130
Tert-Butyl Formate (TBF)	70	130
1,1,1,2-Tetrachloroethane	70	130
1,1,2,2-Tetrachloroethane	70	130
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	70	130
Tetrachloroethene	70	130
Tetrahydrofuran	70	130
Toluene	70	130

TABLE A-XIV: Aqueous LCS Recovery Limits (continued)

Aqueous LCS/D Analyte	Lower % Recovery Limit	Upper % Recovery Limit
1,2,4-Trichlorobenzene	70	130
1,2,3-Trichlorobenzene	70	130
1,2,3-Trichloropropane	70	130
1,1,1-Trichloroethane	70	130
1,1,2-Trichloroethane	70	130
Trichloroethene	70	130
Trichlorofluoromethane	70	130
1,2,4-Trimethylbenzene	70	130
1,3,5-Trimethylbenzene	70	130
Vinyl acetate	*60	*140
Vinyl Chloride	70	130
Xylenes (total)	70	130
m/p-Xylenes	70	130
o-Xylenes	70	130

* These compounds are poor performing analytes which cannot exceed 60-140% recovery. All others cannot exceed 70-130%.

TABLE A-XV: Solid LCS Recovery Limits

Solid LCS/LCSD Analyte	Lower % Recovery Limit	Upper % Recovery Limit
Acetone	70	130
Acetonitrile	*60	*140
Acrolein	*60	*140
Acrylonitrile	*60	*140
Benzene	70	130
Benzyl chloride	70	130
Bromobenzene	70	130
Bromochloromethane	70	130
Bromodichloromethane	70	130
Bromoform	70	130
Bromomethane (Methyl bromide)	70	130
2-Butanone (MEK)	70	130
n-Butylbenzene	70	130
tert-Butylbenzene	70	130
sec-Butylbenzene	70	130
Carbon disulfide	70	130
Carbon tetrachloride	70	130
Chlorobenzene	70	130
2-Chloro-1,3-Butadiene	70	130
2-Chloroethylvinyl ether	70	130
Chloroethane	70	130
Chloroform	70	130
Chloromethane (Methyl chloride)	*60	*140
3-Chloropropene (Allyl chloride)	*60	*140
2-Chlorotoluene	70	130
4-Chlorotoluene	70	130
Cyclohexane	70	130
Cyclohexanone	70	130
Dibromochloromethane	70	130
1,2-Dibromo-3-chloropropane	70	130
1,2-Dibromoethane (EDB)	70	130
Dibromomethane (Methylene	70	130
1,2-Dichlorobenzene	70	130
1,3-Dichlorobenzene	70	130
1,4-Dichlorobenzene	70	130
trans-1,4-Dichloro-2-butene	70	130
Dichlorodifluoromethane	*60	*140
1,1-Dichloroethane	70	130
1,2-Dichloroethane	70	130
cis-1,2-Dichloroethene	70	130
trans-1,2-Dichloroethene	70	130
1,1-Dichloroethene	70	130

TABLE A-XV: Solid LCS Recovery Limits (continued)

Solid LCS/LCSD Analyte	Lower % Recovery Limit	Upper % Recovery Limit
1,3-Dichloropropane	70	130
2,2-Dichloropropane	70	130
1,1-Dichloropropene	70	130
cis-1,3-Dichloropropene	70	130
trans-1,3-Dichloropropene	70	130
1,2-Dichloropropane	70	130
Diisopropyl ether (IPE)	70	130
1,4-Dioxane	*60	*140
Ethyl acetate	70	130
Ethyl ether	70	130
Ethylbenzene	70	130
Ethyl methacrylate	70	130
Hexachlorobutadiene	70	130
Hexane	70	130
2-Hexanone	70	130
Isopropylbenzene	70	130
p-Isopropyltoluene	70	130
Isobutyl alcohol	*60	*140
Methacrylonitrile	70	130
Methyl acetate	70	130
Methylcyclohexane	70	130
Methyl iodide (Iodomethane)	70	130
Methyl methacrylate	70	130
4-Methyl-2-Pentanone	70	130
Methyl tertiary butyl ether (MTBE)	70	130
Methylene chloride	70	130
Naphthalene	70	130
n-Propylbenzene	70	130
Propionitrile (Ethyl cyanide)	70	130
Styrene	70	130
1,1,1,2-Tetrachloroethane	70	130
1,1,2,2-Tetrachloroethane	70	130
Tetrachloroethene	70	130
Tetrahydrofuran	70	130
Toluene	70	130
1,2,4-Trichlorobenzene	70	130
1,2,3-Trichlorobenzene	70	130
1,1,1-Trichloroethane	70	130
1,1,2-Trichloroethane	70	130
Trichloroethene	70	130
Trichlorofluoromethane	70	130

TABLE A-XV: Solid LCS Recovery Limits (continued)

Solid LCS/LCSD Analyte	Lower % Recovery Limit	Upper % Recovery Limit
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	70	130
1,2,3-Trichloropropane	70	130
1,2,4-Trimethylbenzene	70	130
1,2,3-Trichloropropane	70	130
1,2,4-Trimethylbenzene	70	130
1,3,5-Trimethylbenzene	70	130
Vinyl acetate	70	130
Vinyl Chloride	70	130
Xylenes (total)	70	130
m/p-Xylenes	70	130
o-Xylene	70	130

* These compounds are poor performing analytes which cannot exceed 60-140% recovery. All others cannot exceed 70-130%.

APPENDIX B: LEVEL 1 AND LEVEL 2 DATA REVIEW GUIDANCE

All data generated by the instruments in Target must be reviewed for accuracy and compliance with Shealy SOPs and the Shealy QAMP to include calibrations, quality controls, and client samples.

B.1. Level 1 review shall be done by the analyst running each instrument or another qualified analyst when following completion of the batch. Review may be started while the batch is running provided all QC passes and there is no requirement for a closing CCV (i.e. 6200, DoD QSM).

B.2. Level 2 review must be completed after level 1 review is done. Level 2 must be completed by a peer.

B.3. ***Level 1 review of the analytical batch***

- Verification of passing tune using the correct BFB method.
- Ensure last sample is within 12-hours of BFB injection.
- Target review of CCV, LCS, and Method Blank for correct integrations.
- Review of Target Sample and ISTD reports for CCV acceptability.
- Review of Recovery Reports, and ISTD reports for LCS acceptability.
- Review of Method blank for acceptability of required LOQ for each program area.
- Export sample information from LIMS 4 batch.
- Re-quantitation or reprocessing of samples to ensuring Lab Sample IDs, Client Sample IDs, and dilution factors are correct.
    - For solids, verify the correct sample weight and % moisture.
    - Target review of samples for proper integrations.
    - Mark manual integrations with correct reason code.
- Note any over range/over dilute compounds for reanalysis in run log.
- Target review MS/MSD samples for proper integrations.

- Review Recovery Reports and ISTD reports for acceptability.
- Import data into LIMS 4 batch, calculate, and verify any flags.
- Mark L1 complete
- Generate Nonconformance memo(s), if applicable
- Upload

B.4. ***Level 1 review of initial calibrations***

- Target method zeroed and all analytes reset to “averaged” prior to processing any points.
- Correct peak selection/retention times are verified by processing the midpoint first. (Level 5 for aqueous, level 4 for solid.)
- Target integration events are verified using the lowest level each analyte is reported from. (e.g. integration events for benzene must be set at the level 1 standard, but acetone integration events must be set at the level 3 standard.)
- All levels of the ICAL are processed and target reviewed for proper peak selection and integrations. Manual integrations in initial calibrations should be avoided. Integration events must be auto calculated and/or modified to achieve proper integration for each analyte. This will ensure these analytes are properly detected in client samples while using that method.
- Verify all compounds meet the requirements outlined in Section 10.7.5 including CCCs and SPCCs.
- Note any levels removed, compounds marked linear/weighted linear or failing compounds.
- Disable all points on compounds that fail completely either at the ICAL stage or the ICV stage.
    - Must be designated “Averaged” to avoid unwanted flags in ICAL report.
- Verify reporting parameters for each compound.
- Once completed, save method to “Source Methods.”
- Reprocess Initial Calibration Verification (ICV) and check for acceptability according to Table A-XII.

- Reprocess all data files that have been completed with the newly saved method.

B.5. ***Level 2 review of the analytical batch***

- Verify passing tune and tune window.
- Verify files are looking to correct location for Internal Standard quantitation according to program area requirements.
- Verify CCV, LCS, and Method Blanks meet acceptable criteria.
- Verify manual integrations, recovery reports, and ISTD reports for all samples within the batch.
- Verify all data uploaded to LIMS are calculated properly.

B.6. ***Level 2 review of initial calibrations will include any appropriate requirements of Appendix B, Section 4 in addition to the following items***:

- Verification of CCCs and SPCCs acceptability according to appropriate method requirements.
- Verify using Initial Calibration Report that all compounds have enough points in their curves and they pass appropriate RSD/r2 requirements.
- Ensure any compound that fails calibration is disabled.
- Verify method has correct %D or %Difference and response factor criteria
- Verify correct surrogate addition loop size.
- Verify files are looking to correct location for Internal Standard quantitation according to program area requirements.

APPENDIX C: REQUIREMENTS FOR METHOD 624.1

C.1. Method 624.1 is required for demonstration of compliance with NPDES wastewater discharge permits. The standard analyte list and reporting limits are listed in Table C-I.

C.1.1. For demonstration of capability (DOC), the 'X' and 's' criteria in Table C-II must be met.

C.2. This method can be applied only to aqueous matrices. If 2-chloroethyl vinyl ether is an analyte of interest the sample must be collected in unpreserved 40 ml VOC vials and analyzed within 7 days of collection. If acrolein is to be determined, analyze the sample within 3 days. To extend the holding time to 14 days, acidify a separate sample to pH 4 - 5 with HCl. The pH adjustment is not required when analyzing for acrylonitrile if acrolein will not be measured.

C.3. Samples must be checked for the presence of chlorine. Chlorine is checked after the samples are analyzed and is documented on the Chemstation sequence print out. If chlorine is present a nonconformance memo (NCM) must be issued to the project manager.

C.4. The tune period for this method is defined as 12 hours. The tuning criteria are the same as for method 8260B/C/D (Table A-IV).

C.4.1. *Initial calibration curve requirements*

C.4.1.1. The initial calibration curve for this method requires at least five levels.

C.4.1.2. Target® compounds must have RSD ≤ 35%.

C.4.1.3. If this requirement cannot be met, a regression curve must be constructed for the non-compliant compounds.

C.4.2. *Continuing calibration verification (CCV)*

C.4.2.1. A continuing calibration verification standard must be analyzed at the start of every 20 samples. The concentration and acceptance criteria for the CCV are listed in Table C-II. This standard must be prepped from a source different than the calibration and can be used as the LCS for the batch.

C.4.2.2. If the responses for all parameters of interests fall within the designated ranges, analysis of actual samples can begin. If any individual response falls outside the range, repeat the test only for those parameters that failed to meet the calibration acceptance criteria. If the response for a parameter does not fall within the range in this second test, a new calibration curve or RF must be prepared for that parameter.

C.4.2.3. The analyst may choose to proceed with the analysis of sample and report only those parameters that meet the acceptance criteria.

1C.4.2.3.1. Matrix Spike and LCS requirements - A full analyte spike is required for method 624.1. The spiking levels are given in Table C-III. For every batch, one LCS and one MS/MSD are required. Each compound of interest must have an acceptable recovery in either the LCS or MS/MSD. Acceptance criteria are listed in Table C-II.

C.4.3. *Qualitative identification*

C.4.3.1. The mass spectrum for each analyte must be comprised of a minimum of 2 m/z's. Refer to table A-V for primary and secondary m/z's

C.4.3.2. The retention time must fall within ±30 seconds of the retention time of the authentic compound.

C.4.3.3. The relative intensities of ions must agree to within ± 20% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 30% and 70%.)

TABLE C-I: Shealy Method 624.1 Standard Reporting List and Reporting Limits

Analytes	CAS Number	Aqueous (mg/L)
Acetone	67-64-1	20
Acrolein	107-02-8	5
Acrylonitrile	107-13-1	5
Benzene	71-43-2	2
Bromodichloromethane	75-27-4	2
Bromoform	75-25-2	2
Bromomethane (Methyl bromide)	74-83-9	2
Carbon tetrachloride	56-23-5	2
Chlorobenzene	108-90-7	2
Chloroethane	75-00-3	2
2-Chloroethyl vinyl ether	110-75-8	5
Chloroform	67-66-3	2
Chloromethane (Methyl chloride)	74-87-3	2
Cis-1,2-Dichloroethene	156-59-2	2
Cis-1,3-Dichloropropene	10061-01-5	2
Dibromochloromethane	124-48-1	2
1,2-Dichlorobenzene	95-50-1	2
1,3-Dichlorobenzene	541-73-1	2
1,4-Dichlorobenzene	106-46-7	2
1,1-Dichloroethane	75-34-3	2
1,2-Dichloroethane	107-06-2	2
1,1-Dichloroethene	75-35-4	2
1,2-Dichloropropane	78-87-5	2
1,4-Dioxane	123-91-1	250
Ethylbenzene	100-41-4	2
Methylene chloride	75-09-2	2
1,1,2,2-Tetrachloroethane	79-34-5	2
Tetrachloroethene	127-18-4	2
Toluene	108-88-3	2
trans-1,2-Dichloroethene	156-60-5	2
Trans-1,3-Dichloropropene	10061-02-6	2
1,1,1-Trichloroethane	71-55-6	2
1,1,2-Trichloroethane	79-00-5	2
Trichloroethene	79-01-6	2
Trichlorofluoromethane	75-69-4	2
1,2,4-Trimethylbenzene	95-63-6	2
Vinyl Chloride	75-01-4	2
Xylenes (total)	1330-20-7	5

TABLE C-II: Method 624.1 Calibration and QC Acceptance Criteria

Analytes	Standard Conc (µg/L)	%Recovery Range for Ccal (%)	Limit [2] for s (%)	Range [3] For $\bar{X}$ (%)	%Recovery Range for LCS and MS/MSD	Limit for RPD
Acrolein[4]	200	60-140	30	50-150	40-160	60
Acrylonitrile[4]	40	60-140	30	50-150	40-160	60
Benzene	20	65-135	33	75-125	37-151	61
Bromodichloromethane	20	65-135	34	50-140	35-155	56
Bromoform	20	70-130	25	57-156	45-169	42
Bromomethane (Methyl bromide)	20	15-185	90	D-206	D-242	61
Carbon tetrachloride	20	70-130	26	65-125	70-140	41
Chlorobenzene	20	65-135	29	82-137	37-160	53
Chloroethane	20	40-160	47	42-202	14-230	78
2-Chloroethyl vinyl ether	20	D [1]-225.0	130	D-252	D-305	71
Chloroform	20	70-135	32	68-121	51-138	54
Chloromethane (Methyl chloride)	20	D-205	472	D-230	D-273	60
Cis-1,2-Dichloroethene	20	N/A	N/A	N/A	N/A	
Cis-1,3-Dichloropropene	20	25-175	79	5-195	D-227	58
Dibromochloromethane	20	70-135	30	69-133	53-149	50
1,2-Dichlorobenzene	20	65-135	31	59-174	18-190	57
1,3-Dichlorobenzene	20	70-130	24	75-144	59-156	43
1,4-Dichlorobenzene	20	65-135	31	59-174	18-190	57
1,1-Dichloroethane	20	70-130	24	71-143	59-155	40
1,2-Dichloroethane	20	70-130	29	72-137	49-155	49
1,1-Dichloroethene	20	50-150	40	19-212	D-234	32
1,2-Dichloropropane	20	35-165	69	19-181	D-210	55
1,4-Dioxane	200	N/A	N/A	N/A	N/A	
Ethylbenzene	20	60-140	34	75-134	37-162	63
Methylene chloride	20	60-140	192	D-205	D-221	28
1,1,2,2-Tetrachloroethane	20	60-140	36	68-136	46-157	61
Tetrachloroethene	20	70-130	23	65-133	64-148	39
Toluene	20	70-130	22	75-134	47-150	41
trans-1,2-Dichloroethene	20	70-130	27	68-143	54-156	45
Trans-1,3-Dichloropropene	20	50-150	52	38-162	17-183	86
1,1,1-Trichloroethane	20	70-130	21	69-151	52-162	36
1,1,2-Trichloroethane	20	70-130	27	75-136	52-150	45
Trichloroethene	20	65-135	29	75-138	70-157	48
Trichlorofluoromethane	20	50-150	50	45-158	17-181	84
1,2,4-Trimethylbenzene	20	N/A	N/A	N/A	N/A	
Vinyl Chloride	20	5-195	100	D-218	D-251	66
Xylenes	40	N/A	N/A	N/A	N/A	

[1]Based on Table 7 of EPA Method 624.1. D = Detected; result must be greater than zero.
For compounds without acceptance criteria, the criteria for method 8260B/C should be followed.
[2]Standard deviation of four recovery measurements.
[3]Average recovery of four recovery measurements.
Criteria were calculated assuming a QC check sample concentration of 50 µg

TABLE C-III: Method 624.1 LCS and MS Compounds and Spike Concentrations

LCS Compounds	Spiking Level in Extract (mg/L)
Acrolein	200
Acrylonitrile	40
Benzene	20
Bromodichloromethane	20
Bromoform	20
Bromomethane (Methyl bromide)	20
Carbon tetrachloride	20
Chlorobenzene	20
Chloroethane	20
2-Chloroethyl vinyl ether	20
Chloroform	20
Chloromethane (Methyl chloride)	20
Cis-1,3-Dichloropropene	20
Dibromochloromethane	20
1,2-Dichlorobenzene	20
1,3-Dichlorobenzene	20
1,4-Dichlorobenzene	20
1,1-Dichloroethane	20
1,2-Dichloroethane	20
1,1-Dichloroethene	20
1,2-Dichloropropane	20
Ethylbenzene	20
Methylene chloride	20
1,1,2,2-Tetrachloroethane	20
Tetrachloroethene	20
Toluene	20
trans-1,2-Dichloroethene	20
Trans-1,3-Dichloropropene	20
1,1,1-Trichloroethane	20
1,1,2-Trichloroethane	20
Trichloroethene	20
Trichlorofluoromethane	20
Vinyl Chloride	20

APPENDIX D: BATCH QC REQUIREMENT SUMMARY

D.1. ***Summary***

D.1.1. This appendix is added to clarify batch QC requirements.

D.2. ***General Principle***

D.2.1. If two different types of analyses, i.e. soil preserved in sodium bisulfate and soil in reagent water, are combined in the same analytical sequence, an NCM is required.

D.3. ***Method 624.1 QC Requirements***

D.3.1. 12-hour tune window.

D.3.2. Up to 20 samples in a batch.

D.3.3. CCV at 20 µg/L, meets criteria for Target® compounds (Table C-II). Can be the same as LCS below.

D.3.4. Method blank.

D.3.5. Full list LCS (Table C-III) at 20 µg/L, including gaseous compounds, meets the recovery criteria for Target® compounds (Table C-II).

D.3.6. If 8260B/C/D analyses are performed in the same sequence, one CCV, one method blank and one LCS can be analyzed and evaluated for both methods for up to 20 samples. The LCS should be prepared at 20 µg/L.

D.3.7. One MS/MSD per batch at 20 µg/L. (separate MS/MSD or MS/DUP for 8260B/C/D)

D.3.8. Surrogate acceptance limits are 70-130%.

D.4. ***5035/8260B/C/D High Level Sample***

D.4.1. *Preparation Procedure*

D.4.1.1. If samples are not field preserved with methanol, approximately 5 grams of soil must be preserved with 5.0 mL of methanol within 48 hours of sample collection.

D.4.1.2. Add 5 µL of 2500 µg/mL surrogate standards.

D.4.1.3. Shake manually or vortex for approximately 2 minutes.

D.4.1.4. Allow the solvent and solid to separate.

D.4.1.5. Add 100µL (or lesser amount if dilution is required) of the extract into a 43 mL VOC vial containing 5mL reagent water and a stir bar.

D.4.1.6. The sample is ready for analysis.

D.4.2. *QC Requirements and Preparation*

D.4.2.1. An extraction batch is created for up to 20 samples when surrogate addition is complete.

D.4.2.2. A batch must contain a method blank (MB) and an LCS.

D.4.2.3. The MB and LCS contain 5.0 mL of methanol.

D.4.2.4. Add 5.0 µL of 2500 ug/ml surrogate standards to both the MB and LCS. Additionally add 125 µL of 100 µg/mL secondary standards to the LCS.

D.4.2.5. Follow steps 3 through 6 in the preceding section to prepare sample for analysis.

D.4.2.6. Samples can be added to the extraction batch within 24 hours of the initiation of the batch.

D.4.3. *Corrective Actions*

D.4.3.1. Analyst should establish surrogate recovery acceptance criteria for high level soil samples with at least 20 data points. The criteria are updated annually. The range is $\bar{x} \pm 3 * s$, in which $\bar{x}$ is the average recovery of all data points; s is the standard deviation of all data points.

D.4.3.2. If the extraction blank and sample fail to meet the surrogate recovery acceptance criteria, they must be reanalyzed.

D.5. ***Non-aqueous VOC Sample*** (soluble in methanol)

D.5.1. *Prep Procedure*

D.5.1.1. If sample's solubility in methanol is low, refer to the procedure for 5035B High Level Sample Prep Procedure. (Appendix D, Section 4)

D.5.1.2. Weigh approximately 1.0 gram of the sample into a 40-mL vial, add methanol to bring the final volume to 10 mL.

D.5.1.3. Mix the sample thoroughly by inverting the vial at least 3 times.

D.5.1.4. Add 0.86 mL (or lesser amount if dilution is required) of the waste dilution into a 43 mL VOC vial containing approximately 30 mL of reagent water. Fill to the top and slowly invert 3 times.

D.5.1.5. The sample is ready for analysis.

D.5.2. *QC Requirement*

D.5.2.1. A batch contains up to 20 samples.

D.5.2.2. A method blank with 10 mL of methanol should be prepared following the steps outlined in Section D.5.1.

D.5.2.3. Samples can be added to the batch within 24 hours of the initiation of the batch.

D.6. ***TCLP VOC QC Requirements***

D.6.1. A leachate blank is analyzed with each leaching batch.

D.6.2. A matrix spike containing all the TCLP compounds (Table A - VIII) is analyzed for each leaching batch.

D.6.3. A reagent water LCS containing all the TCLP compounds (Table A - VIII) and a leachate blank (reagent water MB) is analyzed for each analytical batch in addition to the regular batch LCS for 8260B/C/D or 624.1.

D.6.4. For aqueous TCLP batches, the regular LCS and MB for 8260B/C/D or 624.1 can be used as the TCLP QC. Only a separate MS is required per leachate batch.

D.7. ***Method SM 6200B-2011 QC Requirements***

D.7.1. 12-hour tune window.

D.7.2. Up to 20 samples in a batch.

D.7.3. Perform a CCV every 20 samples or every 12 hours whichever is more frequent. Criteria 70-130%, except for the gases which are 60-140%.

D.7.4. A closing standard must be analyzed at the end of a batch. The criteria for the closing CCV is 70-130%, except for the gases which are 60-140%. An LCS can be analyzed at the end of a batch in lieu of an 8260B/C/D/624.1 CCV (for this method the LCS and CCV are the same).

D.7.5. Method blank.

D.7.6. One LCS at minimum with each sample batch, which can be the same as the CCV from above.

D.7.7. If 8260B/C/D analyses are performed in the same sequence, one CCV, one method blank and one LCS can be analyzed and evaluated for all methods for up to 20 samples. The CCV should be prepared at 50 µg/L.

D.7.8. One MS and MSD per batch

D.7.9. Internal Standard and Surrogate acceptance limits are 70-130%.

D.8. ***Method 8260C QC Requirements***

D.8.1. See Appendix G for 8260C QC requirements

D.9. ***Method 8260D***

D.9.1. See Appendix H for changes to 8260D compared with 8260C.

APPENDIX E - 1,4-Dioxane by Selected Ion Monitoring

1,4-Dioxane may be detected at concentrations below what is possible by typical full scan electron impact mass spectroscopy by utilizing Selected Ion Monitoring (SIM). Primary and secondary ions are selected for quantitative and monitoring purposes. A heated purge is used to enhance the purge efficiency for 1,4-Dioxane and the associated internal standard (IS) 1,4-Dioxane-d8. An appropriate surrogate is selected to monitor recovery efficiency for the analyses. IS 1,4-Dioxane-d8 is added to each calibration standard, sample and QC sample at 100 ug/L. The surrogate is added to each sample and QC sample at 50 ug/L with a recovery limit of 70-130%. The table below outlines the calibration standard concentrations for 1,4-Dioxane and associated surrogate.

TABLE E-I: Calibration Level Aqueous (µg/L) and Solid (µg/kg)

Aqueous Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
1,4-Dioxane	2	10	25	50	100	150
1,2-Dichloroethane-d4 (surr)	2	10	25	50	100	150

The following table lists the ions selected for monitoring for each analyte of interest:

TABLE E-II: Characteristic Ions

Analyte	Primary Ion	Secondary Ion
1,4-Dioxane	88	58
1,2-Dichloroethane-d4 (surr)	65	102
1,4-difluorobenzene (IS)	1144	NA
1,4-Dioxane-d8 (IS)	96	NA

The following table lists the control limits for each analyte of interest:

TABLE E-III: Aqueous and Solid Control Limits

Analyte	Lower Control Limit	Upper Control Limit
1,4-Dioxane	70	130
1,2-Dichloroethane-d4 (surr)	70	130
1,4-Dioxane-d8 (IS)	50	200

APPENDIX F - PREPARATION OF STANDARDS

Note: Refer to *Working Standard Prep Log – Volatiles* [VOA form ME001KC] and *Working Standard Prep Log – AQ CLP* [VOA form ME001KD] for forms used for standard preparation

TABLE F-I: 8260 Primary Standard (Total volume 5mL in Methanol)

Component	Volume Added (µl)	Final Concentration (µg/mL)
2-CEVE STD	250	100
VINYL ACETATE	250	100
8260/624 ADDITIONS MIX	250	100
KETONES	100	200
8260/624 MEGA MIX	250	100

TABLE F-II: 8260 Primary Extra Standard (Total volume 5mL in Methanol)

Component	Volume Added (µl)	Final Concentration (µg/mL)
CYCLOHEXANONE	250	1000
ACROLEIN	250	1000

TABLE F-III: 8260 Secondary Standard (Total volume 5mL in Methanol)

Component	Volume Added (µl)	Final Concentration (µg/mL)
2-CEVE STD	250	100
VINYL ACETATE	250	100
8260/624 ADDITIONS MIX	250	100
KETONES	100	200
8260/624 MEGA MIX	250	100

TABLE F-IV: 8260 Secondary Extra Standard (Total volume 5mL in Methanol)

Component	Volume Added (µL)	Final Concentration (µg/mL)
CYCLOHEXANONE	250	1000
ACROLEIN	250	1000

TABLE F-V: Modified 8260 Primary Standard for OxyBTEX (Total volume 5mL in Methanol)

Component	Volume Added (µl)	Final Concentration (µg/mL)
8260 SURROGATE STD	200	100
SPCC MIX	250	100
CUSTOM OXYBTEX STD	500	100

TABLE F-VI: Modified 8260 Secondary Standard for OxyBTEX (Total volume 5mL in Methanol)

Component	Volume Added (µl)	Final Concentration (µg/mL)
CUSTOM BTEX+ STD	500	100
Oxygenates STD	250	100

TABLE F-VII: 8260 Internal Standard (Total volume 50mL in Methanol)

Component	Volume Added (ml)	Final Concentration (µg/mL)
8260 INTERNAL STD	5	250

TABLE F-VIII: 8260 Surrogate Standard (Total volume 50mL in Methanol)

Component	Volume Added (ml)	Final Concentration (µg/mL)
8260 SURROGATE STD	5	250

TABLE F-IX: 8260 Primary Gas Standard (Total volume 5mL in Methanol)

Component	Volume Added (µl)	Final Concentration (µg/mL)
GASES STD	250	100

TABLE F-X: 8260 Secondary Gas Standard (Total volume 5mL in Methanol)

Component	Volume Added (µl)	Final Concentration (µg/mL)
GASES STD	250	100

TABLE F-XI: 8260 Primary Pentachloroethane Standard (Total volume 2mL in Methanol)

Component	Volume Added (µl)	Final Concentration (µg/mL)
PENTACHLOROETHANE STD	100	100

TABLE F-XII: 8260 Secondary Pentachloroethane Standard (Total volume 2mL in Methanol)

Component	Volume Added (µl)	Final Concentration (µg/mL)
PENTACHLOROETHANE STD	100	100

APPENDIX G: SW-846 8260C Requirements

G.1. ***ICAL***

G.1.1. No CCC's.

G.1.2. RSD criteria for all target analytes ≤ 20 %.

G.1.3. Minimum RFs as per Table G-1 below.

G.1.4. If linear or non-linear calibration used must verify the RL by re-calculating concentrations in the lowest calibration standard using calibration curve. Acceptance criteria is 70-130% Recovery.

G.1.5. SIM - must monitor a minimum of 2 ions per analyte (primary and confirmation)

G.1.6. Must recalibrate if >10% of targets exceed the %RSD or regression criteria.

G.2. ***ICV***

G.2.1. Acceptance criteria 70-130% Recovery. Difficult analytes must meet 40-160% Recovery. Write NCM for all failures (even difficult compounds outside of the 70-130%).

G.3. ***CCV***

G.3.1. Evaluate minimum RFs specified in Table G-I below.

G.3.2. The IS area counts must be 50-200% of area counts in the associated mid-level ICAL std.

G.3.3. %D ≤20 for all targets (recalibrate if >20% targets exceed the %D criteria).

G.4. ***MB***

G.4.1. No corrective action required if concentration of contaminant in sample is >10X the concentration detected in the method blank.

G.5. ***LCS***

G.5.1. See 8260C for defined difficult compounds.

G.5.2. No second source requirement.

G.6. ***LCSD***

G.6.1. RPD acceptance criteria ≤20 (aqueous and solid).

G.7. ***MS/MSD***

G.7.1. RPD acceptance criteria ≤20% aqueous and ≤30% for solids.

TABLE G-I: Recommended Minimum Relative Response Factor Criteria for Initial and Continuing Calibration Verification

Volatile Compounds	Minimum Response Factor (RF)a	Typical Response Factor (RF)b
Dichlorodifluoromethane	0.100	0.327
Chloromethane	0.100	0.537
Vinyl chloride	0.100	0.451
Bromomethane	0.100	0.255
Chloroethane	0.100	0.254
Trichlorofluoromethane	0.100	0.426
1,1-Dichloroethene	0.100	0.313
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100	0.302
Acetone	0.100	0.151
Carbon disulfide	0.100	1.163
Methyl Acetate	0.100	0.302
Methylene chloride	0.100	0.380
trans-1,2-Dichloroethene	0.100	0.351
cis-1,2-Dichloroethene	0.100	0.376
Methyl tert-Butyl Ether	0.100	0.847
1,1-Dichloroethane	0.200	0.655
2-Butanone	0.100	0.216
Chloroform	0.200	0.557
1,1,1-Trichloroethane	0.100	0.442
Cyclohexane	0.100	0.579
Carbon tetrachloride	0.100	0.353
Benzene	0.500	1.368
1,2-Dichloroethane	0.100	0.443
Trichloroethene	0.200	0.338
Methylcyclohexane	0.100	0.501
1,2-Dichloropropane	0.100	0.382
Bromodichloromethane	0.200	0.424
cis-1,3-Dichloropropene	0.200	0.537
trans-1,3-Dichloropropene	0.100	0.515
4-Methyl-2-pentanone	0.100	0.363
Toluene	0.400	1.577
1,1,2-Trichloroethane	0.100	0.518
Tetrachloroethene	0.200	0.606
2-Hexanone	0.100	0.536
Dibromochloromethane	0.100	0.652
1,2-Dibromoethane	0.100	0.634
Chlorobenzene	0.500	1.733
Ethylbenzene	0.100	2.827
meta-/para-Xylene	0.100	1.080
ortho-Xylene	0.300	1.073
Styrene	0.300	1.916

TABLE G-I: Recommended Minimum Relative Response Factor Criteria for Initial and Continuing Calibration Verification (Continued)

Volatile Compounds	Minimum Response Factor (RF)a	Typical Response Factor (RF)b
Bromoform	0.100	0.413
Isopropylbenzene	0.100	2.271
1,1,2,2-Tetrachloroethane	0.300	0.782
1,3-Dichlorobenzene	0.600	1.408
1,4-Dichlorobenzene	0.500	1.427
1,2-Dichlorobenzene	0.400	1.332
1,2-Dibromo-3-chloropropane	0.050	0.129
1,2,4-Trichlorobenzene	0.200	0.806

APPENDIX H: Changes to 8260D Rev 4 Compared to 8260C Rev. 3 (as pertains to this SOP)

H.1. The term mass was replaced with m/z to reflect what is actually being measured by the detector. Area or height was replaced with response.

H.2. Trichlorotrifluoroethane was split into two isomers: 1,1,2-Trichlorofluoroethane and 1,1,1-Trichlorotrifluoroethane.

H.3. A paragraph was added about performing ICV with an alternate source (Section 7.11.3 of SW-846, Test Methods for Evaluating Solid Waste, June 2018, Revision 4, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry, Method 8260D.

H.4. Tune verification was updated from once every 12 hours to once prior to ICAL (Note: as stated in this SOP, the practice of verifying the tune once every 12 hours will continue). The calibration verification frequency was updated to allow for last initial calibration standard to be the start of 12-hour clock for samples analyzed after initial calibration. Clarification was added that a blank is required after initial calibration and continuing calibration verification. Clarification was added requiring monitoring of ISs in CCVs.

Note: Since numerous methods are covered under this SOP, the current practice will be in most cases to follow the most stringent criteria.

Appendix DoD: DoD/DOE Method Specific Quality Control Requirements

Sections found in this appendix supersede and/or supplement the existing sections of this SOP. In addition to the general method performance criteria, these requirements must be met when analyzing samples for the Department of Defense (DoD) and the Department of Energy (DOE) as stipulated in the DoD/DOE Quality System Manual.

Table B-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of BFB or DFTPP from method.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Performance Check (Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation ≤ 20% for DDT. Benzidine and pentachlorophenol shall be present at their normal responses and shall not exceed a tailing factor of 2.	Correct problem, then repeat performance checks.	Flagging is not appropriate.	No samples shall be analyzed until performance check is within criteria. The DDT breakdown and Benzidine/ pentachlorophenol tailing factors are considered overall system checks to evaluate injector port inertness and column performance and are required regardless of the reported analyte list.
Initial calibration (ICAL) for all analytes (including surrogates)	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	Each analyte must meet one of the three options below: Option 1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; *(continued next page)*	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. No samples shall be analyzed until ICAL has passed.

Table B-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial calibration (ICAL) for all analytes (including surrogates) *(Continued)*		Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.			If the specific version of a method requires additional evaluation (e.g., RFs or low calibration standard analysis and recovery criteria) these additional requirements must also be met.
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Calculated for each analyte and surrogate.
Evaluation of Relative Retention Times (RRT)	With each sample.	RRT of each reported analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	NA.	After maintenance is performed which may affect retention times, RRTs may be updated based on the daily CCV. RRTs shall be compared with the most recently updated RRTs.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 20% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

Table B-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within ± 20% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable calibration verification.	Results may not be reported without valid CCVs. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If the specific version of a method requires additional evaluation (e.g., average RFs) these additional requirements must also be met.
Internal Standards (IS)	Every field sample, standard, and QC sample.	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within – 50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV can be used.	Inspect mass spectrometer and GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the Case Narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	NA.

Table B-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid Method Blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all surrogates and all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	Must contain all surrogates and all analytes to be reported. For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	MSD: Must contain all surrogates and all analytes to be reported. The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.

Table B-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project if available; otherwise use DoD/DOE QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch if sufficient sample material is available. If obvious chromatographic interference is present, reanalysis may not be necessary, but the client must be notified prior to reporting data and the failures must be discussed in the Case Narrative.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the Case Narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.

Additional requirements not required by DoD:

Calibration Check Compounds (CCCs) (8260B only) – The %RSD of the response factors for each CCC in the initial calibration must be ≤ 20% for the initial calibration to be considered valid. This criterion must be met before sample analysis begins. Problems similar to those listed under SPCCs could affect this criterion.

CCC Compounds: 1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl Chloride

Table DoD-I: Volatile Organic Compounds and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous and Low-Level Soil mg/L or mg/kg	High Soil mg/kg
Acetone	67-64-1	20	1000
Acetonitrile	75-05-8	10	500
Acrolein	107-02-8	50	2500
Acrylonitrile	107-13-1	50	2500
Benzene	71-43-2	5	250
Benzyl chloride	100-44-7	5	250
Bromobenzene	108-86-1	5	250
Bromochloromethane	74-97-5	5	250
Bromodichloromethane	75-27-4	5	250
Bromoform	75-25-2	5	250
Bromomethane (Methyl bromide)	74-83-9	5	250
2-Butanone (MEK)	78-93-9	10	500
n-Butyl acetate	123-86-4	10	NA
n-Butylbenzene	104-51-8	5	250
tert-Butylbenzene	98-06-06-	5	250
sec-Butylbenzene	135-98-8	5	250
Carbon disulfide	75-15-0	5	250
Carbon tetrachloride	56-23-5	5	250
Chlorobenzene	108-90-7	5	250
Chloroethane	75-00-3	5	250
2-Chloroethyl vinyl ether	110-75-8	10	500
Chloroform	75-00-3	5	250
Chloromethane (Methyl chloride)	74-87-3	5	250
3-Chloropropene (Allyl chloride)	107-05-1	10	500
2-Chlorotoluene	95-49-8	5	250
4-Chlorotoluene	106-43-4	5	250
Cyclohexane	110-82-7	5	250
Cyclohexanone	108-94-1	50	2500
Dibromochloromethane	124-48-1	5	250
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	5	250
1,2-Dibromoethane (EDB)	106-93-4	5	250
Dibromomethane (Methylene bromide)	74-95-3	5	250
1,2-Dichlorobenzene	95-50-1	5	250
1,3-Dichlorobenzene	541-73-1	5	250
1,4-Dichlorobenzene	106-46-7	5	250
trans-1,4-Dichloro-2-butene	110-57-6	10	500
Dichlorodifluoromethane	75-71-8	5	250
1,1-Dichloroethane	75-34-3	5	250
1,2-Dichloroethane	107-06-2	5	250
cis-1,2-Dichloroethene	156-59-2	5	250
trans-1,2-Dichloroethene	156-60-5	5	250

TABLE DoD-I: Volatile Organic Compounds and Standard Reporting Limits (continued)

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous and Low-Level Soil mg/L or mg/kg	High Soil mg/kg
1,1-Dichloroethene	75-35-4	5	250
1,2-Dichloropropane	78-87-5	5	250
1,3-Dichloropropane	142-28-9	5	250
2,2-Dichloropropane	594-20-7	5	250
1,1-Dichloropropene	563-58-6	5	250
cis-1,3-Dichloropropene	10061-01-5	5	250
trans-1,3-Dichloropropene	10061-02-6	5	250
1,2-Dichloropropane	78-87-5	5	250
Diisopropyl ether	108-20-3	5	250
1,4-Dioxane	123-91-1	250	12,000
Ethanol	64-17-5	100	NA
Ethyl acetate	141-78-6	5	250
Ethyl methacrylate	97-63-2	5	250
Ethyl tert-butyl ether	637-92-3	1.0	NA
Ethylbenzene	100-41-4	5	250
Ethyl methacrylate	97-63-2	5	250
Hexachlorobutadiene	87-68-3	5	250
Hexane	105-54-3	2.5	125
2-Hexanone	591-78-6	10	500
Methyl iodide (Iodomethane)	74-88-4	5	250
Isobutyl alcohol	78-83-1	50	2500
Isopropyl acetate (Acetic acid)	108-21-4	2.5	NA
Isopropylbenzene	98-82-8	5	250
p-Isopropyltoluene	99-87-6	5	250
Methacrylonitrile	126-98-7	5	250
Methyl acetate	79-20-9	5	250
Methyl methacrylate	80-62-6	5	250
4-Methyl-2-Pentanone	108-10-1	10	500
Methyl tertiary butyl ether (MTBE)	1634-04-4	5	250
Methylene chloride	75-09-2	5	250
Naphthalene	91-20-3	5	250
2-Nitropropane	79-46-9	5	NA
n-Propylbenzene	103-65-1	5	250
Propionitrile (Ethyl cyanide)	107-12-0	50	2500
Pentachloroethane	76-01-7	5	5
Styrene	100-42-5	5	250
tert-Amyl Alcohol	75-85-4	100	NA
tert-Amyl methyl ether (TAME)	994-05-8	5	NA
tert-Butyl alcohol	75-65-0	100	NA
tert-Butyl Formate	762-75-4	25	NA
1,1,1,2-Tetrachloroethane	630-20-6	5	250
1,1,2,2-Tetrachloroethane	79-34-5	5	250

TABLE DoD-I: Volatile Organic Compounds and Control Limits (continued)

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous and Low-Level Soil mg/L or mg/kg	High Soil mg/kg
Tetrachloroethene	127-18-4	5	250
Tetrahydrofuran	109-99-9	10	500
Toluene	108-88-3	5	250
1,2,4-Trichlorobenzene	96-18-4	5	250
1,2,3-Trichlorobenzene	95-63-6	5	250
1,1,1-Trichloroethane	71-55-6	5	250
1,1,2-Trichloroethane	79-00-5	5	250
Trichloroethene	79-01-6	5	250
Trichlorofluoromethane	75-69-4	5	250
1,2,3-Trichloropropane	96-18-4	5	250
1,2,4-Trimethylbenzene	95-63-6	5	250
1,3,5-Trimethylbenzene	108-67-8	5	250
Vinyl acetate	108-05-4	5	250
Vinyl Chloride	75-01-4	2	500
Xylenes (total)	1330-20-7	5	250
m/p-Xylenes	108-38-3	5	250
o-Xylenes	95-47-6	5	250

TABLE DoD-II: Volatile Organic Surrogate Compounds and Control Limits

Surrogate Compounds	Concentration in Standard (µg/L or mg/kg)	Aqueous Control Limits		Solid Control Limits	
		Lower	Upper	Lower	Upper
1,2-Dichloroethane-d4	50	81	118	71	136
4-Bromofluorobenzene	50	85	114	79	119
Toluene-d8	50	89	112	85	116
Dibromofluoromethane	50	80	119	78	119

TABLE DoD-III: LCS/MS Control Limits for Volatile Organic Compounds

Analyte	Aqueous Control Limits		Soil Control Limits	
	Lower	Upper	Lower	Upper
Acetone	39	160	36	164
Acetonitrile	50	142	54	143
Acrolein	39	155	47	155
Acrylonitrile	63	135	65	134
Allyl chloride	68	130	68	135
Benzene	79	120	77	121
Benzyl chloride	42	138	64	120
Bromobenzene	80	120	78	121
Bromochloromethane	78	123	78	125
Bromodichloromethane	79	125	75	127
Bromoform	66	130	67	132
Bromomethane (Methyl bromide)	53	141	53	143
2-Butanone (MEK)	56	143	51	148
n-Butyl acetate	69	125	62	128
n-Butylbenzene	75	128	70	128
tert-Butylbenzene	78	124	73	125
sec-Butylbenzene	77	126	73	126
Carbon disulfide	64	133	63	132
Carbon tetrachloride	72	136	70	135
Chlorobenzene	82	118	79	120
Chloroethane	60	138	59	139
2-Chloroethyl vinyl ether	51	139	43	149
Chloroform	79	124	78	123
Chloromethane (Methyl chloride)	50	139	50	136
2-Chlorotoluene	79	122	75	122
4-Chlorotoluene	78	122	72	124
Cyclohexane	71	130	67	131
Cyclohexanone	NA	NA	30	156
Dibromochloromethane	74	126	74	126
1,2-Dibromo-3-chloropropane (DBCP)	62	128	61	132
1,2-Dibromoethane (EDB)	77	121	78	122
Dibromomethane (Methylene bromide)	79	123	78	125
1,2-Dichlorobenzene	80	119	78	121
1,3-Dichlorobenzene	80	119	77	121
1,4-Dichlorobenzene	79	118	75	120
Dichlorodifluoromethane	32	152	29	149
1,1-Dichloroethane	77	125	76	125
1,2-Dichloroethane	73	128	73	128
cis-1,2-Dichloroethene	78	123	77	123
trans-1,2-Dichloroethene	75	124	74	125
1,1-Dichloroethene	71	131	70	131
1,2-Dichloropropane	78	122	76	123

TABLE DoD-III: LCS/MS Control Limits for Volatile Organic Compounds (continued)

Analyte	Aqueous Control Limits		Soil Control Limits	
	Lower	Upper	Lower	Upper
1,3-Dichloropropane	80	119	77	121
2,2-Dichloropropane	60	139	67	133
1,1-Dichloropropene	79	125	76	125
cis-1,3-Dichloropropene	75	124	74	126
trans-1,3-Dichloropropene	73	127	71	130
Trans-1,4-Dichloro-2-butene	43	140	62	136
1,4-Dioxane	59	139	55	138
3,3-Dimethyl-1-butanol	49	133	NA	NA
Diisopropyl ether	67	128	69	127
Ethanol	48	151	45	159
Ethyl acetate	55	138	52	139
Ethyl methacrylate	72	126	69	129
Ethyl tert-butyl ether	70	127	72	126
Ethylbenzene	79	121	76	122
Hexachlorobutadiene	66	134	61	135
Hexane	48	143	45	142
2-Hexanone	57	139	53	145
Isobutyl alcohol	63	133	60	135
Isopropyl acetate	63	133	58	131
Isopropylbenzene	72	131	68	134
p-Isopropyltoluene	77	127	73	127
Methacrylonitrile	63	133	66	132
Methyl acetate	56	136	53	144
Methyl methacrylate	67	128	63	134
4-Methyl-2-Pentanone	67	130	65	135
Methyl iodide (Iodomethane)	69	131	71	131
Methyl tertiary butyl ether (MTBE)	71	124	73	125
Methylcyclohexane	72	132	66	133
Methylene chloride	74	124	70	128
Naphthalene	61	128	62	129
2-Nitropropane	49	136	47	150
n-Propylbenzene	76	126	73	125
Pentachloroethane	69	133	69	135
Propionitrile	64	136	68	134
Styrene	78	123	76	124
tert-Amyl methyl ether [TAME]	68	128	73	126
tert-Butyl alcohol	68	129	68	133
1,1,1,2-Tetrachloroethane	78	124	78	125
1,1,2,2-Tetrachloroethane	71	121	70	124
Tetrachloroethene	74	129	73	128
Tetrahydrofuran	57	133	61	135
Toluene	80	121	77	121

TABLE DoD-III: LCS/MS Control Limits for Volatile Organic Compounds (continued)

Analyte	Aqueous Control Limits		Soil Control Limits	
	Lower	Upper	Lower	Upper
1,2,4-Trichlorobenzene	69	130	67	129
1,2,3-Trichlorobenzene	69	129	66	130
1,1,1-Trichloroethane	74	131	73	130
1,1,2-Trichloroethane	80	119	78	121
1,1,2-Trifluoro-1,2,2-trichloroethane (Freon- 113)	70	136	66	136
Trichloroethene	79	123	77	123
Trichlorofluoromethane	65	141	62	140
1,2,3-Trichloropropane	73	122	73	125
1,2,4-Trimethylbenzene	76	124	75	123
1,3,5-Trimethylbenzene	75	124	73	124
Vinyl acetate	54	146	50	151
Vinyl Chloride	58	137	56	135
Xylenes (total)	79	121	78	124
m/p-Xylenes	80	121	77	124
o-Xylene	78	122	77	123

Note: Marginal exceedances are not allowed for those analytes determined by a project to be target analytes without project specific approval.

TABLE DoD-IV: Characteristic Ions for Volatile Organic Compounds

Analyte	Primary	Secondary	Tertiary
Acetone	58	43	
Acetonitrile	40	39	41
Acrolein	56	55	
Acrylonitrile	53	52	51
Benzene	78		
Benzyl chloride	91	126	
Bromobenzene	77	156	158
Bromochloromethane	128	49	130
Bromodichloromethane	83	85	
Bromoform	173	171	
Bromomethane (Methyl bromide)	94	96	
2-Butanone (MEK)	43	72	
n-Butyl acetate	43	56	
n-Butylbenzene	91	134	
tert-Butylbenzene	119	134	
sec-Butylbenzene	105	134	
Carbon disulfide	76		
Carbon tetrachloride	117	119	
Chlorobenzene	112	114	
Chloroethane	64		
Chloroform	83	85	
Chloromethane (Methyl chloride)	50	52	
2-Chloroethyl vinyl ether	63	65	106
2-Chlorotoluene	91	126	
4-Chlorotoluene	91	126	
Cyclohexane	56	69	84
Cyclohexanone	55	69	98
3-Chloroprene (Ally chloride)	76	78	41
Diisopropyl ether (IPE)	87	59	69
Dibromochloromethane	129	127	
1,2-Dibromo-3-chloropropane (DBCP)	75	157	155
1,2-Dibromoethane (EDB)	107	109	
Dibromomethane (Methylene bromide)	93	174	95
1,2-Dichlorobenzene	146	148	113
1,3-Dichlorobenzene	146	148	113
1,4-Dichlorobenzene	146	148	113
Dichlorodifluoromethane	85		
1,1-Dichloroethane	63	65	
1,2-Dichloroethane	62	64	98
cis-1,2-Dichloroethene	96	61	
trans-1,2-Dichloroethene	96	61	98

TABLE DoD-IV: Characteristic Ions for Volatile Organic Compounds (continued)

Analyte	Primary	Secondary	Tertiary
1,1-Dichloroethene	96	61	63
1,3-Dichloropropane	76	78	
2,2-Dichloropropane	77	79	
1,1-Dichloropropene	75	110	77
cis-1,3-Dichloropropene	75	77	
trans-1,3-Dichloropropene	75	77	
1,2-Dichloropropane	63	76	
1,4-Dioxane	88	58	
Ethanol	45	46	
Ethyl acetate	43	88	
Ethylbenzene	106	91	
Ethyl methacrylate	69	41	
Hexachlorobutadiene	225	223	227
2-Hexanone	43	58	
Isopropylbenzene	105	120	
p-Isopropyltoluene	119	134	
4-Methyl-2-Pentanone	43	58	
Methyl tertiary butyl ether (MTBE)	73	57	
Methylene chloride	84	49	51
Naphthalene	128		
2-Nitropropane	43	41	
n-Propylbenzene	91	120	
Pentachloroethane	167	130	132
Propionitrile (Ethyl cyanide)	54	52	55
Styrene	104	51	78
tert-Amyl methyl ether	87	73	
tert-Butyl alcohol	59	57	
1,1,1,2-Tetrachloroethane	131	133	
1,1,2,2-Tetrachloroethane	83	85	
Tetrachloroethene	164	129	131
Tetrahydrofuran	42	72	
Toluene	92	91	
1,2,4-Trichlorobenzene	180	182	145
1,2,3-Trichlorobenzene	180	182	145
1,1,1-Trichloroethane	97	99	61
1,1,2-Trichloroethane	97	83	85
Trichloroethene	130	95	97
Trichlorofluoromethane	101	103	
1,2,3-Trichloropropane	110	97	
1,2,4-Trimethylbenzene	105	120	
1,3,5-Trimethylbenzene	105	120	
Vinyl acetate	86	44	

TABLE DoD-IV: Characteristic Ions for Volatile Organic Compounds (continued)

Analyte	Primary	Secondary	Tertiary
Vinyl Chloride	62	64	
Xylenes (total)	Summation	Summation	
m/p-Xylenes	106	91	
o-Xylenes	106	91	
Pentafluorobenzene (Internal Standard)	168		
1,2-Dichloroethane-d4 (Surrogate Standard)	65	102	
1,4-Difluorobenzene (Internal Standard)	114		
Toluene-d8 (Surrogate Standard)	98		
Chlorobenzene-d5 (Internal Standard)	117	82	119
4-Bromofluorobenzene (Surrogate Standard)	95	174	176
1,4-Dichlorobenzene-d4 (Internal Standard)	152	150	

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Notes

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Daniel J. Wright
General Manager 1

9/20/2021 3:28:34 PM
Kelly M. Nance
Quality Manager

9/22/2021 1:50:30 PM
Robert Zhu
Technical Specialist

9/22/2021 2:07:54 PM
Bradley E. Belding
Operations Manager

9/27/2021 10:36:34 AM
Kristina P. Bouknight
Environmental Health and
Radiation Safety Officer

9/16/2021 11:59:14 AM
James C. Geiger
Supervisor

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Semivolatile Organic Compounds by GC/MS Analysis
METHOD: EPA Methods 625.1/SW-846 8270D/SW-846 8270E
ISSUER: Pace ENV - Local Quality - WCOL

Table of Contents

1.0 Scope and Application

This standard operating procedure (SOP) describes the laboratory procedure for the determination of semi-volatile organic compounds in extracts by gas chromatography/mass spectrometry (GC/MS) based on EPA methods 625.1, SW-846 8270D, and SW-846 8270E.

1.1 Target Analyte List and Limits of Quantitation (LOQ)

The target analytes and the normal LOQ that can be achieved with this procedure are provided in Table 1, Appendix A. The standard reporting limit (SRL) of this method for determining an individual compound is approximately 0.33 mg/kg (wet weight) for soil/sediment samples, 1-200 mg/kg for wastes (dependent of matrix and method of preparation), and 5 µg/L for groundwater samples. Some compounds have higher reporting limits. Refer to the appendices for analyte specific Limits of Quantitation (LOQs). Reporting limits (RL) will be proportionately higher for sample extracts that require dilution.

LOQ are established in accordance with Pace policy and SOPs for method validation and for the determination of detection limits (DL) and quantitation limits (LOQ). DL and LOQ are routinely verified and updated when needed. The current LOQ for each target analyte that can be determined by this SOP as of the effective date of this SOP is provided in Table 1, Appendix A.

The reporting limit (RL) is the value to which analytes are reported as detected or not detected in the final report. When the RL is less than the lower limit of quantitation (LLOQ), all detects and non-detects at the RL are qualitative. The LLOQ is the lowest point of the calibration curve used for each target analyte.

DL, LOQ, and RL are always adjusted to account for actual amounts used and for dilution.

1.2 Applicable Matrices

1.2.1 EPA method 625.1 is applicable to wastewater.

1.2.2 SW-846 8270D, and SW-846 8270E are applicable to solid waste and aqueous matrices.

1.3 Method specific requirements are located in the appendices.

1.3.1 Direct injection of a sample may be used in limited applications.

1.3.2 Additional compounds may be amenable to this method. If non-standard analytes are required, they must be validated by the procedures described in section 11.3.1.1.2 before sample analysis may begin.

1.3.3 The following compounds may require special treatment when being determined by this method:

- Benzidine can be subject to oxidative losses during solvent concentration and exhibits poor chromatography. Neutral extraction should be performed if this compound is expected.

- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.

- N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.

- Pentachlorophenol, α,α-Dimethylphenethylamine, 4-nitrophenol, benzoic acid, 4,6-dinitro-2-methylphenol, 2,4-dinitrophenol, 4-chloro-3-methyphenol, P-phenylenediamine, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

- Hexachlorophene is not amenable to analysis by this method.

- 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method.

2.0 Summary of Method

Aqueous samples are extracted with methylene chloride using continuous liquid/liquid extraction. Solid samples are extracted with methylene chloride/acetone using ultrasonic extraction or microwave extraction. Waste dilution is used for samples that are miscible with solvent. Extracts are dried, concentrated to a volume of 1, 2, or 5 mL, and analyzed by GC/MS.

When samples are extracted using 3520C, Reduced Volume Extraction (RVE) or microwave 3546, the samples are analyzed by Large Volume Injection (LVI). Qualitative identification of the parameters in the extract is performed using the retention time and the relative abundance of characteristic ions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

Extraction procedures are detailed in the following methods:

- *Continuous Liquid-Liquid Extraction by EPA SW-846 Method 3520C* [EXT SOP ME00155]

- *Microwave Extraction by EPA SW-846 Method 3546* [EXT SOP ME00156]

- *Ultrasonic Extraction by EPA SW-846 Method 3550C* [EXT SOP ME00154]

- *Separatory Funnel Liquid-Liquid Extraction by EPA SW-846 Method 3510C* [EXT SOP ME001IZ]

- *Soxhlet Extraction by EPA SW-846 Method 3540C* [EXT SOP ME001LX]

- *Waste Dilution by EPA SW-846 Method 3580A* [EXT SOP ME00150]

3.0 Interferences

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control Section. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. If interference is detected, it is necessary to determine if the source of interference is in the preparation and/or cleanup of the samples; then take corrective action to eliminate the problem.

3.2 The use of high purity reagents, solvents, and gases helps to minimize interference problems. Refer to the Solvent Purity Check policy [QA Policy ME001J8], for the procedure for testing the purity of solvents used for extraction.

3.3 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample.

3.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples.

3.5 Phthalate contamination is commonly observed in this analysis and its occurrence should be carefully evaluated as an indicator of a contamination problem in the sample preparation step of the analysis.

4.0 Definitions

Refer to the Laboratory Quality Manual [QAMP ME0012K] for a glossary of common lab terms and definitions.

4.1 Non-conformance Memo (NCM) - A form used to document a non-conforming event. An analyst must document a non-conformance memo when a non-conforming event occurs. A non-conforming event may include the reporting of analytical data outside of method or SOP criteria, or when there is a deviation from a written policy or procedure. Information in an NCM may be used by project managers to flag data in the report narrative, or by the quality department to track trends and initiate corrective actions, where applicable. Additional

information on the NCM policy and procedure is located in the Complaints and Nonconformances SOP [QA SOP ME001BO].

4.2 Relative Response Factor (RRF) – A measure of the relative mass spectral response of an analyte compared to its internal standard. RRFs are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples.

4.3 Marginal Exceedance (ME) – A marginal exceedance is defined as being beyond the LCS control limit (three standard deviations), but within the ME limits. ME limits are between three and four standard deviations around the mean.

5.0 Health and Safety

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

6.0 Sample Collection, Preservation, Holding Time, and Storage

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory performs samples collection for samples to be analyzed by this SOP in accordance with the Field Services SOP [FS SOP ME001BS]. Refer to this SOP for these instructions.

The laboratory will provide containers for the collection of samples upon client request for analytical services. Bottle kits are prepared in accordance with the Sample Container Shipping SOP [AD SOP ME001DS].

Requirements for container type, preservation, and field quality control (QC) for the common list of test methods offered by Pace are listed in the Pace-WCOL Analytical Methods List [ME002BS].

General Requirements

Matrix	Routine Container	Min. Sample Amount[1]	Preservation	Holding Time
Aqueous	2 x 250 mL amber glass Teflon-lined lid	250 mL	Thermal: ≤6°C Chemical: None	Collection to Prep: 7 days Prep to Analysis: 40 days
Solid	4 oz glass Teflon-lined lid	50 g	Thermal: ≤6°C Chemical: None	Collection to Prep: 14 days Prep to Analysis: 40 days
Waste	4 oz glass Teflon-lined lid	50 g	Thermal: ≤6°C Chemical: None	Collection to Prep: 14 days Prep to Analysis: 40 days

[1]Minimum amount needed for each discrete analysis.

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with the Sample Receiving SOP [AD SOP ME0013H]. Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are stored at 4 ± 2°C until sample preparation. Prepared samples (extracts, digestates, distillates, other) are stored at 4 ± 2°C until sample analysis.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 28 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

7.0 Equipment and Supplies

Note: Refer to the Major Operational Equipment List [QA Control Log ME001PM] for specific details regarding the equipment and data processing software utilized during this procedure.

7.1 Equipment

7.1.1 Gas Chromatograph/Mass Spectrometer System (GC/MS) – An analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

7.1.2 Column – 30 m x 0.25 mm I.D. 0.5-µm film thickness silicon-coated fused-silica capillary column (Zebron Guardian ZB-SV or equivalent)

7.1.3 Mass Spectrometer – Capable of scanning from mass/charge (m/z) 35 to 500 every one second or less, using 70 volts (nominal) electron energy in the electron impact

ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets all of the criteria in Table V when 50 ng of the GC/MS tuning standard is injected through the GC.

7.1.4 GC/MS Interface – Any GC-to-MS interface that gives acceptable calibration points and achieves acceptable tuning performance criteria may be used.

7.1.5 Data System – A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as the Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIH Mass Spectral Library is recommended.

7.2 Supplies

7.2.1 Syringe – 10 µL or 5 µL Hamilton Laboratory grade syringes or equivalent.

7.2.2 Carrier gas – Ultra high purity helium.

8.0 Reagents and Standards

Note: Other volumes of standards and reagents may be prepared to account for expected usage. As long as all ratios are kept constant, this is not considered a deviation from the approved procedure.

Note: All stored reagents and standards must be labelled as required by the Preparation and Documentation of *Laboratory Standards and Reagents* SOP [QA SOP ME001HG], the *Comprehensive Chemical Hygiene, Safety, and Hazard Communication* Plan [HS SOP ME0012D], and the *Quality Assurance Management Plan* [QAMP ME0012K].

Note: All standards used are purchased from any approved vendor that can provide a certificate of analysis. Follow manufacturer expiration date unless otherwise specified below.

Note: The standards listed in 8.1.2 to 8.1.7 should be refrigerated at ≤ 6 °C when not in use. Refrigeration at –10°C to –20°C may be used if it can be demonstrated that analytes do not fall out of solution at this temperature. Certified solutions purchased from a vendor must be replaced per the manufacturer's recommended expiration date. Stock standard solutions prepared in-house must be replaced after one year or sooner if comparison with QC check samples indicates a problem. When solutions are mixed together, regardless of the source, they must be replaced after the manufacturer's expiration date or one year (whichever occurs first) or sooner if problems are indicated. The standards must be replaced at least once every six months.

8.1 Standards

8.1.1 A minimum five-point calibration curve is prepared. The low point must be at or below the reporting limit. Refer to appendices for typical calibration levels for all analytes. Large Volume Injection (LVI) is a 5 µL injection for which all standards are prepared as outlined in the appendices. Other calibration levels may be used, depending on instrument capability; however, the low standard must support the reporting limit and the high standard defines the range of the calibration.

8.1.2 Initial Calibration Verification standard (ICV) - prepared from a source independent of the source of standards used to prepared for the initial calibration.

8.1.3 Continuing Calibration Verification (CCV) - prepared from the same source as the standards for the initial calibration. The CCV is the verification of the initial calibration that is required during the course of analysis at periodic intervals.

8.1.4 Internal Standard (IS) - compounds in the I.S. Mix are: acenaphthene-d10, chrysene-d12, 1, 4-dichlorobenzene-d5, naphthalene-d8, perylene-d12, and phenanthrene-d10.

8.1.4.1 Internal Standards are added to all standards and extracts to result in 20 ng injected onto the column. For example, if the volume of an extract used were 1 ml, 5 µL of a 4000 µg/mL internal standard solution would be added for a 1 µL injection.

8.1.5 Surrogate Standard Spiking Solution – Prepare as indicated in the preparative methods. All standards prepared for surrogate, LCS, and matrix spiking solutions must be analyzed by GC/MS prior to use for sample extraction. See appropriate preparation SOP. Surrogate compounds are listed in Table IX. DoD surrogate compounds and control limits listed in Table DoD-II.

8.1.5.1 Spiking solution acceptance criteria is 80-120%.

8.1.6 GC/MS Tuning Standard - A methylene chloride solution containing 50 µg/mL of decafluorotriphenylphosphine (DFTPP) is prepared. Pentachlorophenol, benzidine, and DDT, are included in the Tuning Standard at 50 µg/mL.

8.1.7 Laboratory Control Spiking Solution – Prepare as indicated in the preparative methods. All standards prepared for surrogate, LCS, and matrix spiking solutions must be analyzed by GC/MS prior to use for sample extraction. See appropriate preparation SOP. LCS compounds and levels are listed in Tables VII and VIII. DoD specific LCS/MS compounds and control limits are found in Table DoD-III for aqueous samples and DoD-IV for solid samples.

8.1.7.1 Spiking solution acceptance criteria is 80-120%.

8.1.8 Matrix Spike Solution – Prepare as indicated in the preparative methods. See preparation SOP. The matrix spike compounds and levels are the same as the LCS compounds.

9.0 Procedure

9.1 Equipment Preparation

9.1.1 Support Equipment - Incubators, water baths, refrigerator units, freezer units, bottle top dispensers, pipettes, thermometers, ovens, and syringes are maintained and verified as required by the Equipment and Instrumentation SOP [QA SOP ME002JT].

9.1.2 **Instrument**

9.1.2.1 The instrument is tuned for DFTPP (decafluorotriphenylphosphine), calibrated and verified each 12-hour shift with one or more continuing calibration standard(s). Recommended instrument conditions are listed in Table V.

9.1.2.2 Instrument Tuning – At the beginning of every twelve-hour shift when analyses are to be performed, the GC/MS system must be checked to see if acceptable performance criteria (Table V) are achieved for DFTPP.

9.1.2.2.1 Inject 50 ng of the GC/MS tuning standard into the GC/MS system. The mass spectrum of DFTPP must be acquired in the following manner: three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required and must be accomplished using a single scan acquired no more than 20 scans prior to the beginning of the elution of DFTPP. Do not subtract part of the DFTPP peak. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved. Any maintenance or adjustments to the instrument and the acceptable standard file name must be documented to show that the system has returned to control. The performance criteria in Table V must be achieved before any samples, blanks, or standards are analyzed.

9.1.2.2.2 The GC/MS tuning standard should also be used to evaluate the inertness of the chromatographic system. Benzidine and pentachlorophenol should not exhibit excessive tailing. The tailing factor for benzidine and for pentachlorophenol should be < 2.

9.1.2.2.3 DDT is included in the tuning standard, and breakdown must be ≤ 20%. Refer to Section 12 for the appropriate calculations.

9.2 Initial Calibration

NOTE: All standards and extracts are allowed to warm to room temperature before injecting.

NOTE: Depending on the target compounds required by the client, it may be necessary to use more than one calibration standard.

9.2.1 Internal Standard Calibration Procedure – Internal standards are listed in Table VI-A. Use the base peak m/z as the primary m/z for quantitation of the standards. If interferences are noted, use one of the next two most intense masses for quantitation.

9.2.2 Compounds should be assigned to the Internal Standard (IS) with the closest retention time.

9.2.3 Prepare calibration standards at a minimum of five concentration levels for each parameter of interest. Add the internal standard mixture to result in 20 ng on column. (For example, if the volume of the calibration standard used is 1 mL, add 5 µL of the 4000 µg/mL internal standard solution for a 1µL injection). The concentrations of all analytes are listed in Tables X-A and X-B.

9.2.4 Several sub-lists are used to encompass all target compounds listed in this SOP. The compounds in each sub-list match the compounds in each mix. A single sub-list can be recalibrated at any time without recalibrating the entire method list. If a sub-list is recalibrated, all compounds within that mix must be calibrated for all levels of the curve (all levels that apply to each particular compound). A calibration history must be generated from Target to trace when each sub-list has been recalibrated.

9.2.5 Calibration Sequence – Calibration standards must be analyzed in sequence from lowest to highest concentration to minimize the change that carryover from a higher concentration standard will boost the area of a lower concentration standard.

9.2.6 Calibration Point Replacement – replacing a calibration standard may sometimes be needed to correct for a technical problem that occurred during analysis such as power failure, incomplete injection of the standard or similar situation. Replacement of one standard, when analyzed within 24 hours of original analysis time and replacing all analytes in the original standard, is permitted. The replacement of the standard must be approved by the department supervisor/manager; approval and the reason for replacement must documented and kept with the technical record.

9.2.7 **ICAL Evaluation**

9.2.7.1 Curve Fit - Analyze each calibration standard and tabulate the area of the primary characteristic m/z against concentration for each compound and internal standard. Calculate response factors (RF), average response factors, and the percent RSD of the response factors for each compound using the equations in Sections 10.7.1 and 10.7.3. Sample analysis should only begin after these steps have been completed.

9.2.7.2 The Mean Relative Response Factor (RRF) must be calculated for all compounds using the equation in 10.7.1. Calculate the Percent Relative Standard Deviation (% RSD) of the RRF values for the initial calibration using the equation in 12.8.3. If the % RSD of any analyte in the initial calibration is within ± 20% (or ± within

15% for DoD), then average response factor may be used for quantitation. For those analytes with a % RSD >20% (or > 15% for DoD), a linear curve fit may be used. The correlation coefficient must be ≥ 0.995. The Target data system measures the square of the correlation coefficient, which must be ≥ 0.990. The ICAL cannot be forced through zero.

9.2.7.3 Weighting of Data Points - In linear fits, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason, it is preferable to increase the weighting of the lower concentration points. 1/Concentrations weighting (often called 1 / X2 weighting) will improve accuracy at the low end of the curve and should be used.

9.2.7.4 Up to 10% of the compounds in the initial calibration are allowed to fail the criteria in section 9.2.9.2. Any data generated by the use of the RRF from a calibration that does not meet the above criteria must be qualified as estimated and used for screening data only. In order to report non-detects, it must be demonstrated that there is adequate sensitivity to detect the failed compounds at the applicable lower quantitation limit.

9.2.7.5 For any regulatory compliance samples, all target project compounds must meet the initial calibration criteria in section 9.2.

9.2.7.6 Demonstration of sensitivity: For compounds that require a linear fit, the low point of the ICAL must be reprocessed against the current ICAL. Treat the low point as an unknown sample and recalculate the concentrations. The low point concentrations for compounds using a linear fit should be 70-130% of the true value. Compounds that do not meet this criteria should be considered out of control and corrective action must be taken such as re-defining the lower limit of quantitation and/or reporting those “out of control” compounds as estimated values when the concentration is near the low point when the data quality requirements of a project dictate such a need.

9.2.7.7 If more than 10% of the compounds included in the initial calibration exceed the 20% RSD limit and do not meet the minimum correlation coefficient (0.99) for alternative curve fits, the system is considered too reactive and corrective action must be taken and the initial calibration re-analyzed.

9.2.7.8 The relative response factor (RRF) at each calibration concentration for each semi-volatile target compound should be greater than or equal to the compound's acceptable relative minimum response factor listed in Table VI. If this criterion is not met, corrective action should be taken. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front

end of the analytical column, and active sites in the column or chromatographic system.

9.2.7.9 If time remains in the 12-hour period initiated by the DFTPP injection, samples may be analyzed. Otherwise, proceed to continuing calibration.

9.2.7.10 Quantitation is performed using the calibration curve or average response factor from the initial curve, not the continuing calibration

9.2.7.11 Alternatively, either of these two methods may be used to determine calibration function acceptability for linear and non-linear curves. Both procedures refit the calibration data back to the calibration model and evaluates the difference between the measured and the true amounts or concentrations used to create the model.

9.2.7.11.1 % Error - Percent error between the calculated and expected amounts of an analyte should be ≤ 30% for all standards. For some data uses, ≤50% may be acceptable for the lowest calibration point.

9.2.7.11.2 Relative Standard Error (RSE) - The RSE acceptance limit criterion for the calibration model is the same as the RSD limit (Section 9.2.7.2).

9.2.8 Initial Calibration Verification/Second Source Calibration Verification (ICV) - A standard that is prepared from a source independent of standards for the initial calibration. The ICV contains all TCL (target compound list) compounds and its concentration should be at or near the mid-level of the initial calibration. The concentration of the ICV, determined from the analysis, is compared to the known value of the standard to determine the accuracy of the ICAL.

9.2.8.1.1 The recovery should be between 70-130%. For DOD, the recovery should be 80-120%. If any compounds fail to meet the recovery requirements, corrective action will be taken. The ICV must be analyzed after the ICAL and before sample analysis can begin.

9.2.9 Continuing Calibration Verification (Instrument Performance Check) - At the start of each 12-hour period, the GC/MS tuning standard must be analyzed. A 50 ng or less injection of DFTPP must result in a mass spectrum which meets the criteria given in Table V. The tailing factors for benzidine and pentachlorophenol should meet criteria described in Section 9.1.2.2.2.

9.2.9.1 Following a successful DFTPP analysis, the continuing calibration verification (CCV) standard(s) are analyzed. The standards must contain all semi-volatile analytes, including all required surrogates. A continuing calibration standard is analyzed for each sub-list at or near the mid-point of the curve.

9.2.9.2 The following criteria must be met for the continuing calibration to be acceptable:

9.2.9.2.1 Each of the analytes in the calibration verification standard should meet the recommended minimum response factors as noted in Table VI. This same check is applied during the initial calibration.

9.2.9.2.2 If the minimum response factors are not met, the system should be evaluated, and corrective action should be taken before sample analysis begins.

9.2.9.2.3 Calculate the percent drift or percent difference of the response factors for each target analyte using the equations in 10.7.4. The percent drift or percent difference should be less than or equal to 20% for each target analyte.

9.2.9.2.4 Up to 10% of the compounds in the continuing calibration are allowed to fail the criteria in section 9.2.9.2.3. In order to report non-detects, it must be demonstrated that there is adequate sensitivity to detect the failed compounds at the applicable lower quantitation limit. For any regulatory compliance samples, all target project compounds must meet the continuing calibration validation criteria in section 9.2.9.2.3.

9.2.9.2.4.1. Any analyte that is not a target compound is not required to pass the above criteria.

9.2.9.2.5 The internal standard response must be within 50-200% of the response in the mid-level of the initial calibration.

9.2.9.2.5.1. The internal standard retention times must be ± 30 seconds of the retention times in the mid-level of the initial calibration.

9.2.9.2.6 Once the above criteria have been met, sample analysis may begin. Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs. Analysis may proceed until 12 hours from the injection of the DFTPP have passed.

9.3 Analysis

Batch Definition – Batches are defined at the sample preparation stage. Batches should be kept together through the entire 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 *Quality Assurance Management Plan* [QAMP ME0012K] for further details of the batch definition.

9.3.1 All samples must be analyzed using the same instrument conditions as the preceding continuing calibration standard.

9.3.2 Add internal standard to the extract to result in 20 ng injected on column (for example, 5 µL internal standard solution in 1 mL of extract for a 1 µL injection). Mix thoroughly before injection into the instrument.

9.3.3 Inject the sample extract into the GC/MS system using the same injection technique as used for the standards.

9.3.4 The data system will determine the concentration of each analyte in the extract using calculations equivalent to those in Section 10.7. Quantitation is based on the initial calibration, not the continuing calibration.

9.3.5 Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst or automatically by the data system.

9.3.6 Target compounds identified by the data system are evaluated using the criteria listed in Section 10.2.

9.3.7 Library searches of peaks present in the chromatogram that are not target compounds (Tentatively Identified Compounds, TICs) may be performed if requested by the client. They are evaluated using the criteria in Section 10.1.

9.3.8 **Dilutions** – If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range, or close to the CCV concentration. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

9.3.8.1 Guidance for Dilutions Due to Matrix – If the sample is initially run at a dilution and the baseline rise is less than the height of the internal standards, or if individual non-target peaks are less than two times the height of the internal standards, the sample should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgment. For example, samples containing organic acids or are too viscous to allow proper injection into the inlet, may need to be analyzed at a higher dilution to avoid destroying the column or damaging the injection syringe.

9.3.8.2 Reporting Dilutions – The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

9.3.9 Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, refrigerate them at 4 ± 2°C, protected from light in Teflon-lined screw cap vials.

9.3.10 Internal standard criteria for samples

9.3.10.1 If the retention time for any internal standard changes by more than ± 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

9.3.10.2 If the retention time of any internal standard in any sample varies by more than ± 0.1 minute from the preceding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.

9.3.10.3 The internal standard response must be within 50 – 200% of the response in the last continuing calibration standard.

9.3.11 Percent Moisture – Analytical results may be reported as dry or wet weight, as required by the client. Percent mmoisture must be determined if results will be reported as dry weight. Refer to the *Percent Solid and Percent Moisture in Solids and Semi-solids* SOP [EXT SOP ME0013F] for determination of percent moisture.

9.3.12 Troubleshooting Guide

9.3.12.1 Daily Instrument Maintenance - In addition to the checks listed in the instrument maintenance schedule in the PAS - WCOL QAMP, the following daily maintenance should be performed:

- Clip column as necessary.
- Install new injection port liner as necessary.
- Install new septum as necessary.

9.3.12.2 Major Maintenance - A new calibration is necessary following major maintenance. Major maintenance includes changing the column, cleaning the repeller, cleaning the source, and/or replacing the electron multiplier. Refer to the manufacturer's manual for specific guidance.

9.3.12.3 All maintenance performed must be recorded in the instrument maintenance log with each entry dated and initialed by the analyst that is performing the maintenance.

9.3.12.4 Maintenance Items:

9.3.12.4.1 Replace pump oil as needed.

9.3.12.4.2 Change septa that are regularly pierced as often as needed.

9.3.12.4.3 Change gas line dryers as needed.

9.3.12.4.4 Replace electron multiplier as needed.

9.3.12.4.5 Replace GC injector glass liner weekly or as often as needed.

9.3.12.4.6 Replace GC column as needed.

9.3.12.4.7 Check gases daily to ensure that supply is sufficient for the day's activity, and pressures are set as described in the SOP.

9.3.12.4.8 Check daily to ensure the pressure on the primary regulator never runs below 100 psi.

9.3.12.4.9 Clean source as needed

10.0 Data Analysis and Calculations

PAS - WCOL uses Target®/AIM processing software for GC/MS analysis. The processing software is set to err on the side of positive identification. The analyst must ensure that the auto-processed integration is acceptable using the criteria in the sections below.

10.1 Qualitative Identification

10.1.1 Tentatively Identified Compounds (TICS) – For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample spectra with the nearest library searches shall the mass spectral interpretation specialist assign a tentative identification. Guidelines for making tentative identification are:

10.1.1.1 Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

10.1.1.2 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 should be between 30% and 70%.)

10.1.1.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

10.1.1.4 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.

10.1.1.5 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.

10.1.1.6 Automatic background subtraction can severely distort spectra from samples with unresolved hydrocarbons.

10.1.2 Manual Integration

10.1.2.1 Manual changes to automated integration is called manual integration. Manual integration is sometimes necessary to correct inaccurate automated integrations but must never be used to meet QC criteria or to substitute for proper instrument maintenance and/or method set-up. To assure that all manual integrations are performed consistently and are ethically justified, all manual integrations must be performed, reviewed, and recorded in accordance with corporate SOP ENV-SOP-CORQ-0006, Manual Integration.

10.2 Quantitative Identification - An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a reference standard of the suspected compound (reference standard spectrum). Mass spectra for the reference standard may be obtained via the user's GC/MS by analysis of the calibration standards (for reference, the standard must be run within the same twelve-hour period as the sample) or from the NBS library.

10.2.1 The following criteria must be satisfied to verify identification of a compound:

10.2.1.1 The characteristic ions of a compound must maximize in the same scan or within one scan of each other. Selection of a peak by the processing software where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

10.2.1.2 Correspondence of the sample component spectra to the standard component's spectra.

10.2.1.3 The sample quantifying and qualifying ions match the reference standard quantifying and qualifying ions.

10.2.1.3.1 The quantifying and qualifying ions in the reference standard should also be present in the sample.

10.2.1.3.2 The relative intensities of ions should agree to within ± 30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20% and 80%).

10.2.1.3.3 Elution of the sample quantifying and qualifying ions must occur at the same GC retention time as the reference standard ions.

10.2.1.3.4 The retention time (RT) window position is established for each analyte and surrogate once per ICAL. The position shall be set using the mid-point standard of the initial calibration curve.

10.2.1.3.5 The relative retention time (RRT) of each target analyte in each calibration standard must be within ± 0.06 RRT units of the RRT of the standard component.

Note: Care must be taken to ensure that identification of peaks is not hindered by poor resolution. When analytes co-elute the identification criteria might be satisfied but each component spectrum will contain extraneous ions contributed by the co-eluting compound.

10.2.1.4 If the criteria above are not met, the following actions are taken:

10.2.1.4.1 If the qualifying ions are present and the sample component spectra matches the standard component spectra, but the ions fail the relative abundance test, the compound will be flagged with a “Q” by Target®.

10.2.1.4.2 If the ions are not present within the RT or the spectra does not match the standard, then the analyst will mark the sample undetected.

10.2.1.4.3 If the processing software chooses an inappropriate quantifying ion, then the analyst must change the quantifying ion.

10.2.1.4.3.1. If an alternate ion was selected by the analyst, the compound will be flagged with an “H” by Target®.

10.2.1.4.4 If the baseline selection is improper, the correct peak is missed, a coelution is integrated or the peak is partially integrated, the analyst must manually integrate the peak and the compound will be flagged with an “M” by Target/AIM. The analyst must document the reason for the manual integration which can be accomplished via the Target/AIM system.

10.2.1.4.5 If a false positive detection is made by the processing software then the analyst must mark the peak undetected and this action will be flagged with an “!” in Target review.

10.2.1.5 However, if in the technical judgment of the analyst the identification and integration made by the software is correct, the analyst shall report that identification and proceed with quantitation.

10.3 Mass chromatogram searches - Certain compounds are unstable in the calibration standard and cannot be calibrated in the normal way. In particular, the compound hexachlorophene (CAS 10-30-4) falls into this category and is required for Appendix IX analysis. For this analyte, a mass chromatogram search is made.

10.3.1 Hexachlorophene – Display the mass chromatograms for mass 196 and mass 198 for the region of the chromatogram from at least 2 minutes before chrysene-d12 to at least 4 minutes after chrysene-d12. If peaks for both ions coincide then the analyst evaluates the spectrum for the presence of hexachlorophene. No quantitation is possible.

10.4 Anyone evaluating data should be trained to know how to handle isomers with identical mass spectra and close elution times. These include:

Chloronaphthalenes	Phenanthrene, anthracene
Dichlorobenzenes	Fluoranthene, pyrene
Methylphenols, benzyl alcohol	Benzo(b) and (k)fluoranthene
Trichlorophenols	Chrysene, benzo(a)anthracene

10.4.1 Extra precautions concerning these compounds are to more closely scrutinize retention time vs. the calibration standard and also to check that all isomers have distinct retention times.

10.4.2 Isomers are considered resolved if the peaks are at least 50% resolved (i.e., the height of the valley between two isomer peaks is ≤ 50% of the average of the two peak heights, or 1−[valley height]/[average peak height] is ≥50%). This can be accomplished via the Target/AIM system.

10.5 A second category of problem compounds would be the poor responders or compounds that chromatograph poorly. These include:

Benzoic acid	3,3'-Dichlorobenzidine
Chloroanilines	Benzyl alcohol
Nitroanilines	4,6-Dinitro-2-methylphenol
2,4-Dinitrophenol	a,a-Dimethylphenethylamine
4-Nitrophenol	p-Phenylenediamine
Pentachlorophenol	

10.6 Manually checking the integrations would be appropriate for these compounds. All manual integration chromatograms must include the reason for the integration, the date the integration was performed and the initials of the analyst performing the manual integration. This information should be included in the final data package.

Benzoic acid	Pentachlorophenol
Chloroanilines	3,3'-Dichlorobenzidine
Nitroanilines	Benzyl alcohol
2,4-Dinitrophenol	4,6-Dinitro-2-methylphenol
4-Nitrophenol	a,a-Dimethylphenethylamine

p-Phenylenediamine

10.7 Calculations

See the Laboratory Quality Assurance Manual [QAMP ME0012K] for equations for common calculations.

10.7.1 Relative response factor

$RF = (A_x C_{is}) / (A_{is} C_x)$

Where: A_x= Area of the characteristic ion for the compound being measured
A_{is}= Area of the characteristic ion for the specific internal standard
C_x = Concentration of the compound being measured (µg/L)
C_{is} = Concentration of the specific internal standard (µg/L)

10.7.2 Calculation of Tentatively Identified Compounds (TICs) – The calculation of TICs is identical to the above calculations with the following exceptions:

Ax = Area of the total ion chromatogram for the compound being measured
Ais = Area of the total ion chromatogram for the nearest internal standard without interference
RF = 1

10.7.3 Percent Relative Standard Deviation for Initial Calibration

$$\%RSD = \frac{SD}{\overline{RF}} x100$$

Where: $\overline{RF}$ = Mean of RFs from initial calibration for a compound
SD = Standard deviation of RFs from initial calibration for a compound

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(RF_i - \overline{RF})^2}{N-1}}$$

Where: Rf *i* = RF for each of the calibration levels.
N = Number of RF values.

10.7.4 Continuing calibration percent difference and percent drift

$$\% \text{ Diff} = \frac{RF - \overline{RF}}{\overline{RF}} x100$$

Where: RF = Compound's response factor in the CCV

$\overline{RF}$ = Compound's average response factor in the initial calibration.

$$\% \text{ Drift} = \frac{\text{Calc. Conc.} - \text{Theoretical Conc.}}{\text{Theoretical Conc.}} \text{ X } 100$$

10.7.5 Concentration in the extract – The concentration of each identified analyte and surrogate in the extract is calculated from the linear curve fit to the initial calibration points, or from the average RF of the initial calibration.

10.7.5.1 Average response factor – If the %RSD of the response factors in the initial calibration is ≤ 20%, the average response factor from the initial calibration may be used for quantiation.

$$C_{ex} = (R_x C_{is}) / (R_{is})$$

10.7.5.2 Linear fit

$$Cex = [A+B]*[(R_x C_{is})/(R_{is})]$$

Where:
C_{ex} = Concentration in extract, µg/mL
R_x = Response for analyte
R_{is} = Response for internal standard
C_{is} = Concentration of internal standard
A = Intercept
B = Slope

10.7.6 The concentration in the sample is then calculated

10.7.6.1 Aqueous calculation

$$\text{Concentration, µg/L} = (C_{ex} V_t) / (V_0)$$

Where:

V_t = Volume of total extract, µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1 mL extract will mean V_t = 10,000 µL. If half of the base/neutral extract and half of the acid extract are combined, V_t = 2,000 µL.)

V_o = Volume of water extracted (mL).

10.7.6.2 Sediment/Soil, Sludge (on a dry-weight basis) and Waste (normally on a wet-weight basis)

Concentration, g/kg = $(C_{ex}V_t) / (W_sD)$

Where:

W_s = Weight of sample extracted or diluted in grams.
D = (100 - % moisture in sample)/100, for a dry weight basis or 1 for a wet weight basis.

10.7.7 MS/MSD percent recovery

Matrix Spike Recovery = $[(S_{SR} – S_R) / (S_A)]*[100\%]$

Where: S_{SR} = Spike sample result
S_R = Sample result
S_A = Spike added

10.7.8 Relative Percent Difference (RPD) for MS/MSD

RPD = $[(M_{SR} – MSD_R) / (1/2)(M_{SR}+MSD_R)]*[100\%]$

Where: MS_R = Matrix spike result
MSD_R = Matrix spike duplicate result

10.7.9 % Error

$$\% \text{ Error} = \frac{X_i - X_i'}{X_i} \times 100$$

Where:
x_i' = Measured amount of analyte at calibration level i, in mass or concentration units.
x_i = True amount of analyte at calibration level i, in mass or concentration units.

10.7.10 Relative Standard Error (RSE)

$$RSE = \sqrt{\sum_{i=1}^{n}\left[\frac{x_i' - x_i}{x_i}\right]^2 \Big/ (n-p)}$$

Where:
x_i' = Measured amount of analyte at calibration level i, in mass or concentration units.
x_i = True amount of analyte at calibration level i, in mass or concentration units.
p = Number of terms in fitting equation (average = 1, linear = 2)
n= Number of calibration points.

11.0 Quality Control and Method Performance

11.1 Quality Control

The following QC samples are prepared and analyzed with each batch of samples. Refer to appendices for state and/or program specific method performance criteria, which superseded and/or supplement the general method performance criteria prescribed in this

QC Item	Frequency
Method Blank (MB)	1 per batch
Laboratory Control Sample (LCS)	1 per batch
Laboratory Control Sample Duplicate (LCSD)	As needed
Matrix Spike (MS)	1 per batch
Matrix Spike Duplicate (MSD)	1 per batch
Sample Duplicate	As needed

11.2 Instrument QC

The following Instrument QC checks are performed.

QC Item	Frequency
Instrument Tuning	Every 12 hours
Initial Calibration	As needed or if calibration verification is outside of acceptance criteria
Initial Calibration Verification	After calibration
Continuing Calibration Verification	Every 12 hours
Instrument Blank	Every 12 hours if MB is not available

11.2.1 **Method Blank (Laboratory Reagent Blank)** – One method blank (MB) must be processed with each preparation and/or analytical batch. The method blank consists of a similar matrix to the batch of associated samples in which no target analytes or interferences are present at concentrations that impact the analytical results. The method blank is to contain all reagents specific to the method that is carried through the entire analytical procedure,

including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data.

11.2.1.1 The method blank must not contain any analyte of interest at or above ½ the LOQ or project-specific requirements. (Note: see appendices for state or program specific requirements). If the method blank contains an analyte of interest at or above ½ the LOQ, then the method blank and associated samples must be reanalyzed. If the method blank contamination is confirmed, the entire batch must be re-prepared and reanalyzed. If associated samples include J flags with a detection at or above ½ the LOQ, any sample with a reportable result (including J value results) must be re-extracted and re-analyzed. Where permitted by the program area or client, the following exceptions apply. Any method blank that does not meet acceptance criteria, is flagged on the data report with a B flag.

11.2.1.1.1 The method blank detection is not present in the sample.

11.2.1.1.2 The sample concentration is ≥10x the blank concentration.

11.2.1.2 The method blank should have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, corrective action should be taken. The method blank should be reanalyzed if the analyst feels that the failure could be attributed to instrument problems. If the analyst feels that the failure is due to a poor extraction, entire batch must be sent for re-extraction

11.2.2 **Instrument Blank** – Instruments must be evaluated for contamination during each 12-hour analytical run. This may be accomplished by analysis of a method blank. If a method blank is not available, an instrument blank must be analyzed. An instrument blank consists of methylene chloride with the internal standards added. The instrument blank is evaluated in the same way as the method blank.

11.2.3 **Laboratory Control Sample (LCS)** – Analyze an LCS for each batch of samples. Due to the large number of analytes in 8270, not all compounds may be spiked into the LCS. The list of analytes and their concentrations for the LCS are located in table VII and VIII.

11.2.3.1 If any surrogate is outside established control limits, the system is out of control and corrective action must occur.

11.2.3.2 When there are a large number of analytes in the LCS or MS spike, there is a high statistical probability that a few analytes will recover outside of control limits. This may not indicate that the system is out of control, therefore corrective action may not be necessary. The number of allowable marginal exceedances (ME is based on the number of analytes in the LCS. If more analytes exceed the LCS control limits than is allowed, corrective action is necessary, up to and including re-extraction and re-analysis. Specific program or state requirements such as South Carolina may supersede this allowance. The number of allowable marginal exceedances is as follows:

Number of Analytes in LCS	Number Allowed as Marginal Exceedances
>90	5
71-90	4
51-70	3
31-50	2
11-30	1
<11	0

11.2.3.3 LCS compounds are included in Table VII with appropriate spike concentrations.

11.2.3.4 Some clients require a full analyte spike list for the LCS. The added compounds must be statistically evaluated to determine if the in-house recovery limits are achievable and realistic.

11.2.4 **Matrix Spike (MS) and Matrix Spike Duplicate (MSD)** – For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are the same as the LCS. Compare the percent recovery and relative percent difference (RPD) to those in the laboratory specific generated limits.

11.2.4.1 See Section 11.2.3.2 for the number of allowable marginal exceedances. If more analytes exceed the control limits than is allowed, corrective action must occur. The initial corrective action will be to check all calculations. If the calculations are correct, check the recovery of that analyte in the Laboratory Control Sample (LCS). If the recovery of the analyte in the LCS is within limits, then matrix interference has been demonstrated and the laboratory operation may proceed. Analytical reports will show qualifier flags in such cases.

11.2.4.2 If the recovery for any component is outside QC limits for the matrix spike/spike duplicate and the LCS, the laboratory is out of control and corrective action will be taken. Corrective action may include re-preparation and reanalysis of the batch. A Nonconforming memo (NCM) will be generated to document the corrective action taken.

11.2.4.3 Every effort is made to ensure that an MS/MSD pair is included in every batch. In the event that there is insufficient sample to analyze an MS/MSD pair or MS and sample duplicate, an LCSD should be included in the batch.

11.2.4.4 The matrix spike/spike duplicate must be analyzed at the same dilution as the most concentrated reportable analysis as the parent sample (the un-spiked sample).

11.2.5 **Surrogates -** Every sample, blank, and QC sample is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the percent recovery falls within the required limits. The compounds routinely included in the surrogate spiking solution, along with recommended standard concentrations, are listed in Table IX. Refer to Table DoD-II for DoD specific surrogate concentrations and acceptance criteria.

11.2.5.1 If any surrogate is outside the acceptance limits, an NCM must be written and the following corrective actions must take place (except for dilutions ≥ 5X).

- Check all calculations for error.
- Ensure that instrument performance is acceptable. If the system is demonstrated to be out of control, all steps taken to return the system to control must be fully documented as part of the corrective action.
- Recalculate the data and/or reanalyze the extract if either of the above checks reveals a problem.
- If the above corrective actions have taken place and the result is a surrogate that is outside of the limits, re-extract and reanalyze the sample. If the re-extract is outside of limits, flag the data as “Estimated Concentration” due to demonstrated matrix effect.
- It is only necessary to re-extract/reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

11.2.6 **Data Quality Objectives (DQO)** – Refer to project-specific Quality Assurance plans for DQO information.

11.3 Method Performance

11.3.1 Method Validation

11.3.1.1 Detection Limits

Detection limits (DL) and limits of quantitation (LOQ) are established at initial method setup and verified on an on-going basis thereafter. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 Method Validation and Instrument Verification and to the Method Validation SOP [QA Policy ME003BF] for these procedures.

11.3.1.1.1 For the standard analyte list, an acceptable MDL study is required before analysis of samples may begin.

11.3.1.1.2 For non-standard analytes, an MDL study should be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration

11.3.2 Control Limits

11.3.2.1 In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits should be reviewed at least semi-annually, and updated as needed. The recovery limits are mean recovery ± 3 standard deviations. Control limits for compliance with the Department of Defense Quality Systems Manual can be found in Appendix DoD.

11.3.2.1.1 All surrogate, LCS, and MS/MSD recoveries must be entered into PAS - WCOL Laboratory Information Management System (LIMS) when

available or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions not greater than five.

11.3.2.1.2 Refer to the *Trend Analysis of Data Using Control Charts* policy [QA Policy ME001IW] for further details of control limits.

11.3.3 **Level 1 and Level 2 Review** – The items listed below are verified during review.

11.3.3.1 Initial Calibration Review

- Method zeroed to reflect correct times
- New ICAL saved to source method
- Instrument Blank analyzed before the ICAL
- 5 Point minimum calibration-8270D/E,625
- ICAL arranged by sublist, and low to high concentrations
- Calibration compound concentration correct in Target table.
- ICAL summaries are attached to the beginning of each sublist and contain all data file names which comprise that sublist
- Sublists calibrated as one event, i.e., continuously
- ICV is a separate source from the ICAL
- DFTPP meets criteria
- Pentachlorophenol tailing factor criteria met <2
- Benzidine tailing factor criteria met < 2
- Review points removed
- Review levels dropped
- % RSD meets criteria (625.1 ≤ 35) (8270D/E ≤ 20)(DOD <15)
- If linear regression used, then $R^2 \geq 0.99$ (8270D/E), R2 >0.920 (625.1)
- Isomeric pairs checked for correct peak assignment
- Automatic integration verified
- Manual integration verified/initialed/documented
- Standards within expiration date
- % Error (≤35%) or RSE (criteria same as %RSD criteria) meets criteria (RSE only for 625.1)

11.3.3.2 Continuing Calibration Batch Review

- Verify date of Initial Calibration
- ICAL copied from the source method
- DFTPP meets criteria
- Compounds are within 20% of true value for 8270D/E (see Table 625.1-3 for625.1 analysis)
- Isomeric pairs checked for correct peak assignment
- Correct initial calibration used for quantitation
- Manual integration verified documented
- Standards within expiration date

11.3.3.3 Sample Review

- Samples analyzed within tune time 12 hours for 8270E/625.1
- Correct initial calibration used for quantitation
- MB or Instrument Blank analyzed before samples
- Extraction holding time met
- Analytical holding time met
- Internal standard criteria met
- Surrogates within limits
- Results of dilution within upper half of calibration range
- Triple plots reviewed for hits
- Manual integrations verified documented
- LCS within QC limits (method list for 625.1 – see Table 625.1-3)
- Method Blank meets QC limits
- TCLP units and conversion correctly applied (if applicable)
- Instrument Run Log has been reviewed
- Prep batch information correctly imported into Target
- Non-conformances (NCMs) documented, if applicable

11.4 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to the Demonstration of Capability SOP [QA SOP ME001F2] for more information.

12.0 Data Review and Corrective Action

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employee's complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance

with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to the Data Review SOP [QA SOP ME003LP] for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

13.0 Pollution Prevention

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

14.0 Modifications

14.1 A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer

to Pace ENV corporate SOP ENV-SOP-CORQ-0011 Method Validation and Instrument Verification for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

14.1.1 The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

14.1.2 Directions for analysis of PAH only samples are in Attachment PAH.

14.1.3 Department of Defense method requirements are in Appendix DoD.

14.1.4 Direction for analysis of North Carolina samples are in Appendix NC.

14.1.5 Direction for analysis of SIM only samples are in Appendix SIM.

14.1.6 Directions for analysis by EPA method 625.1 are in Appendix 625.1.

14.1.7 Differences between methods 8270D and 8270E, along with a table summarizing the QC requirements for 8270E are listed in Appendix 8270E.

14.1.8 For compliance samples, all procedures outlined in this SOP must be followed without exception. In the event that a deviation from this SOP cannot be avoided (i.e., demonstrated and uncorrectable matrix interference, non-compatible matrix, insufficient sample amount due to client or laboratory error) it is imperative that an NCM is completed that clearly documents the anomaly. This anomaly must be communicated to the client such that corrective action activity including sample recollection can be performed. Based upon the type and severity of anomaly, the results may not be appropriate for compliance reporting. Discussions between the client, the appropriate regulatory authority, and PAS - WCOL will be necessary to resolve certain anomalies. In any case, it is essential that any anomaly is documented on an NCM.

15.0 Responsibilities

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 Attachments

16.1 Appendix A – Tables

16.1.1 Table I – PAS-WCOL Primary Standards and Standard Reporting Limits

16.1.2 Table II – PAS-WCOL Appendix IX Standard and Standard Reporting Limits

16.1.3 Table III-A – Reportable Analytes for PAS-WCOL Standard Tests, Primary Standard

16.1.4 Table III-B – Reportable Analytes for PAS_WCOL Standard Tests, Appendix IX Standard

16.1.5 Table IV – Recommended Instrument Conditions

16.1.6 Table V – DFTPP Key Ions and Ion Abundance Criteria

16.1.7 Table VI – Recommended Minimum RRF Acceptance Criteria for Initial and Continuing Calibration

16.1.8 Table VI-A – Characteristic Ions for PAS-WCOL Primary Standard

16.1.9 Table VI-B – Characteristic Ions for PAS-WCOL Appendix IX Standard

16.1.10 Table VII – Method 8270D/E LCS Spiking Compounds and Concentrations

16.1.11 Table VIII – TCLP BNA LCS Compounds and Concentrations

16.1.12 Table IX – Method 8270D/E Surrogate Compounds and Concentrations

16.1.13 Table X-A – Possible Calibration Levels, Primary Standard

16.1.14 Table X-B – Possible Calibration Levels, Appendix IX Standard

16.1.15 Table X-C – Possible Calibration Levels, Appendix IX Standard – For Microwave 3546 and 3520C RVE with a LVI of 5uL

16.1.16 Table IX – Initial Demonstration Recovery and Precision Limits

16.2 Appendix PAH

16.3 Appendix NC

16.4 Appendix DoD

16.5 Appendix SIM

16.6 Appendix 625.1

16.7 Appendix 8270E

17.0 References

NOTE: Where references exclude a date or edition, the latest edition of the referenced document adopted/recognized by the laboratory's accreditation bodies applies. Refer to the Quality Assurance Management Plan [QAMP ME0012K] for details.

17.1 *Consolidated Quality System Manual (QSM) for Environmental Laboratories*. Department of Defense (DoD) / Department of Energy (DoE).

17.2 *Consolidated Quality System Manual (QSM) for Environmental Laboratories*. Department of Defense (DoD) / Department of Energy (DoE).

17.3 *General Requirements for the Competence of Testing and Calibration Laboratories.* International Standard ISO/IEC 17025.

17.4 *Laboratory Accreditation Standards*. TNI Standard. The NELAC Institute.

17.5 SW846, Test Methods for Evaluating Solid Waste, *Determinative Chromatographic Separations*, Revision 4, Update V July 2014, Method 8000D.

17.6 SW846, *Semi-volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*, Revision 5, Update V, July 2014, Test Methods for Evaluating Solid Waste, Method 8270D.

17.7 SW846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, December 1996, *Section 3520C, and 3550C*.

17.8 SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, December 1996, *Sample Preparation for Organic Volatile Compounds, Method 3580A.*

17.9 J.W. Eichelberger, L.E. Harris, and W.L. Budde, *"Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography/Mass Spectrometry"*, Analytical Chemistry, 47, 995 (1975).

17.10 *Base/Neutrals and Acids by GC/MS, US EPA Method 625.1*, December 2014, US Environmental Protection Agency.

17.11 SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III December 1996, *Test Methods for Evaluating Solid Waste, Method 3546*.

17.12 SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, December 1996, *Test Methods for Evaluating Solid Waste, Method 3540C.*

17.13 SW846, Update VI, Semi-volatile Organic Compounds by Gas Chromatography/Mass Spectrometry. Revision 6, June 2018, *Test Methods for Evaluating Solid Waste, Method 8270E*

18.0 Revision History

Revision #	Revision Date	Section Modified	Modification	Reason for Modification
17	**09/27/2021**	Table SIM-II	Update 1,4-dioxane aqueous limits to 40-90	SC DHEC audit finding #9
		Table SIM-III	Update the 1,4-dioxane aqueous limits to 40-90	SC DHEC audit finding #9

APPENDIX A – TABLES

TABLE I. PAS - WCOL Primary Standards* and Standard Reporting Limits

Analyte	CAS number	Standard Reporting Limits	
		Aqueous µg/L	Soil/Sediment µg/kg
1-Chloronaphthalene	90-13-1	4.0	67
1,2,4-Trichlorobenzene	120-82-1	4.0	67
1,2-Dichlorobenzene	95-50-1	4.0	67
1,3-Dichlorobenzene	541-73-1	4.0	67
1,4-Dichlorobenzene	106-46-7	4.0	67
Bis (2-chloro-1-methylethyl) ether; or (2,2'-oxybis(1-chloropropane))**	108-60-1	4.0	67
2,4,5-Trichlorophenol	95-95-4	4.0	67
2,4,6-Trichlorophenol	88-06-2	4.0	67
2,4-Dichlorophenol	120-83-2	4.0	67
2,4-Dimethylphenol	105-67-9	4.0	67
2,4-Dinitrophenol	51-28-5	20	330
2,4-Dinitrotoluene	121-14-2	8.0	130
2,6-Dinitrotoluene	606-20-2	8.0	130
2-Chloronaphthalene	91-58-7	4.0	67
2-Chlorophenol	95-57-8	4.0	67
2-Methylnaphthalene	91-57-6	0.8	13
2-Methylphenol	95-48-7	4.0	67
2-Nitroaniline	88-74-4	8.0	130
2-Nitrophenol	88-75-5	4.0	67
3,3'-Dichlorobenzidine	91-94-1	4.0	67
3,3'-Dimethylbenzidine	119-93-7	20	330
3-Nitroaniline	99-09-2	8.0	130
4,6-Dinitro-2-methylphenol	534-52-1	20	330
4-Bromophenyl phenyl ether	101-55-3	4.0	67
4-Chloro-3-methylphenol	59-50-7	4.0	67
4-Chloroaniline	106-47-8	8.0	130
4-Chlorophenyl phenyl ether	7005-72-3	4.0	67
3 & 4-Methylphenol	106-44-5	4.0	67
4-Nitroaniline	100-01-6	8.0	130
4-Nitrophenol	100-02-7	20	330

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Semivolatile Organic Compounds by GC/MS Analysis
METHOD: EPA Methods 625.1/SW-846 8270D/SW-846 8270E
ISSUER: Pace ENV - Local Quality - WCOL

Analyte	CAS number	Standard Reporting Limits	
		Aqueous µg/L	Soil/Sediment µg/kg
Acenaphthene	83-32-9	0.8	13
Acenaphthylene	208-96-8	0.8	13
Anthracene	120-12-7	0.8	13
Azobenzene	103-33-3	4.0	67
Benzidine	92-87-5	20	330
Benzoic Acid	65-85-0	20	330
Benzo(a)anthracene	56-55-3	0.8	13
Benzo(a)pyrene	50-32-8	0.8	13
Benzo(b)fluoranthene	205-99-2	0.8	13
Benzo(g,h,i)perylene	191-24-2	0.8	13
Benzo(k)fluoranthene	207-08-9	0.8	13
Bis(2-Chloroethoxy)methane	111-91-1	4.0	67
Bis(2-chloroethyl)ether	111-44-4	4.0	67
Bis(2-ethylhexyl)phthalate	117-81-7	8.0	130
Butyl benzyl phthalate	85-68-7	4.0	67
Caprolactam***	105-60-2	4.0	67
Carbazole	86-74-8	4.0	67
Chrysene	218-01-9	0.8	13
DEET	134-62-3	8.0	130
Dibenz(a,h)anthracene	53-70-3	0.8	13
Dibenzofuran	132-64-9	4.0	67
Diethylphthalate	84-66-2	4.0	67
Dimethyl phthalate	131-11-3	4.0	67
Di-n-butyl phthalate	84-74-2	4.0	67
Di-n-octylphthalate	117-84-0	4.0	67
Fluoranthene	206-44-0	0.8	13
Fluorene	86-73-7	0.8	13
Hexachlorobenzene	118-74-1	4.0	67
Hexachlorobutadiene	87-68-3	4.0	67
Hexachlorocyclopentadiene	77-47-4	20	330
Hexachloroethane	67-72-1	4.0	67
Indeno(1,2,3-cd)pyrene	193-39-5	0.8	13
Isophorone	78-59-1	4.0	67
Naphthalene	91-20-3	0.8	13

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Semivolatile Organic Compounds by GC/MS Analysis
METHOD: EPA Methods 625.1/SW-846 8270D/SW-846 8270E
ISSUER: Pace ENV - Local Quality - WCOL

Analyte	CAS number	Standard Reporting Limits	
		Aqueous µg/L	Soil/Sediment µg/kg
Nitrobenzene	98-95-3	4.0	67
N-Nitroso-di-n-propylamine	621-64-7	4.0	67
N-Nitrosodimethylamine	62-75-9	4.0	67
N-Nitrosodiphenylamine	86-30-6	4.0	67
Pentachlorophenol	87-86-5	20	330
Phenanthrene	85-01-8	0.8	13
Phenol	108-95-2	4.0	67
Piperonyl butoxide	51-03-6	8.0	130
Pyrene	129-00-0	4.0	67
Pyridine	110-86-1	8.0	130

*The PAS - WCOL primary standard is the standard normally used at PAS - WCOL. Additional standards, such as the Appendix IX standard may be necessary to include all target analytes required for some clients.

** Bis (2-chloro-1-methylethyl) ether or (2,2'-oxybis(1-chloropropane)) was formally known as bis(2-chloroisopropyl)ether

***Project specific compound

TABLE II. PAS - WCOL Appendix IX Standard* and Standard Reporting Limits

Semi-Volatiles	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Soil/Sediment µg/kg
1,1'-Biphenyl	92-52-4	4.0	67
1,2,4,5-Tetrachlorobenzene	95-94-3	4.0	67
1,3,5-Trinitrobenzene	99-35-4	25	830
1,3-Dinitrobenzene	99-65-0	25	830
1,4-Dinitrobenzene	100-25-4	5.0	330
1,4-Dioxane	123-41-1	5.0	330
1,4-Naphthoquinone	130-15-4	10	330
1-Chloronaphthalene	90-13-1	5.0	330
1-Methylnaphthalene	90-12-0	0.8	13
1-Naphthylamine	134-32-7	10	330
2,3,4,6-Tetrachlorophenol	58-90-2	8.0	130
2,3,5,6-Tetrachlorophenol	935-95-5	10	330
2,6-Dichlorophenol	87-65-0	4.0	130
2-Acetylaminofluorene	53-96-3	25	830
2-Naphthylamine	91-59-8	10	330
2-Picoline	109-06-8	10	330
3,3'-Dimethylbenzidine	119-93-7	25	830
3-Methylchloanthrene	56-49-5	10	330
4,4'-Methylene bis(2-chloroaniline)	101-14-4	10	330
4-Aminobiphenyl	62-67-1	10	330
4-Nitroquinoline-1-oxide	56-57-5	25	830
5-Nitro-o-toluidine	99-55-8	10	330
7,12-Dimethylbenz(a)anthracene	57-97-6	10	330
a,a-Dimethyl-phenethylamine	122-09-8	50	830
Acetophenone	98-86-2	4.0	130
Acrylamide	79-06-1	10	330
Aniline	62-53-3	8.0	130
Aramite	140-57-8	25	830
Atrazine	1912-24-9	4.0	67
Azobenzene****	103-33-3	5.0	330
Benzaldhyde	100-52-7	8.0	130
Benzidine	92-87-5	25	830
Benzoic Acid	65-85-0	50	1700
Benzyl alcohol	100-51-6	8.0	130

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Semivolatile Organic Compounds by GC/MS Analysis
METHOD: EPA Methods 625.1/SW-846 8270D/SW-846 8270E
ISSUER: Pace ENV - Local Quality - WCOL

Semi-Volatiles	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Soil/Sediment µg/kg
Chlorobenzilate	510-15-6	10	330
Diallate***	2303-16-4	10	330
Diallate-isomer1	2303-16-4	10	330
Diallate-isomer2	2303-16-4	10	330
Dibenz(a,j)acridine	224-42-0	10	330
Dibenz(a,e)pyrene	192-65-4	10	330
Dichlorofenthion	97-17-6	10	330
Dimethoate**	60-51-5	10	330
Dinoseb	88-85-7	10	330
Diphenylamine	122-39-4	10	330
Disulfoton**	298-04-4	10	330
Ethyl methanesulfonate	62-50-0	10	330
Ethyl Parathion	56-38-2	25	830
Famphur***	52-85-7	25	830
Hexachlorophene	70-30-4	100	3300
Hexachloropropene	1888-71-7	10	330
Hexadecane	544-76-3	5.0	330
Indene	95-13-6	10	330
Isodrin***	465-73-6	10	330
Isosafrole	120-58-1	10	330
Kepone	143-50-0	10	330
m-Dinitrobenzene	99-65-0	10	330
Methapyrilene	91-80-5	25	830
Methoxychlor	72-43-5	10	330
Methyl methacrylate	80-62-6	10	330
Methyl methanesulfonate	66-27-3	10	330
Methyl parathion**	298-00-0	25	830
Mirex	2385-85-5	10	330
N-Decane	124-18-5	5.0	330
N,N-Diethyl-m-toluamide (DEET)	134-62-3	10	330
N-Nitrosodiethylamine	55-18-5	10	330
N-Nitrosodimethylamine	62-75-9	4.0	67
N-Nitroso-di-n-butylamine	924-16-3	10	330
N-Nitrosomethylethylamine	10595-95-6	10	330
N-Nitrosomorpholine	59-89-2	10	330

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Semivolatile Organic Compounds by GC/MS Analysis
METHOD: EPA Methods 625.1/SW-846 8270D/SW-846 8270E
ISSUER: Pace ENV - Local Quality - WCOL

Semi-Volatiles	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Soil/Sediment µg/kg
N-Nitrosopiperidine	100-75-4	01	330
N-Nitrosopyrrolidine	930-55-2	10	330
octa-Decane	593-45-3	4.0	67
o,o-Diethyl-o-pyrazinyl ester	297-97-2	10	330
o,o,o-Triethyl-Phosphorothioate**	126-68-1	10	330
o-Toluidine	95-53-4	10	330
p-Benzoquinone	106-51-4	10	330
p-(Dimethylamino)azobenzene	60-11-7	10	330
Parathion**	56-38-2	25	830
p-Chlorobenzilate***	510-15-6	10	330
Pentachlorobenzene	608-93-5	10	330
Pentachloroethane	76-01-7	10	330
Pentachloronitrobenzene	82-68-8	25	830
Phenacetin	62-44-2	10	330
Phorate**	298-02-2	10	330
p-Phenylenediamine	106-50-3	50	830
Piperonyl butoxide (PIP)	51-03-6	10	330
Pronamide	23950-58-5	10	330
Pyridine	110-86-1	8.0	130
Quinoline	91-22-5	10	330
Safrole	94-59-7	10	330
Sulfotepp**	3689-24-5	10	330
Sym-Trinitrobenzene	99-35-4	10	330
Thionazin**	297-97-2	10	330
Tributylphosphate	126-73-8	10	330

*The Appendix IX standard contains additional analytes required for the Appendix IX list. The PAS - WCOL primary standard must also be analyzed to include the entire Appendix IX list

** May also be analyzed by method 8141A, which can achieve lower reporting limits

*** May also be analyzed by method 8081A, which can achieve lower reporting limits

****Azobenzene is formed by decomposition of 1,2-diphenylhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Semivolatile Organic Compounds by GC/MS Analysis
METHOD: EPA Methods 625.1/SW-846 8270D/SW-846 8270E
ISSUER: Pace ENV - Local Quality - WCOL

TABLE III-A. Reportable Analytes for PAS - WCOL Standard Tests, Primary Standard

Analytes	CAS Number	PAS - WCOL Standard List	TCLP	TCL	Appendix IX
1,2,4-Trichlorobenzene	120-82-1	X		X	X
1,2-Dichlorobenzene	95-50-1	X		X	X
1,3-Dichlorobenzene	541-73-1	X		X	X
1,4-Dichlorobenzene	106-46-7	X	X	X	X
Bis (2-chloro-1-methylethyl) ether or (2,2'-oxybis(1-chloropropane))*	108-60-1	X		X	X
2,4,5-Trichlorophenol	95-95-4	X	X	X	X
2,4,6-Trichlorophenol	88-06-2	X	X	X	X
2,4-Dichlorophenol	120-83-2	X		X	X
2,4-Dimethylphenol	105-67-9	X		X	X
2,4-Dinitrophenol	51-28-5	X		X	X
2,4-Dinitrotoluene	121-14-2	X	X	X	X
2,6-Dinitrotoluene	606-20-2	X		X	X
2-Chloronaphthalene	91-58-7	X		X	X
2-Chlorophenol	95-57-8	X		X	X
2-Methylnaphthalene	91-57-6	X		X	X
2-Methylphenol	95-48-7	X	X	X	X
2-Nitroaniline	88-74-4	X		X	X
2-Nitrophenol	88-75-5	X		X	X
3,3'-Dichlorobenzidine	91-94-1	X		X	X
3-Nitroaniline	99-09-2	X		X	X
4,6-Dinitro-2-methylphenol	534-52-1	X		X	X
4-Bromophenyl phenyl ether	101-55-3	X		X	X
4-Chloro-3-methylphenol	59-50-7	X		X	X
4-Chloroaniline	106-47-8	X		X	X
4-Chlorophenyl phenyl ether	7005-72-3	X		X	X
4 & 3-Methylphenol	106-44-5	X	X	X	X
4-Nitroaniline	100-01-6	X		X	X
4-Nitrophenol	100-02-7	X		X	X
Acenaphthene	83-32-9	X		X	X
Acenaphthylene	208-96-8	X		X	X
Anthracene	120-12-7	X		X	X

Analytes	CAS Number	PAS - WCOL Standard List	TCLP	TCL	Appendix IX
Benzo(a)anthracene	56-55-3	X		X	X
Benzo(a)pyrene	50-32-8	X		X	X
Benzo(b)fluoranthene	205-99-2	X		X	X
Benzo(g,h,i)perylene	191-24-2	X		X	X
Benzo(k)fluoranthene	207-08-9	X		X	X
Bis(2-Chloroethoxy)methane	111-91-1	X		X	X
Bis(2-chloroethyl)ether	111-44-4	X		X	X
Bis(2-ethylhexyl)phthalate	117-81-7	X		X	X
Butyl benzyl phthalate	85-68-7	X		X	X
Caprolactam**	105-60-2	X		X	
Carbazole	86-74-8	X		X	
Chrysene	218-01-9	X		X	X
Dibenz(a,h)anthracene	53-70-3	X		X	X
Dibenzofuran	132-64-9	X		X	X
Diethylphthalate	84-66-2	X		X	X
Dimethyl phthalate	131-11-3	X		X	X
Di-n-butyl phthalate	84-74-2	X		X	X
Di-n-octylphthalate	117-84-0	X		X	X
Fluoranthene	206-44-0	X		X	X
Fluorene	86-73-7	X		X	X
Hexachlorobenzene	118-74-1	X	X	X	X
Hexachlorobutadiene	87-68-3	X	X	X	X
Hexachlorocyclopentadiene	77-47-4	X		X	X
Hexachloroethane	67-72-1	X	X	X	X
Indeno(1,2,3-cd)pyrene	193-39-5	X		X	X
Isophorone	78-59-1	X		X	X
Naphthalene	91-20-3	X		X	X
Nitrobenzene	98-95-3	X	X	X	X
N-Nitroso-di-n-propylamine	621-64-7	X		X	X
N-Nitrosodiphenylamine	86-30-6	X		X	X
Pentachlorophenol	87-86-5	X	X	X	X
Phenanthrene	85-01-8	X		X	X
Phenol	108-95-2	X		X	X
Pyrene	129-00-0	X		X	X

* Bis (2-chloro-1-methylethyl) ether or (2,2-oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl) ether
**Project specific compound

TABLE III-B. Reportable Analytes for PAS - WCOL Standard Tests, Appendix IX Standard

Semivolatiles	CAS Number	PAS - WCOL Standard List	TCLP	TCL	Appendix IX
1,2,4,5-Tetrachlorobenzene	95-94-3				X
1,3,5-Trinitrobenzene	99-35-4				X
1,3-Dinitrobenzene	99-65-0				X
1,4-Naphthoquinone	130-15-4				X
1-Chloronaphthalene	90-13-1			X	
1-Naphthylamine	134-32-7				X
2,3,4,6-Tetrachlorophenol	58-90-2				X
2,6-Dichlorophenol	87-65-0				X
2-Acetylaminofluorene	53-96-3				X
2-Naphthylamine	91-59-8				X
2-Picoline	109-06-8				X
3,3'-Dimethylbenzidine	119-93-7			X	X
3-Methylchloanthrene	56-49-5				X
4-Aminobiphenyl	62-67-1				X
4-Nitroquinoline-1-oxide	56-57-5				X
5-Nitro-o-toluidine	99-55-8				X
7,12-Dimethylbenz(a)anthracene	57-97-6				X
a,a-Dimethyl-phenethylamine	122-09-8				X
Acetophenone	98-86-2			X	X
Aniline	62-53-3				X
Aramite	140-57-8				X
Azobenzene	103-33-3			X	
Benzidine	92-87-5			X	
Benzoic acid	65-85-0			X	
Benzyl alcohol	100-51-6			X	X
Diallate	2303-16-4				X
Dichlorofenthion	97-17-6				
Dimethoate*	60-51-5				X
Diphenylamine****	122-39-4				X
Disulfoton*	298-04-4				X
Ethyl methanesulfonate	62-50-0				X
Famphur*	52-85-7				X
Hexachlorophene***	70-30-4				X

Semivolatiles	CAS Number	PAS - WCOL Standard List	TCLP	TCL	Appendix IX
Hexachloropropene	1888-71-7				X
Isodrin**	465-73-6				X
Isosafrole	120-58-1				X
Methapyrilene	91-80-5				X
Methyl methanesulfonate	66-27-3				X
Methyl parathion*	298-00-0				X
N,N-Diethyl-m-toluamide (DEET)	134-62-3			X	
N-Nitrosodiethylamine	55-18-5				X
N-Nitrosodimethylamine	62-75-9			X	X
N-Nitroso-di-n-butylamine	924-16-3				X
N-Nitrosomethylethylamine	10595-95-6				X
N-Nitrosomorpholine	59-89-2				X
N-Nitrosopiperidine	100-75-4				X
N-Nitrosopyrrolidine	930-55-2				X
o,o,o-Triethyl-Phosphorothioate*	126-68-1				X
o-Toluidine	95-53-4				X
p-(Dimethylamino)azobenzene	60-11-7				X
Parathion*	56-38-2				X
p-Chlorobenzilate**	510-15-6				X
Pentachlorobenzene	608-93-5				X
Pentachloroethane	76-01-7				X
Pentachloronitrobenzene	82-68-8				X
Phenacetin	62-44-2				X
Phorate*	298-02-2				X
p-Phenylenediamine	106-50-3				X
Piperonyl butoxide (PIP)	51-03-6			X	
Pronamide	23950-58-5				X
Pyridine	110-86-1		X		X
Safrole	94-59-7				X
Sulfotepp*	3689-24-5				X
Thionazin*	297-97-2				X

*May also be analyzed by method 8140 or 8141, which can achieve lower reporting limits

**May also be analyzed by method 8080 or 8081, which can achieve lower reporting limits

***Hexachlorophene is a required analyte for Appendix IX. This compound is not stable, and therefore not included in the calibration standard. The characteristic ions for hexachlorophene are searched for in the chromatogram. (See Section 10.3).

****Diphenylamine is a required compound for Appendix IX. N-nitrosodiphenylamine decomposes in the injection port to form diphenylamine. Therefore, these two compounds cannot be distinguished. Diphenylamine is not included in the calibration standard.

TABLE IV. Recommended Instrument Conditions

Mass range	35-500 amu
Scan time	±1 second/scan
Initial column temperature / Hold time	45°C for 2 minutes
Column temperature program	45 – 280 °C at 30 °C/min, 10 °C/min to 325,
Final column temperature / Hold time	325 °C for 4.25 minutes (or until at least one minute after benzo(g,h,i)perylene has eluted)
Injector temperature	250 – 300 °C
Transfer line temperature	250 – 300 °C
Source temperature	According to manufacturer's specifications
Injector	Grob-type, split / splitless
Sample volume	0.5, 1, or 5 µl
Carrier gas	Helium at 30 cm/sec

TABLE V. DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	10 – 80% of Base Peak
68	< 2% of mass 69
70	< 2% of mass 69
127	10 – 80% of Base Peak
197	< 2% of mass 198
198	Base peak, or > 50% of mass 442
199	5 – 9% of mass 198
275	10 – 60% of Base Peak
365	> 1% of mass 198
441	Present, but <24% mass 442
442	Base Peak or > 50% of mass 198
443	15 – 24% of mass 442

TABLE VI: Recommended Minimum RRF Acceptance Criteria for Initial and Continuing Calibration

Analyte	Minimum RRF	Analyte	Minimum RRF
Benzaldehyde	0.01	Diethylphthalate	0.01
Phenol	0.800	Fluorene	0.900
bis-(2-Chloroethyl)ether	0.500	4-Chlorophenyl-phenylether	0.400
2-Chlorophenol	0.800	4-Nitroaniline	0.01
2-Methylphenol	0.700	4,6-Dinitro-2-methylphenol	0.01
Bis (2-chloro-1-methylethyl) ether (or 2,2'-oxybis(1-Chloropropane))*	0.01	N-Nitrosodiphenylamine	0.01
Acetophenone	0.01	1,2,4,5- Tetrachlorobenzene	0.01
4-Methylphenol	0.600	4-Bromophenyl-phenylether	0.100
N-Nitroso-di-n-propylamine	0.500	Hexachlorobenzene	0.100
Hexachloroethane	0.300	Atrazine	0.01
Nitrobenzene	0.200	Pentachlorophenol	0.050
Isophorone	0.400	Phenanthrene	0.700
2-Nitrophenol	0.100	Anthracene	0.700
2,4-Dimethylphenol	0.100	Di-n-butylphthalate	0.01
bis(2-Chloroethoxy)methane	0.300	Fluoranthene	0.600
2,4-Dichlorophenol	0.200	Pyrene	0.600
Naphthalene	0.700	Butylbenzylphthalate	0.01
4-Chloroaniline	0.01	3,3'-Dichlorobenzidine	0.01
Hexachlorobutadiene	0.01	Benzo(a)anthracene	0.800
Caprolactam	0.01	Chrysene	0.700
4-Chloro-3-methylphenol	0.100	bis(2-Ethylhexyl)phthalate	0.01
2-Methylnaphthalene	0.400	Di-n-octylphthalate	0.01
Hexachlorocyclopentadiene	0.05	Benzo(b)fluoranthene	0.500
2,4,6-Trichlorophenol	0.100	Benzo(k)fluoranthene	0.500
2,4,5-Trichlorophenol	0.100	Benzo(a)pyrene	0.500
1,1'-Biphenyl	0.01	Indeno(1,2,3-cd)pyrene	0.500
2-Chloronaphthalene	0.800	Dibenzo(ah)anthracene	0.400
2-Nitroaniline	0.01	Benzo(g,h,i)perylene	0.500
Dimethylphthalate	0.01	carbazole	0.01
2,6-Dinitrotoluene	0.100	dimethylphalate	0.01
Acenaphthylene	0.900	2,3,4,6 Tetrachlorophenol	0.01
3-Nitroaniline	0.01		
Acenaphthene	0.900		
2,4-Dinitrophenol	0.01		

Analyte	Minimum RRF	Analyte	Minimum RRF
4-Nitrophenol	0.01		
Dibenzofuran	0.800		
2,4-Dinitrotoluene	0.100		

TABLE VI-A. Characteristic Ions for PAS - WCOL Primary Standard

Analyte	Primary	Secondary	Tertiary
1,2,4-Trichlorobenzene	180	182	145
1,2-Dichlorobenzene	146	148	111
1,3-Dichlorobenzene	146	148	111
1,4-Dichlorobenzene	146	148	111
1,4-Dichlorobenzene-d4 (Internal Standard)	152	150	115
1-Methylnaphthalene	142	141	115
Bis (2-chloro-1-methylethyl) ether (or 2,2'-oxybis(1-chloropropane))*	45	77	79
2,4,5-Trichlorophenol	196	132	97
2,4,6-Tribromophenol (Surrogate Standard)	330	332	62
2,4,6-Trichlorophenol	196	198	200
2,4-Dichlorophenol	162	164	98
2,4-Dimethylphenol	122	107	121
2,4-Dinitrophenol	184	154	63
2,4-Dinitrotoluene	165	63	182
2,6-Dinitrotoluene	165	89	121
2-Chloronaphthalene	162	164	127
2-Chlorophenol	128	130	64
2-Fluorobiphenyl (Surrogate Standard)	172	171	N/A
2-Fluorophenol (Surrogate Standard)	112	64	92
2-Methylnaphthalene	142	141	115
2-Methylphenol	108	107	77
2-Nitroaniline	138	65	92
2-Nitrophenol	139	65	109
3,3'-Dichlorobenzidine	252	254	N/A
3-Nitroaniline	138	65	92
4,6-Dinitro-2-methylphenol	198	121	105
4-Bromophenylphenylether	248	250	141
4-Chloro-3-methylphenol	107	142	144
4-Chloroaniline	127	65	129
4-Chlorophenylphenylether	204	206	141
4 & 3-Methylphenol	108	107	77
4-Nitroaniline	138	65	92
4-Nitrophenol	109	65	81
Acenaphthene	153	152	154
Acenaphthene-d10 (Internal Standard)	164	162	160
Acenaphthylene	152	151	153

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Semivolatile Organic Compounds by GC/MS Analysis
METHOD: EPA Methods 625.1/SW-846 8270D/SW-846 8270E
ISSUER: Pace ENV - Local Quality - WCOL

Analyte	Primary	Secondary	Tertiary
Anthracene	178	176	179
Atrazine	200	173	215
Benzaldehyde	77	105	106
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
Bis(2-chloroethoxy)methane	93	63	95
Bis(2-Chloroethyl)ether	93	63	95
Bis(2-ethylhexyl)phthalate	149	167	279
Butylbenzylphthalate	149	91	206
Caprolactam	113	55	56
Carbazole	167	139	N/A
Chrysene	228	226	229
Chrysene-d12 (Internal Standard)	240	236	120
Dibenz(a,h)anthracene	278	279	139
Dibenzofuran	168	139	84
Diethylphthalate	149	177	150
Dimethylphthalate	163	164	194
Di-n-Butylphthalate	149	150	104
Di-n-octylphthalate	149	150	N/A
Fluoranthene	202	101	100
Fluorene	166	165	167
Hexachlorobenzene	284	142	249
Hexachlorobutadiene	225	223	227
Hexachlorocyclopentadiene	237	272	235
Hexachloroethane	117	201	199
Indeno(1,2,3-cd)pyrene	276	N/A	N/A
Isophorone	82	138	95
Naphthalene	128	129	102
Naphthalene-d8 (Internal Standard)	136	N/A	N/A
Nitrobenzene	77	123	51
Nitrobenzene-d5 (Surrogate Standard)	82	54	128
N-Decane	52	71	99
N-Nitrosodimethylamine	74	42	44
N-Nitroso-di-n-propylamine	70	42	130
N-Nitrosodiphenylamine	169	168	167

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Semivolatile Organic Compounds by GC/MS Analysis
METHOD: EPA Methods 625.1/SW-846 8270D/SW-846 8270E
ISSUER: Pace ENV - Local Quality - WCOL

Analyte	Primary	Secondary	Tertiary
Octa-Decane	57	85	127
Pentachlorophenol	266	264	268
Perylene-d12 (Internal Standard)	264	260	265
Phenanthrene	178	176	179
Phenanthrene-d10 (Internal Standard)	188	N/A	N/A
Phenol	94	66	65
Phenol-d5 (Surrogate Standard)	99	71	N/A
Pyrene	202	101	100
Terphenyl-d14 (Surrogate Standard)	244	122	N/A

*Bis (2-chloro-1-methylethyl) ether (or 2,2'-oxybis(1-chloropropane)) was formally known as bis(2-chloroisopropyl)ether

TABLE VI-B. Characteristic Ions for PAS - WCOL Appendix IX Standard

Analyte	Primary	Secondary	Tertiary
1,2,4,5-Tetrachlorobenzene	216	179	107
1,3,5-Trinitrobenzene	74	75	213
1,3-Dinitrobenzene	168	76	50
1,4-Naphthoquinone	158	102	76
1-Chloronaphthalene	162	127	164
1-Naphthylamine	143	115	89
2,3,4,6-Tetrachlorophenol	232	131	166
2,6-Dichlorophenol	162	164	98
2-Acetylaminofluorene	181	223	152
2-Naphthylamine	143	115	89
2-Picoline	93	66	78
3,3'-Dimethylbenzidine	212	N/A	N/A
3-Methylchloanthrene	268	252	126
4-Aminobiphenyl	169	168	115
4-Nitroquinoline-1-oxide	190	89	160
5-Nitro-o-toluidine	152	106	N/A
7,12-Dimethylbenz(a)anthracene	256	241	120
a,a-Dimethyl-phenethylamine	58	134	N/A
Acetophenone	105	77	120
Aniline	66	93	65
Aramite 1	185	191	319
Aramite 2	185	191	319
Azobenzene	77	182	51
Benzoic acid	105	122	77
Benzyl alcohol	79	108	51
Diallate	86	70	234
Dichlorofenthion	223	279	97
Dimethoate	87	93	125
Disulfoton	88	89	97
Ethyl methanesulfonate	79	109	97
Famphur	218	125	93
Hexachloropropene	213	141	117
Isodrin	193	263	147
Isosafrole 1	131	162	104
Isosafrole 2	131	162	104
Methapyrilene	58	97	191
Methyl methanesulfonate	80	79	65
Methyl parathion	109	125	263
N,N-Diethyl-m-toluamide (DEET)	119	190	91

Analyte	Primary	Secondary	Tertiary
N-Nitrosodiethylamine	102	44	56
N-Nitrosodimethylamine	74	42	44
N-Nitroso-di-n-butylamine	84	57	116
N-Nitrosomethylethylamine	88	42	43
N-Nitrosomorpholine	56	116	86
N-Nitrosopiperidine	42	55	114
N-Nitrosopyrrolidine	100	41	69
o,o,o-Triehtyl-phosphorothioate	198	121	97
o-Toluidine	106	107	77
p-(Dimethylamino)azobenzene	120	225	77
Parathion	97	291	109
p-Chlorobenzilate	251	139	111
Pentachlorobenzene	250	215	108
Pentachloroethane	167	117	83
Pentachloronitrobenzene	237	142	295
Phenacetin	108	137	179
Phorate	121	75	260
p-Phenylenediamine	108	80	52
Piperonyl butoxide	176	177	149
Pronamide	173	255	145
Pyridine	79	52	50
Safrole	162	104	131
Sulfotepp	322	202	97
Thionazin	107	96	248

TABLE VII. Method 8270D/E LCS Spiking Compounds and Concentrations

LCS Compounds	Concentration in Spiking Standard (µg/mL)	Concentration in Spiking Standard (µg/mL) for LVI
1-Chloronaphthalene	40	8
1,2-Dichlorobenzene	40	8
1,3-Dichlorobenzene	40	8
1,4-Dichlorobenzene	40	8
1,2,4-Trichlorobenzene	40	8
2-Chloronaphthalene	40	8
2-Chlorophenol	40	8
2-Methylphenol (o-cresol)	40	8
2-Methylnaphthalene	40	8
2-Nitroaniline	40	8
2-Nitrophenol	40	8
2,4-Dichlorophenol	40	8
2,4-Dimethylphenol	40	8
2,4-Dinitrophenol	80	16
2,4-Dinitrotoluene	40	8
2,6-Dinitrotoluene	40	8
2,4,5-Trichlorophenol	40	8
2,4,6-Trichlorophenol	40	8
3-Methylphenol (m-cresol)	40	8
3-Nitroaniline	40	8
3,3'-Dichlorobenzidine	40	8
4-Bromophenyl phenyl ether	40	8
4-Chloroaniline	40	8
4-Chlorophenyl phenyl ether	40	8
4-Methylphenol (p-cresol)	40	8
4-Nitroaniline	40	8
4-Nitrophenol	80	16
4-Chloro-3-methylphenol	40	8
4,6-Dinitro-2-methylphenol (Dinitro-o-cresol)	40	8
Acenaphthene	40	8
Acenaphthalene	40	8
Anthracene	40	8
Azobenzene	40	8
Benz(a)anthracene	40	8
Benzidine	200	40
Benzo(a)pyrene	40	8

LCS Compounds	Concentration in Spiking Standard (µg/mL)	Concentration in Spiking Standard ((µg/mL) for LVI
Benzo(b)fluoranthene	40	8
Benzo(g,h,i)perylene	40	8
Benzo(k)fluoranthene	40	8
Benzyl butyl phthalate	40	8
Bis(2-chloroethoxy)methane	40	8
Bis(2-chloroethyl)ether	40	8
Bis(2-chloroisopropyl)ether	40	8
Bis(2-ethylhexyl)phthalate	40	8
Caprolactam*	40	8
Carbazole	40	8
Chrysene	40	8
Dibenz(a,h)anthracene	40	8
Dibenzofuran	40	8
Diethylphthalate	40	8
Dimethylphthalate	40	8
Di-n-butylphthalate	40	8
Di-n-octylphthalate	40	8
Fluoranthene	40	8
Fluorene	40	8
Hexachlorobenzene	40	8
Hexachlorobutadiene	40	8
Hexachlorocyclopentadiene	200	40
Hexachloroethane	40	8
Ideno(1,2,3-cd)pyrene	40	8
Isophorone	40	8
Naphthalene	40	8
Nitrobenzene	40	8
N-Nitrosodimethylamine	40	8
N-Nitroso-di-n-propylamine	40	8
N-Nitrosodiphenylamine	40	8
Pentachlorophenol	80	16
Phenanthrene	40	8
Phenol	40	8
Pyrene	40	8
Pyridine	40	8

Recovery limits for the LCS and for matrix spikes are generated from historical data and are maintained by the QA department. LCS samples extracted by 3520C RVE and microwave 3546 are spiked at 20% of the concentration.

*Project Specific Compound

TABLE VIII. TCLP BNA LCS Compounds and Concentrations

LCS Compounds	Concentration in Spiking Standard (µg/mL)
1,4-Dichlorobenzene	40
2,4,5-Trichlorophenol	40
2,4,6-Trichlorophenol	40
2,4-Dinitrotoluene	40
2-Methylphenol	40
3-Methylphenol	40
4-Methylphenol	40
Hexachlorobenzene	40
Hexachlorobutadiene	40
Hexachloroethane	40
Nitrobenzene	40
Pentachlorophenol	40
Pyridine	40

TABLE IX. Method 8270D/E Surrogate Compounds and Concentrations

Surrogate Compounds	Concentration in Standard (µg/mL)
2,4,6-Tribromophenol	40
2-Fluorobiphenyl	40
2-Fluorophenol	40
Nitrobenzene-d5	40
Phenol-d5/-d6	40
Terphenyl-d14	40

Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

TABLE X-A. Possible Calibration Levels (not all are required), Primary Standard, µg/mL

Analyte 1 µl injection	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
1,2,4-Trichlorobenzene	1	2	5	10	15	20	25	30
1,2-Dichlorobenzene	1	2	5	10	15	20	25	30
1,3-Dichlorobenzene	1	2	5	10	15	20	25	30
1,4-Dichlorobenzene	1	2	5	10	15	20	25	30
Bis (2-chloro-1-methylethyl) ether (or 2,2'-oxybis(1-chloropropane))*	1	2	5	10	15	20	25	30
2,4,5-Trichlorophenol	1	2	5	10	15	20	25	30
2,4,6-Trichlorophenol	1	2	5	10	15	20	25	30
2,4-Dichlorophenol	1	2	5	10	15	20	25	30
2,4-Dimethylphenol	1	2	5	10	15	20	25	30
2,4-Dinitrophenol	2	4	10	20	30	40	50	60
2,4-Dinitrotoluene	1	2	5	10	15	20	25	30
2,6-Dinitrotoluene	1	2	5	10	15	20	25	30
2-Chloronaphthalene	1	2	5	10	15	20	25	30
2-Chlorophenol	1	2	5	10	15	20	25	30
2-Methylnaphthalene	1	2	5	10	15	20	25	30
2-Methylphenol	1	2	5	10	15	20	25	30
2-Nitroaniline	1	2	5	10	15	20	25	30
2-Nitrophenol	1	2	5	10	15	20	25	30
3,3'-Dichlorobenzidine	1	2	5	10	15	20	25	30
3-Nitroaniline	1	2	5	10	15	20	25	30
4,6-Dinitro-2-methylphenol	1	2	5	10	15	20	25	30
4-Bromophenyl phenyl ether	1	2	5	10	15	20	25	30
4-Chloro-3-methylphenol	1	2	5	10	15	20	25	30
4-Chloroaniline	1	2	5	10	15	20	25	30
4-Chlorophenyl phenyl ether	1	2	5	10	15	20	25	30
4-Methylphenol	1	2	5	10	15	20	25	30
4-Nitroaniline	1	2	5	10	15	20	25	30
4-Nitrophenol	2	4	10	20	30	40	50	60
Acenaphthene	1	2	5	10	15	20	25	30
Acenaphthylene	1	2	5	10	15	20	25	30
Anthracene	1	2	5	10	15	20	25	30
Benzo(a)anthracene	1	2	5	10	15	20	25	30
Benzo(a)pyrene	1	2	5	10	15	20	25	30
Benzo(b)fluoranthene	1	2	5	10	15	20	25	30
Benzo(g,h,i)perylene	1	2	5	10	15	20	25	30
Benzo(k)fluoranthene	1	2	5	10	15	20	25	30

Analyte 1 µl injection	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
Bis(2-chloroethoxy)methane	1	2	5	10	15	20	25	30
Bis(2-chloroethyl)ether	1	2	5	10	15	20	25	30
Bis(2-ethylhexyl)phthalate	1	2	5	10	15	20	25	30
Butyl benzyl phthalate	1	2	5	10	15	20	25	30
Caprolactam**	1	2	5	10	15	20	25	30
Carbazole	1	2	5	10	15	20	25	30
Chrysene	1	2	5	10	15	20	25	30
Dibenz(a,h)anthracene	1	2	5	10	15	20	25	30
Dibenzofuran	1	2	5	10	15	20	25	30
Diethylphthalate	1	2	5	10	15	20	25	30
Dimethyl phthalate	1	2	5	10	15	20	25	30
Di-n-butyl phthalate	1	2	5	10	15	20	25	30
Di-n-octylphthalate	1	2	5	10	15	20	25	30
Fluoranthene	1	2	5	10	15	20	25	30
Fluorene	1	2	5	10	15	20	25	30
Hexachlorobenzene	1	2	5	10	15	20	25	30
Hexachlorobutadiene	1	2	5	10	15	20	25	30
Hexachlorocyclopentadiene	5	10	25	50	75	100	125	150
Hexachloroethane	1	2	5	10	15	20	25	30
Indeno(1,2,3-cd)pyrene	1	2	5	10	15	20	25	30
Isophorone	1	2	5	10	15	20	25	30
Naphthalene	1	2	5	10	15	20	25	30
Nitrobenzene	1	2	5	10	15	20	25	30
N-Nitroso-di-n-propylamine	1	2	5	10	15	20	25	30
N-Nitrosodiphenylamine	1	2	5	10	15	20	25	30
Pentachlorophenol	2	4	10	20	30	40	50	60
Phenanthrene	1	2	5	10	15	20	25	30
Phenol	1	2	5	10	15	20	25	30
Pyrene	1	2	5	10	15	20	25	30

*2,2'-oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether.
**Project Specific Compound

TABLE X-B. Possible Calibration Levels (not all are required), Appendix IX Standard, µg/mL

Semivolatiles 1 µl injection	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
1,2,4,5-Tetrachlorobenzene	1	2	5	10	15	20	25	30
1,3,5-Trinitrobenzene	1	2	5	10	15	20	25	30
1,3-Dinitrobenzene	1	2	5	10	15	20	25	30
1,4-Naphthoquinone	1	2	5	10	15	20	25	30
1-Chloronaphthalene	1	2	5	10	15	20	25	30
1-Naphthylamine	1	2	5	10	15	20	25	30
2,3,4,6-Tetrachlorophenol	1	2	5	10	15	20	25	30
2,6-Dichlorophenol	1	2	5	10	15	20	25	30
2-Acetylaminofluorene	1	2	5	10	15	20	25	30
2-Naphthylamine	1	2	5	10	15	20	25	30
2-Picoline	1	2	5	10	15	20	25	30
3,3'-Dimethylbenzidine	5	10	25	50	75	100	125	150
3-Methylcholanthrene	1	2	5	10	15	20	25	30
4-Aminobiphenyl	1	2	5	10	15	20	25	30
4-Nitroquinoline-1-oxide	1	2	5	10	15	20	25	30
5-Nitro-o-toluidine	1	2	5	10	15	20	25	30
7,12-Dimethylbenz(a)anthracene	1	2	5	10	15	20	25	30
a,a-Dimethyl-phenethylamine	1	2	5	10	15	20	25	30
Acetophenone	1	2	5	10	15	20	25	30
Aniline	1	2	5	10	15	20	25	30
Aramite	1	2	5	10	15	20	25	30
Azobenzene*	1	2	5	10	15	20	25	30
Benzidine	5	10	25	50	75	100	125	150
Benzoic acid	1	2	5	10	15	20	25	30
Benzyl alcohol	1	2	5	10	15	20	25	30
Diallate	1	2	5	10	15	20	25	30
Dichlorofenthion	1	2	5	10	15	20	25	30
Dimethoate	1	2	5	10	15	20	25	30
Disulfoton	1	2	5	10	15	20	25	30
Ethyl methanesulfonate	1	2	5	10	15	20	25	30
Famphur	1	2	5	10	15	20	25	30
Hexachloropropene	1	2	5	10	15	20	25	30
Isodrin	1	2	5	10	15	20	25	30
Isosafrole 1+2	1	2	5	10	15	20	25	30
Methapyrilene	1	2	5	10	15	20	25	30
Methyl methanesulfonate	1	2	5	10	15	20	25	30
Methyl parathion	1	2	5	10	15	20	25	30

Semivolatiles 1 µl injection	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
N,N-Diethyl-m-toluamide (DEET)	1	2	5	10	15	20	25	30
N-Nitrosodiethylamine	1	2	5	10	15	20	25	30
N-Nitrosodimethylamine	1	2	5	10	15	20	25	30
N-Nitroso-di-n-butylamine	1	2	5	10	15	20	25	30
N-Nitrosomethylethylamine	1	2	5	10	15	20	25	30
N-Nitrosomorpholine	1	2	5	10	15	20	25	30
N-Nitrosopiperidine	1	2	5	10	15	20	25	30
N-Nitrosopyrrolidine	1	2	5	10	15	20	25	30
o,o,o-Triethyl-phosphorothioate	1	2	5	10	15	20	25	30
o-Toluidine	1	2	5	10	15	20	25	30
p-(Dimethylamino)azobenzene	1	2	5	10	15	20	25	30
Parathion	1	2	5	10	15	20	25	30
p-Chlorobenzilate	1	2	5	10	15	20	25	30
Pentachlorobenzene	1	2	5	10	15	20	25	30
Pentachloroethane	1	2	5	10	15	20	25	30
Pentacloronitrobenzene	1	2	5	10	15	20	25	30
Phenacetin	1	2	5	10	15	20	25	30
Phorate	1	2	5	10	15	20	25	30
p-Phenylenediamine	10	20	50	100	200	250	300	400
Piperonyl butoxide (PIP)	1	2	5	10	15	20	25	30
Pronamide	1	2	5	10	15	20	25	30
Pyridine	1	2	5	10	15	20	25	30
Safrole	1	2	5	10	15	20	25	30
Sulfotepp	1	2	5	10	15	20	25	30
Thionazin	1	2	5	10	15	20	25	30

*Azobenzene is formed by decomposition of 1,2-diphenylhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Samples extracted by microwave 3546, and 3520C RVE are analyzed with a large volume injection of 5 µL (for standards and samples).

TABLE X-C. Possible Calibration Levels (not all are required), Appendix IX Standard, µg/mL – For Microwave 3546 and 3520C RVE with a LVI of 5 µL**

Semivolatiles 5 µl injection	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
1,2,4,5-Tetrachlorobenzene	0.2	0.4	1	2	3	4	5	6
1,3,5-Trinitrobenzene	0.2	0.4	1	2	3	4	5	6
1,3-Dinitrobenzene	0.2	0.4	1	2	3	4	5	6
1,4-Naphthoquinone	0.2	0.4	1	2	3	4	5	6
1-Chloronaphthalene	0.2	0.4	1	2	3	4	5	6
1-Naphthylamine	0.2	0.4	1	2	3	4	5	6
2,3,4,6-Tetrachlorophenol	0.2	0.4	1	2	3	4	5	6
2,6-Dichlorophenol	0.2	0.4	1	2	3	4	5	6
2-Acetylaminofluorene	0.2	0.4	1	2	3	4	5	6
2-Naphthylamine	0.2	0.4	1	2	3	4	5	6
2-Picoline	0.2	0.4	1	2	3	4	5	6
3,3'-Dimethylbenzidine	1	2	5	10	15	20	25	30
3-Methylcholanthrene	0.2	0.4	1	2	3	4	5	6
4-Aminobiphenyl	0.2	0.4	1	2	3	4	5	6
4-Nitroquinoline-1-oxide	0.2	0.4	1	2	3	4	5	6
5-Nitro-o-toluidine	0.2	0.4	1	2	3	4	5	6
7,12-Dimethylbenz(a)anthracene	0.2	0.4	1	2	3	4	5	6
a,a-Dimethyl-phenethylamine	0.2	0.4	1	2	3	4	5	6
Acetophenone	0.2	0.4	1	2	3	4	5	6
Aniline	.4	.8	2	4	6	8	10	12
Aramite	0.2	0.4	1	2	3	4	5	6
Azobenzene*	0.2	0.4	1	2	3	4	5	6
Benzidine	1	2	5	10	15	20	25	30
Benzoic acid	0.4	0.8	2	4	6	8	10	12
Benzyl alcohol	.4	.8	2	4	6	8	10	12
Diallate	0.2	0.4	1	2	3	4	5	6
Dichlorofenthion	0.2	0.4	1	2	3	4	5	6
Dimethoate	0.2	0.4	1	2	3	4	5	6
Disulfoton	0.2	0.4	1	2	3	4	5	6
Ethyl methanesulfonate	0.2	0.4	1	2	3	4	5	6
Famphur	0.2	0.4	1	2	3	4	5	6
Hexachloropropene	0.2	0.4	1	2	3	4	5	6
Isodrin	0.2	0.4	1	2	3	4	5	6
Isosafrole 1+2	0.2	0.4	1	2	3	4	5	6
Methapyrilene	0.2	0.4	1	2	3	4	5	6
Methyl methanesulfonate	0.2	0.4	1	2	3	4	5	6
Methyl parathion	0.2	0.4	1	2	3	4	5	6

Semivolatiles 5 µl injection	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
N,N-Diethyl-m-toluamide (DEET)	0.2	0.4	1	2	3	4	5	6
N-Nitrosodiethylamine	0.2	0.4	1	2	3	4	5	6
N-Nitrosodimethylamine	0.2	0.4	1	2	3	4	5	6
N-Nitroso-di-n-butylamine	0.2	0.4	1	2	3	4	5	6
N-Nitrosomethylethylamine	0.2	0.4	1	2	3	4	5	6
N-Nitrosomorpholine	0.2	0.4	1	2	3	4	5	6
N-Nitrosopiperidine	0.2	0.4	1	2	3	4	5	6
N-Nitrosopyrrolidine	0.2	0.4	1	2	3	4	5	6
o,o,o-Triethyl-phosphorothioate	0.2	0.4	1	2	3	4	5	6
o-Toluidine	0.2	0.4	1	2	3	4	5	6
p-(Dimethylamino)azobenzene	0.2	0.4	1	2	3	4	5	6
Parathion	0.2	0.4	1	2	3	4	5	6
p-Chlorobenzilate	0.2	.4	1	2	3	4	5	6
Pentachlorobenzene	0.2	0.4	1	2	3	4	5	6
Pentachloroethane	0.2	0.4	1	2	3	4	5	6
Pentacloronitrobenzene	0.2	4	1	2	3	4	5	6
Phenacetin	0.2	0.4	1	2	3	4	5	6
Phorate	0.2	0.4	1	2	3	4	5	6
p-Phenylenediamine	2	4	10	20	30	40	50	60
Piperonyl butoxide (PIP)	0.2	0.4	1	2	3	4	5	6
Pronamide	0.2	0.4	1	2	3	4	5	6
Pyridine	0.2	0.4	1	2	3	4	5	6
Safrole	0.2	0.4	1	2	3	4	5	6
Sulfotepp	0.2	0.4	1	2	3	4	5	6
Thionazin	0.2	0.4	1	2	3	4	5	6

*Azobenzene is formed by decomposition of 1,2-diphenylhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

**LVI of 5 µL applies to standards and samples.

TABLE XI. Initial Demonstration Recovery and Precision Limits

Compound	Spiking Concentration (µg/L)	Limit for Relative Standard Deviation	Limit for Average % Recovery (Aqueous)	Limit for Average % Recovery (Soil)
1,1'- Biphenyl	40	40	30-130	49-110
1,2 Diphenylhydrazine	40	40	30-130	47-119
1,2,4-Trichlorobenzene	40	40	20-90	46-99
1,2-Dichlorobenzene	40	40	20-110	39-94
1,3-Dichlorobenzene	40	40	17-112	37-92
1,4-Dichlorobenzene	40	40	18-113	39-92
1-Methylnapthalene	40	40	42-126	30-130
2,3,4,6-Tetrachlorophenol	40	40	30-130	NA
2,4,5 Trichlorophenol	40	40	30-123	46-122
2,4,6-Trichlorophenol	40	40	30-125	38-115
2,4-Dichlorophenol	40	40	30-121	41-113
2,4-Dimethylphenol	40	40	20-125	33-123
2,4-Dinitrophenol	80	40	11-126	45-127
2,4-Dinitrotoluene	40	40	51-128	48-124
2,6-Dinitrotoluene	40	40	48-123	47-125
2-Chloronaphthalene	40	40	46-100	31-127
2-Chlorophenol	40	40	30-123	37-106
2-Methyl-4,6-dinitrophenol	40	40	56-128	40-130
2-Methylnapthalene	40	40	40-132	40-106
2-Methylphenol	40	40	37-115	32-107
2-Nitroaniline	40	40	49-128	45-123
2-Nitrophenol	40	40	36-123	35-108
3 & 4 Methylphenol	40	40	44-112	39-108
3,3'Dichlorobenzidine	40	40	10-126	46-113
3-Nitroaniline	40	40	31-128	24-127
4-Bromophenyl phenyl ether	40	40	30-124	46-118

Compound	Spiking Concentration (µg/L)	Limit for Relative Standard Deviation	Limit for Average % Recovery (Aqueous)	Limit for Average % Recovery (Soil)
4-Chloraniline	40	40	10-128	10-125
4-Chloro-3-methylphenol	40	40	58-125	49-118
4-Chlorophenyl phenyl ether	40	40	55-121	47-116
4-Nitroaniline	40	40	30-135	48-127
4-Nitrophenol	80	40	31-145	18-154
Acenaphthene	40	40	30-122	46-114
Acenaphthylene	40	40	30-130	44-122
Acetophenone	40	40	30-130	48-111
Anthracene	40	40	55-122	50-119
Atrazine	40	40	25-121	48-116
Benz(a)anthracene	40	40	40-125	47-121
Benzaldehyde	40	40	20-115	40-117
Benzidine	200	40	10-115	10-115
Benzo(a)pyrene	40	40	40-128	55-134
Benzo(b)fluoranthene	40	40	32-145	28-139
Benzo(g,h,i)perylene	40	40	42-128	36-125
Benzo(k)fluoranthene	40	40	50-135	47-130
Benzyl Alcohol	40	40	30-130	13-121
Benzylbutyl phthalate	40	40	54-135	46-128
Bis(2-chloroethoxy)methane	40	40	40-119	39-108
Bis(2-chloroethyl)ether	40	40	35-114	32-105
Bis(2-chloroisopropyl)ether	40	40	34-110	31-102
Bis(2-ethylhexyl)phthalate	40	40	50-133	45-128
Caprolactam	40	40	30-130	43-121
Carbazole	40	40	30-130	47-128
Chrysene	40	40	50-130	45-126
Dibenzo(a,h)anthracene	40	40	30-130	45-122
Dibenzofuran	40	40	30-118	45-112
Diethyl phthalate	40	40	40-125	49-123

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Semivolatile Organic Compounds by GC/MS Analysis
METHOD: EPA Methods 625.1/SW-846 8270D/SW-846 8270E
ISSUER: Pace ENV - Local Quality - WCOL

Compound	Spiking Concentration (µg/L)	Limit for Relative Standard Deviation	Limit for Average % Recovery (Aqueous)	Limit for Average % Recovery (Soil)
Dimethyl phthalate	40	40	40-127	48-120
Di-n-butyl phthalate	40	40	40-127	51-129
Di-n-octylphthalate	40	40	55-143	49-142
Fluoranthene	40	40	40-128	50-123
Fluorene	40	40	30-124	48-117
Hexachlorobenzene	40	40	30-125	44-122
Hexachlorobutadiene	40	40	24-110	33-103
Hexachlorocyclopentadiene	200	40	22-122	18-121
Hexachloroethane	40	40	28-116	30-96
Indeno(1,2,3-cd)pyrene	40	40	50-125	45-123
Isophorone	40	40	30-130	41-113
Naphthalene	40	40	40-122	36-110
Nitrobenzene	40	40	39-123	33-114
N-Nitrosodimethylamine	40	40	30-130	24-107
N-Nitroso-di-n-propylamine	40	40	39-119	32-115
N-Nitrosodiphenylamine	40	40	30-123	53-150
Pentachlorophenol	80	40	34-137	27-138
Phenanthrene	40	40	40-123	49-117
Phenol	40	40	30-130	36-108
Pyrene	40	40	40-126	47-119

APPENDIX PAH

MODIFICATIONS REQUIRED FOR ANALYSIS OF POLYNUCLEAR AROMATIC HYDROCARBONS (PAH) ONLY SAMPLES BY 8270D/E

REQUIREMENTS FOR PAH ONLY ANALYSIS

1. All requirements for DFTPP stated in 9.1.2.2 must be met.

2. Calibration standards should include all the PAH compounds (Table PAH-1) plus the base neutral surrogates and internal standard compounds.

3. Initial calibration: All compounds % RSD ± 20%, or a linear curve with a correlation coefficient ≥ 0.995 may be used. The Target data system measures the square of the correlation coefficient, which must be ≥ 0.990.

4. Continuing calibration verification
   The percent difference or drift of all analytes must be within the limits set in Table XI.

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Semivolatile Organic Compounds by GC/MS Analysis
METHOD: EPA Methods 625.1/SW-846 8270D/SW-846 8270E
ISSUER: Pace ENV - Local Quality - WCOL

Table PAH-I. PAS-WCOL Standard Reporting Limits, PAH Compounds Only

PAH Analytes	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
Acenaphthene	83-32-9	0.8	13
Acenaphthylene	208-96-8	0.8	13
Anthracene	120-12-7	0.8	13
Benzo(a)anthracene	56-55-3	0.8	13
Benzo(a)pyrene	50-32-8	0.8	13
Benzo(b)fluoranthene	205-99-2	0.8	13
Benzo(g,h,i)perylene	191-24-2	0.8	13
Benzo(k)fluoranthene	207-08-9	0.8	13
Chrysene	218-01-9	0.8	13
Dibenz(a,h)anthracene	53-70-3	0.8	13
Fluoranthene	206-44-0	0.8	13
Fluorene	86-73-7	0.8	13
Indeno(1,2,3-cd)pyrene	193-39-5	0.8	13
Naphthalene	91-20-3	0.8	13
Phenanthrene	85-01-8	0.8	13
Pyrene	129-00-0	0.8	13

APPENDIX NC - North Carolina Requirements

Sections found in this appendix replace and/or supplement the existing sections of the SOP. These requirements must be met when analyzing samples for North Carolina.

QUALITY CONTROL

- Method Blank Acceptance Criteria – replaces Section 11.2.1.1 and 11.2.1.2 sub-sections.

- ***Method Blank (MB)*** – One method blank must be processed with each preparation and/or analytical batch. The method blank consists of a similar matrix to the batch of associated samples in which no target analytes or interferences are present at concentrations that impact the analytical results. The method blank is to contain all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data.

  - For a method blank to be acceptable for use with the accompanying samples, the concentration of analytes detected in the blank must be ≤ one-half (1/2) the practical quantitation limit (LOQ) or project specific reporting limits.

    - The acceptance criterion for common laboratory contaminants is that no analytes should exceed the LOQ.

  - If the MB does not meet the criteria above, the source of the contamination shall be investigated and measures taken to minimize or eliminate the problem. The affected samples shall be reprocessed or data shall be appropriately qualified. All steps taken to return the system to control must be fully documented.

  - If reanalysis is not possible due to limited sample volume or holding time, the samples associated with the contaminated blank must be evaluated as to the best corrective action for the samples (e.g., reprocessing or data qualifying codes). In all cases the corrective action(s) must result in the completion of an NCM.

- ***LOQ Check*** – The laboratory must analyze or back-calculate a standard at or below the lowest reported concentration (LOQ) each day North Carolina compliance samples are analyzed. The analytes of interest must recover within 30-150%. If any analytes are not detected, re-analyze LOQ check and affected samples. If reanalysis fails, reprep and reanalyze.

APPENDIX DoD - DoD/DOE Method Specific Quality Control Requirements

Sections found in this appendix supersede and/or supplement the existing sections of this SOP. In addition to the general method performance criteria, these requirements must be met when analyzing samples for the Department of Defense (DoD) and the Department of Energy (DOE) as stipulated in the DoD/DOE Quality System Manual.

Table B-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of BFB or DFTPP from method.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Performance Check (Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation ≤ 20% for DDT. Benzidine and pentachlorophenol shall be present at their normal responses and shall not exceed a tailing factor of 2.	Correct problem, then repeat performance checks.	Flagging is not appropriate.	No samples shall be analyzed until performance check is within criteria. The DDT breakdown and Benzidine/ pentachlorophenol tailing factors are considered overall system checks to evaluate injector port inertness and column performance and are required regardless of the reported analyte list.
Initial calibration (ICAL) for all analytes (including surrogates)	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	Each analyte must meet one of the three options below: Option 1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; *(continued next page)*	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. No samples shall be analyzed until ICAL has passed.

Table B-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial calibration (ICAL) for all analytes (including surrogates) *(Continued)*		Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.			If the specific version of a method requires additional evaluation (e.g., RFs or low calibration standard analysis and recovery criteria) these additional requirements must also be met.
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Calculated for each analyte and surrogate.
Evaluation of Relative Retention Times (RRT)	With each sample.	RRT of each reported analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	NA.	After maintenance is performed which may affect retention times, RRTs may be updated based on the daily CCV. RRTs shall be compared with the most recently updated RRTs.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 20% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

Table B-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within ± 20% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable calibration verification.	Results may not be reported without valid CCVs. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If the specific version of a method requires additional evaluation (e.g., average RFs) these additional requirements must also be met.
Internal Standards (IS)	Every field sample, standard, and QC sample.	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within – 50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV can be used.	Inspect mass spectrometer and GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the Case Narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	NA.

Table B-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid Method Blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all surrogates and all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	Must contain all surrogates and all analytes to be reported. For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	MSD: Must contain all surrogates and all analytes to be reported. The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.

Table B-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project if available; otherwise use DoD/DOE QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch if sufficient sample material is available. If obvious chromatographic interference is present, reanalysis may not be necessary, but the client must be notified prior to reporting data and the failures must be discussed in the Case Narrative.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the Case Narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.

TABLE DoD-I. Semi-volatile Organic Target Compounds and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Soil/Sediment µg/kg
1,2,4-Trichlorobenzene	120-82-1	5.0	330
1,2-Dichlorobenzene	95-50-1	5.0	330
1,2-Diphenylhydrazine	122-66-7	5.0	330
1,3-Dichlorobenzene	541-73-1	5.0	330
1,4-Dichlorobenzene	106-46-7	5.0	330
Bis (2-chloro-1-methylethyl) ether (or 2,2'-oxybis(1-chloropropane))**	108-60-1	5.0	330
2,4,5-Trichlorophenol	95-95-4	5.0	330
2,4,6-Trichlorophenol	88-06-2	5.0	330
2,4-Dichlorophenol	120-83-2	5.0	330
2,6-Dichorophenol	87-65-0	5.0	330
2,4-Dimethylphenol	105-67-9	5.0	330
2,4-Dinitrophenol	51-28-5	25	830
2,4-Dinitrotoluene	121-14-2	10.0	330
2,6-Dinitrotoluene	606-20-2	10.0	330
2-Chloronaphthalene	91-58-7	5.0	330
2-Chlorophenol	95-57-8	5.0	330
2-Methylnaphthalene	91-57-6	5.0	330
2-Methylphenol	95-48-7	5.0	330
2-Nitroaniline	88-74-4	10.0	330
2-Nitrophenol	88-75-5	10.0	330
3,3'-Dichlorobenzidine	91-94-1	10.0	330
3-Nitroaniline	99-09-2	10.0	330
4,6-Dinitro-2-methylphenol	534-52-1	25	830
4-Bromophenyl phenyl ether	101-55-3	5.0	330
4-Chloro-3-methylphenol	59-50-7	5.0	330
4-Chlorophenyl phenyl ether	7005-72-3	5.0	330
3 & 4-Methylphenol	106-44-5	10.0	330
4-Nitroaniline	100-01-6	10.0	330
4-Nitrophenol	100-02-7	25	830
Acenaphthene	83-32-9	5.0	330
Acenaphthylene	208-96-8	5.0	330
Anthracene	120-12-7	5.0	330
Benzidine	92-87-5	25	830
Benzoic Acid	65-85-0	250	8,300
Benzo(a)anthracene	56-55-3	5.0	330

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Soil/Sediment µg/kg
Benzo(a)pyrene	50-32-8	5.0	330
Benzo(b)fluoranthene	205-99-2	5.0	330
Benzo(g,h,i)perylene	191-24-2	5.0	330
Benzo(k)fluoranthene	207-08-9	5.0	330
Benzyl Alcohol	100-51-6	10	330
Bis(2-Chloroethoxy)methane	111-91-1	5.0	330
Bis(2-chloroethyl)ether	111-44-4	5.0	330
Bis(2-ethylhexyl)phthalate	117-81-7	5.0	330
Butyl benzyl phthalate	85-68-7	5.0	330
Carbazole	86-74-8	5.0	330
Chrysene	218-01-9	5.0	330
Dibenz(a,h)anthracene	53-70-3	5.0	330
Dibenzofuran	132-64-9	5.0	330
Diethylphthalate	84-66-2	5.0	330
Dimethyl phthalate	131-11-3	5.0	330
Di-n-butyl phthalate	84-74-2	5.0	330
Di-n-octylphthalate	117-84-0	5.0	330
Fluoranthene	206-44-0	5.0	330
Fluorene	86-73-7	5.0	330
Hexachlorobenzene	118-74-1	5.0	330
Hexachlorobutadiene	87-68-3	5.0	330
Hexachloroethane	67-72-1	5.0	330
Indeno(1,2,3-cd)pyrene	193-39-5	5.0	330
Isophorone	78-59-1	5.0	330
Naphthalene	91-20-3	5.0	330
Nitrobenzene	98-95-3	5.0	330
N-Nitrosodimethylamine	62-75-9	5.0	330
N-Nitroso-di-n-propylamine	621-64-7	5.0	330
N-Nitrosodiphenylamine	86-30-6	5.0	330
N-Nitrosodipyrrolidine	930-55-2	5.0	330
Pentachlorophenol	87-86-5	25	830
Phenanthrene	85-01-8	5.0	330
Phenol	108-95-2	5.0	330
Pyrene	129-00-0	5.0	330

* The PAS - WCOL primary standard is the standard normally used at PAS - WCOL. Additional standards, such as the Appendix IX standard may be necessary to include all target analytes required for some clients.

** Bis (2-chloro-1methylethyl)ether or (2,2'-oxybis(1-chloropropane)) was formerly known as bis(2-chloroisopropyl)ether.

TABLE DoD-II. Semi-volatile Organic Surrogate Compounds and Control Limits

Surrogate Compounds	Concentration in Standard (µg/mL)	Aqueous Control Limits		Solid Control Limits	
		Lower	Upper	Lower	Upper
2,4,6-Tribromophenol	50	43	140	39	132
2-Fluorobiphenyl	50	44	119	44	115
2-Fluorophenol	50	19	119	35	115
Nitrobenzene-d5	50	44	120	37	122
Phenol-d5/-d6	50	N/A	N/A	33	122
Terphenyl-d14	50	50	134	54	127

TABLE DoD-III. LCS/MS Control Limits for Semi-volatile Organic Target Compounds

Analyte	Aqueous Control Limits		Solid Control Limits	
	Lower	Upper	Lower	Upper
Polynuclear Aromatics				
2-Methylnaphthalene	40	121	38	122
Acenaphthene	47	122	40	123
Acenaphthylene	41	130	32	132
Anthracene	57	123	47	123
Benz[a]anthracene	58	125	49	126
Benzo[a]pyrene	54	128	45	129
Benzo[b]fluoranthene	53	131	45	132
Benzo[k]fluoranthene	57	129	47	132
Benzo[g,h,i]perylene	50	134	43	134
Chrysene	59	123	50	124
Dibenz[a,h]anthracene	51	134	45	134
Fluoranthene	57	128	50	127
Fluorene	52	124	43	125
Indeno[1,2,3-cd]pyrene	52	134	45	133
Naphthalene	40	121	35	123
Phenanthrene	59	120	50	121
Pyrene	57	126	47	127
Phenolic/Acidic				
2,4,5-Trichlorophenol	53	123	41	124
2,4,6-Trichlorophenol	50	125	39	126
2,4-Dichlorophenol	47	121	40	122
2,4-Dimethylphenol	31	124	30	127
2,4-Dinitrophenol	23	143	10	160
2-Chlorophenol	38	117	34	121
2-Methylphenol	30	117	32	122

Analyte	Aqueous Control Limits	Solid Control Limits

	Lower	Upper	Lower	Upper
Phenolic/Acidic continued				
2-Nitrophenol	47	123	36	123
3 & 4-Methylphenol	29	110	34	119
4,6-Dinitro-2-methylphenol	44	137	29	132
4-Chloro-3-methylphenol	52	119	45	122
Pentachlorophenol	35	138	25	133
Basic				
3-3'-Dichlorobenzidine	27	129	22	121
4-Chloroaniline	33	117	17	106
Phthalate Esters				
Bis(2-ehtylhexyl)phthalate	55	135	51	133
Butyl benzyl phthalate	53	134	48	132
Di-n-butyl phthalate	59	127	51	128
Di-n-octyl phthalate	51	140	45	140
Diethyl phthalate	56	125	50	124
Dimethyl phthalate	45	127	48	124
Nitrosoamines				
N-Nitrosodi-n-propylamine	49	119	36	120
N-Nitrosodimethylamine	NA	NA	23	120
N-Nitrosodiphenylamine	51	123	38	127
Chlorinated Aliphatics				
Bis(2-chloroethoxy)methane	48	120	36	121
Bis(2-chloroethyl)ether	43	118	31	120
Bis(2-chloroisopropyl)ether	37	130	33	131
Hexachlorobutadiene	22	124	32	123
Hexachloroethane	21	115	28	117
Halogenated Aromatics				
1,2,4-Trichlorobenzene	29	116	34	118
1,2-Dichlorobenzene	32	111	33	117
1,3-Dichlorobenzene	28	110	30	115
1,4-Dichlorobenzene	29	112	31	115
2-Chloronaphthtalene	40	116	41	114
4-Bromophenyl phenyl ether	55	124	46	124
4-Chlorophenyl phenyl ether	53	121	45	121
Hexachlorobenzene	53	125	45	122

Analyte	Aqueous Control Limits		Solid Control Limits	
	Lower	Upper	Lower	Upper
Nitroaromatics				
2,4-Dinitrotoluene	57	128	48	126
2,6-Dinitrotoluene	57	124	46	124
2-Nitroaniline	55	127	44	127
3-Nitroaniline	41	128	33	119
Nitrobenzene	45	121	34	122
Neutral Aromatics				
Carbazole	60	122	50	123
Dibenzofuran	53	118	44	120
Others				
1,2-Diphenylhydrazine	49	122	41	125
Benzyl alcohol	31	112	29	122
Isophorone	42	124	30	122

NOTE: Marginal exceedances are not allowed for those analytes determined by a project to be target analytes without project specific approval.

TABLE DoD-IV. LCS/MS Control Limits for SIM Semi-volatile Organic Target Compounds

Analyte	Aqueous Control Limits		Solid Control Limits	
	Lower	Upper	Lower	Upper
Polynuclear Aromatics				
2-Methylnaphthalene	39	114	39	114
Acenaphthene	48	114	44	111
Acenaphthylene	35	121	39	116
Anthracene	53	119	50	114
Benz[a]anthracene	59	120	54	122
Benzo[a]pyrene	53	120	50	125
Benzo[b]fluoranthene	53	126	53	128
Benzo[k]fluoranthene	54	125	56	123
Benzo[g,h,i]perylene	44	128	49	127
Chrysene	57	120	57	118
Dibenzo[a,h]anthracene	44	131	50	129
Fluoranthene	58	120	55	119
Fluorene	50	118	47	114
Indeno[1,2,3-cd]pyrene	48	130	49	130
Naphthalene	43	114	38	111
Phenanthrene	53	115	49	113
Pyrene	58	132	55	117

NOTE: Marginal exceedances are not allowed for those analytes determined by a project to be target analytes without project specific approval.

TABLE DoD-V. Characteristic Ions for Semi-volatile Organic Target Compounds

Analyte	Primary	Secondary	Tertiary
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
1,4-Dichlorobenzene	146	148	111
1,4-Dichlorobenzene-d14 (Internal Standard)	152	150	115
2,2'-oxybis(1-chloropropane)**	445	77	79
2,4,5-Trichlorophenol	196	132	97
2,4,6-Tribromophenol (Surrogate Standard)	330	332	62
2,4,6-Trichlorophenol	196	198	200
2,4-Dichlorophenol	162	164	98
2,6-Dichorophenol	162	164	98
2,4-Dimethylphenol	122	107	121
2,4-Dinitrophenol	184	154	63
2,4-Dinitrotoluene	165	63	182
2,6-Dinitrotoluene	165	89	121
2-Chloronaphthalene	162	164	127
2-Chlorophenol	128	130	64
2-Fluorobiphenyl (Surrogate Standard)	172	171	N/A
2-Fluorophenol (Surrogate Standard)	112	64	92
2-Methylnaphthalene	142	141	115
2-Methylphenol	108	107	77
2-Nitroaniline	138	65	92
2-Nitrophenol	139	65	109
3,3'-Dichlorobenzidine	252	254	
3-Nitroaniline	138	65	92
4,6-Dinitro-2-methylphenol	198	121	105
4-Bromophenyl phenyl ether	248	250	141
4-Chloro-3-methylphenol	107	142	144
4-Chlorophenyl phenyl ether	204	206	141
3 & 4-Methylphenol	108	107	77
4-Nitroaniline	138	65	92
4-Nitrophenol	109	65	81
Acenaphthene	153	152	154
Acenaphthene-d10 (Internal Standard)	164	162	160
Acenaphthylene	152	151	153
Anthracene	178	176	179
Benzidine	184	92	185

Analyte	Primary	Secondary	Tertiary
Benzoic Acid	122	105	77
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
Benzyl Alcohol	108	79	77
Bis(2-Chloroethoxy)methane	93	63	95
Bis(2-chloroethyl)ether	93	63	95
Bis(2-ethylhexyl)phthalate	149	167	279
Butyl benzyl phthalate	149	91	206
Carbazole	167	139	N/A
Chrysene	228	226	229
Chrysene-d12 (Internal Standard)	240	236	120
Dibenz(a,h)anthracene	278	279	139
Dibenzofuran	168	139	84
Diethylphthalate	149	177	150
Dimethyl phthalate	163	164	194
Di-n-butyl phthalate	149	150	104
Di-n-octylphthalate	149	150	N/A
Fluoranthene	202	101	100
Fluorene	166	165	167
Hexachlorobenzene	284	142	249
Hexachlorobutadiene	225	223	227
Hexachloroethane	117	201	199
Indeno(1,2,3-cd)pyrene	276	N/A	N/A
Isophorone	82	138	95
Naphthalene	128	129	102
Naphthalene-d8 (Internal Standard)	136	N/A	N/A
Nitrobenzene	77	123	51
Nitrobenzene-d5 (Surrogate Standard)	82	54	128
N-Nitrosodimethylamine	74	42	44
N-Nitroso-di-n-propylamine	70	42	130
N-Nitrosodiphenylamine	169	168	167
N-Nitrosodipyrrolidine	100	41	42
Pentachlorophenol	266	264	268
Perylene-d12 (Internal Standard)	264	260	265
Phenanthrene	178	176	179
Phenanthrene-d10 (Internal Standard)	188	N/A	N/A
Phenol	94	66	65
Phenol-d5/d6 (Surrogate Standard)	99	42	71

Analyte	Primary	Secondary	Tertiary
Pyrene	202	101	100
Terphenyl-d14 (Surrogate Standard)	244	122	N/A

** Bis(2-chloro-1-methylethyl) ether or (2,2'-oxybis(1-chloropropane)) was formally known as bis(2-chloroisopropyl)ether.

APPENDIX SIM - Selected Ion Monitoring (SIM) Analysis

Selected Ion Monitoring (SIM) technique is sometimes required to achieve lower LOQ for certain compounds. Unless otherwise stated EPA 8270D/E method and quality control acceptance criteria requirements are followed for the analysis of SIM analysis.

For SIM analysis, the DFTPP instrument performance check is required as stated in 10.3.

A minimum of five-point initial calibration and daily calibration verification are required before sample analysis can begin. The initial calibration compounds must meet the minimum RF as listed in Table VI and the relative retention time (RRT) of each compound in each calibration standard must agree with 0.06 of the RRT. For South Carolina samples, a low-level scan must be performed initially or simultaneously. For most operations, the calibration standards are to be prepared at 0.10, 0.20, 0.40, 0.80, and 1.0 ng/uL,for each target compound of interest and the associated DMCs. If optional analysis by SIM is to be performed, the analyst shall add sufficient amount of the internal standard solution to each accurately measured aliquot of sample extract to result in a 0.40 ng/µL concentration of each internal standard.

The acceptance criteria for the initial calibration RSD ± 20% (15% for DOD) and calibration verification %D ± 20%. The acceptance criterion for the second source standard (ICV) is 30% of the expected concentration (20% for DOD).

The concentration for each of the initial calibration standard is project dependent. The calibration verification standard must be at/or near the mid-level standard of the initial calibration.

When setting up the instrument acquisition method, the dwell time and resolution parameters are defaulted to 100ms and low, respectively. In the “dwell” column, enter the amount of time to spend sampling a specific ion. 100ms is satisfactory for two to three ions. For more than three ions, use a shorter dwell time (such as 30 or 50ms) to ensure there will be enough data points to define the peaks.

TABLE SIM-I. PAS - WCOL Method 8270D/E (SIM) Standard Reporting List and Reporting Limits

Analytes	CAS Number	Aqueous (µg/L)	Soils (µg/kg)
1,4-Dioxane	123-91-1	0.20	6.7
1-Methylnaphthalene	90-12-0	0.20	6.7
2-Methylnaphthalene	91-57-6	0.20	6.7
Acenaphthene	83-32-9	0.20	6.7
Acenaphthylene	208-96-8	0.20	6.7
Anthracene	120-12-7	0.20	6.7
Benzo(a)anthracene	56-55-3	0.20	6.7
Benzo(a)pyrene	50-32-8	0.20	6.7
Benzo(b)fluoranthene	205-99-2	0.20	6.7
Benzo(g,h,i)perylene	191-24-2	0.20	6.7
Benzo(k)fluoranthene	207-08-9	0.20	6.7
Chrysene	218-01-9	0.20	6.7
Dibenz(a,h)anthracene	53-70-3	0.20	6.7
Fluoranthene	206-44-0	0.20	6.7
Fluorene	86-73-7	0.20	6.7
Indeno(1,2,3-cd)pyrene	193-39-5	0.20	6.7
Naphthalene	91-20-3	0.20	6.7
Pentachlorophenol	87-86-5	1	33
Phenanthrene	85-01-8	0.20	6.7
Pyrene	129-00-0	0.20	6.7

TABLE SIM-II. PAS - WCOL Method 8270D/E (SIM) LCS/MS Recovery Limits

Analytes	Aqueous (%)	Soils (%)
1,4-Dioxane	40-90	10-120
1-Methylnaphthalene	34-124	19-129
2-Methylnaphthalene	70-130	70-130
Acenaphthene	70-130	70-130
Acenaphthylene	70-130	70-130
Anthracene	70-130	70-130
Benzo(a)anthracene	70-130	70-130
Benzo(a)pyrene	70-130	70-130
Benzo(b)fluoranthene	70-130	70-130
Benzo(g,h,i)perylene	70-130	70-130
Benzo(k)fluoranthene	70-130	70-130
Chrysene	70-130	70-130
Dibenz(a,h)anthracene	70-130	70-130
Fluoranthene	70-130	70-130
Fluorene	70-130	70-130
Indeno(1,2,3-cd)pyrene	70-130	70-130
Naphthalene	70-130	70-130
Pentachlorophenol	70-130	70-130
Phenanthrene	70-130	70-130
Pyrene	70-130	70-130

TABLE SIM-III. PAS - WCOL Method 8270D/E (SIM) Surrogate Recovery Limits

Analytes	Aqueous (µg/L)	Soils (µg/kg)
2-Methylnaphthalene-d10	15-139	17-119
Fluoranthene-d10	23-154	37-135
1,4-Dioxane-d8	40-90	10-120

APPENDIX 625.1

MODIFICATIONS REQUIRED FOR ANALYSIS OF WASTEWATER FOLLOWING METHOD 625.1
REQUIREMENTS FOR METHOD 625.1

Method 625.1 is required for demonstration of compliance with NPDES wastewater discharge permits. The standard analyte list and reporting limits are listed in Table 625.1-1.

2. This method can be applied only to aqueous matrices.

3. The tune period for this method is defined as 12 hours that begins after tune and calibration verification, not to exceed 15 hours total.

4. Initial calibration curve requirements:

   4.1 Target compounds must have RSD/RSE ± 35%.

   4.2 Average RF may be used if the RSD < 35%. Alternatively, a linear fit curve can be used, but must be weighted inversely proportional to the concentration. The R^2 must be greater than 0.92 or the RSE must be less than 35%. If not, recalibrate.

5. Continuing calibration verification requirements: The CCV must be from a second source. When analytes fail acceptance criteria, analyze a second CCV. If the analytes still do not pass, repair the system or recalibrate and repeat. See Table 625.1-2 for acceptance criteria.

6. MS/MSD and LCS requirements: A full analyte spike is required for method 625.1. The spiking levels are given in Table 625.1-3. For every batch of 20 samples, a LCS and a MS/MSD pair are required.

7. Qualitative identification

   · The relative intensities of ions must agree to within ± 20% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 30% and 70%.)

TABLE 625.1-I. PAS - WCOL Method 625.1 Standard Reporting List and Reporting Limits

Analytes	CAS Number	Aqueous (µg/L)
1,2,4-Trichlorobenzene	120-82-1	2
1,3-Dichlorobenzene	541-73-1	2
1,4-Dichlorobenzene	106-46-7	2
1.2-Dichlorobenzene	95-50-1	2
Bis (2-chloro-1methylethyl)ether or (2,2'-oxybis(1-chloropropane))	108-60-1	2
2,4,6-Trichlorophenol	88-06-2	2
2,4-Dichlorophenol	120-83-2	2
2,4-Dimethylphenol	105-67-9	2
2,4-Dinitrophenol	51-28-5	10
2,4-Dinitrotoluene	121-14-2	4
2.6-Dinitrotoluene	606-20-2	4
2-Chloronaphthalene	91-58-7	2
2-Chlorophenol	95-57-8	2
2-Nitrophenol	88-75-5	4
3,3'-Dichlorobenzidine	91-94-1	4
4,6-Dinitro-2-methylphenol	534-52-1	10
4-Bromophenyl phenyl ether	101-55-3	2
4-Chloro-3-methylphenol	59-50-7	2
4-Chlorophenyl phenyl ether	7005-72-3	2
4-Nitrophenol	100-02-7	4
Acenaphthene	83-32-9	2
Acenaphthylene	208-96-8	2
alpha-Terpineol	98-55-5	2
Anthracene	120-12-7	2
Azobenzene*	103-33-3	2
Benzidine	92-87-5	10
Benzo(a)anthracene	56-55-3	2
Benzo(a)pyrene	50-32-8	2
Benzo(b)fluoranthene	205-99-2	2
Benzo(g,h,i)perylene	191-24-2	2
Benzo(k)fluoranthene	207-08-9	2
Bis(2-chloroethoxy)methane	111-91-1	2
Bis(2-chloroethyl)ether	111-44-4	2
Bis(2-ethylhexyl)phthalate	117-81-7	10
Butyl benzyl phthalate	85-68-7	2
Chrysene	218-01-9	2
Dibenz(a,h)anthracene	53-70-3	2
Diethylphthalate	84-66-2	2

Analytes	CAS Number	Aqueous (µg/L)
Dimethyl phthalate	131-11-3	2
Di-n-butyl phthalate	84-74-2	10
Di-n-octylphthalate	117-84-0	2
Fluoranthene	206-44-0	2
Fluorene	86-73-7	2
Hexachlorobenzene	118-74-1	2
Hexachlorobutadiene	87-68-3	2
Hexachlorocyclopentadiene*	77-47-4	10
Hexachloroethane	67-72-1	2
Indeno(1,2,3-cd)pyrene	193-39-5	2
Isophorone	78-59-1	2
Naphthalene	91-20-3	2
Nitrobenzene	98-95-3	2
N-Nitrosodimethylamine*	62-75-9	2
N-Nitroso-di-n-propylamine	621-64-7	2
N-Nitrosodiphenylamine	86-30-6	2
Pentachlorophenol	87-86-5	10
Phenanthrene	85-01-8	2
Phenol	108-95-2	2
Pyrene	129-00-0	2

* Compounds are not on the EPA 625.1 list, but the Priority Pollutant list

TABLE 625.1-II. PAS - WCOL Method 625.1 LCS/MS/MSD Recovery Limits

Analytes	ICV/CCV % Recovery	DOC Std Dev (%)	DOC % Recovery	LCS/MS/MSD % Recovery	LCS/MS/MSD RPD Limit (%)
1,2,4-Trichlorobenzene	61-130	30	57-130	44-142	50
1,3-Dichlorobenzene	60-140	42	D-172	D-172	42
1,4-Dichlorobenzene	60-140	32	20-124	20-124	32
1.2-Dichlorobenzene	60-140	31	32-129	32-129	31
Bis (2-chloro-1-methylethyl)ether or (2,2'-oxybis(1-chloropropane))	60-140	76	36-166	36-166	76
2,4,6-Trichlorophenol	69-130	35	52-129	37-144	58
2,4-Dichlorophenol	64-130	30	53-122	39-135	50
2,4-Dimethylphenol	58-130	35	42-120	32-120	58
2,4-Dinitrophenol	39-173	79	D-173	D-191	132
2,4-Dinitrotoluene	53-130	25	48-127	39-139	42
2.6-Dinitrotoluene	68-137	29	68-137	50-158	48
2-Chloronaphthalene	70-130	15	65-120	60-120	24
2-Chlorophenol	55-130	37	36-120	23-134	61
2-Nitrophenol	61-163	33	45-167	29-182	55
3,3'-Dichlorobenzidine	18-213	65	8-213	D-262	108
4,6-Dinitro-2-methylphenol	56-130	122	53-130	D-181	203
4-Bromophenyl phenyl ether	70-130	26	65-120	53-127	43
4-Chloro-3-methylphenol	68-130	44	41-128	22-147	73
4-Chlorophenyl phenyl ether	57-145	36	38-145	25-158	61
4-Nitrophenol	35-130	79	13-129	D-132	131
Acenaphthene	70-130	29	60-132	47-145	48
Acenaphthylene	60-130	45	54-126	33-145	74
alpha-Terpineol	60-140	40	46-163	46-163	40
Anthracene	58-130	40	43-120	27-133	66
Azobenzene	60-140	40	D-150	D-150	40
Benzidine	60-140	40	D-150	D-150	40
Benzo(a)anthracene	42-133	32	42-133	33-143	53
Benzo(a)pyrene	32-148	43	32-148	17-163	72
Benzo(b)fluoranthene	42-140	43	42-140	24-159	71
Benzo(g,h,i)perylene	13-195	61	D-195	D-219	97
Benzo(k)fluoranthene	25-146	38	25-146	11-162	63
Bis(2-chloroethoxy)methane	52-164	32	49-165	33-184	54
Bis(2-chloroethyl)ether	52-130	65	43=126	12-158	108

Analytes	ICV/CCV % Recovery	DOC Std Dev (%)	DOC % Recovery	LCS/MS/MSD % Recovery	LCS/MS/MSD RPD Limit (%)
Bis(2-ethylhexyl)phthalate	43-137	50	29-137	8-158	82
Butyl benzyl phthalate	43-140	36	D-140	D-152	60
Chrysene	44-140	53	44-140	17-168	87
Dibenz(a,h)anthracene	13-200	75	D-200	D-227	126
Diethylphthalate	47-130	60	D-120	D-120	100
Dimethyl phthalate	50-130	110	D-120	D-120	183
Di-*n*-butyl phthalate	52-130	28	8-120	1-120	47
Di-*n*-octylphthalate	21-132	42	19-132	4-146	69
Fluoranthene	47-130	40	43-121	26-137	66
Fluorene	70-130	23	70-120	59-121	38
Hexachlorobenzene	38-142	33	8-142	D-152	55
Hexachlorobutadiene	68-130	38	38-120	24-120	62
Hexachlorocyclopentadiene *	60-140	4	D-150	D-150	40
Hexachloroethane	55-130	32	55-120	40-120	52
Indeno(1,2,3-cd)pyrene	13-151	60	D-151	D-171	99
Isophorone	52-180	56	47-180	21-196	93
Naphthalene	70-130	39	36-120	21-133	65
Nitrobenzene	54-158	37	54-158	35-180	62
N-Nitrosodimethylamine*	60-140	50	D-150	D-150	50
N-Nitroso-di-*n*-propylamine	59-170	52	14-198	D-230	87
N-Nitrosodiphenylamine	60-140	40	D-150	D-150	40
Pentachlorophenol	42-152	52	38-152	14-176	86
Phenanthrene	67-130	24	65-120	54-120	39
Phenol	48-130	39	17-120	5-120	64
Pyrene	70-130	30	70-120	52-120	49

* Compounds are not on the EPA 625.1 list, but the Priority Pollutant list

TABLE 625.1-III. Method 625.1 LCS and MS Compounds and Spike Concentrations

LCS Compounds	Spiking Level in Extract (µg/mL)
1,2,4-Trichlorobenzene	40
1,3-Dichlorobenzene	40
1,4-Dichlorobenzene	40
1.2-Dichlorobenzene	40
Bis (2-chloro-1methylethyl)ether or 2,2'-oxybis(1-chloropropane)	40
2,4,6-Trichlorophenol	40
2,4-Dichlorophenol	40
2,4-Dimethylphenol	40
2,4-Dinitrophenol	80
2,4-Dinitrotoluene	40
2.6-Dinitrotoluene	40
2-Chloronaphthalene	40
2-Chlorophenol	40
2-Nitrophenol	40
3,3'-Dichlorobenzidine	40
4.6-Dinitro-2-methylphenol	40
4-Bromophenyl phenyl ether	40
4-Chloro-3-methylphenol	40
4-Chlorophenyl phenyl ether	40
4-Nitrophenol	80
Acenaphthene	40
Acenaphthylene	40
alpha-Terpineol	80
Anthracene	40
Azobenzene	40
Benzidine	200
Benzo(a)anthracene	40
Benzo(a)pyrene	40
Benzo(b)fluoranthene	40
Benzo(g,h,i)perylene	40
Benzo(k)fluoranthene	40
Dibenz(a,h)anthracene	40
Bis(2-chloroethoxy)methane	40
Bis(2-chloroethyl)ether	40
Bis(2-ethylhexyl)phthalate	40
Butyl benzyl phthalate	40
Chrysene	40
Diethylphthalate	40

LCS Compounds	Spiking Level in Extract (µg/mL)
Dimethyl phthalate	40
Di-n-butyl phthalate	40
Di-n-octylphthalate	40
Fluoranthene	40
Fluorene	40
Hexachlorobenzene	40
Hexachlorobutadiene	40
Hexachlorocyclopentadiene	200
Hexachloroethane	40
Indeno(1,2,3-cd)pyrene	40
Isophorone	40
Naphthalene	40
Nitrobenzene	40
N-Nitrosodimethylamine	40
N-Nitroso-di-n-propylamine	40
N-Nitrosodiphenylamine	40
Pentachlorophenol	80
Phenanthrene	40
Phenol	40
Pyrene	40

APPENDIX 8270E

The following is a summary of the changes made to 8270E, Rev. 6 as compared to the procedures and criteria stated in this SOP. A complete list of all the changes may be found in SW846, Semi-volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 6, Update VI, June 2018, Test Methods for Evaluating Solid Waste, Method 8270E, Appendix A.

Topic	8270D	8270E
Mass Spectrometer Requirements	'Capable of scanning from 35 to 500 amu every 1 sec or less, using 71 volts (nominal) electron energy in the electron impact ionization mode.'	'Capable of acquiring mass spectra from mass/charge (m/z) 35 to 500 at a rate fast enough to acquire at least 5 (but preferably 10 or more) mass spectra across each chromatographic peak of interest, using 70 volts (nominal) electron energy in the electron impact ionization mode.
Tune frequency	'The GC/MS must be tuned to meet the recommended DFTPP criteria prior to the initial calibration and for each twelve-hour period during which analyses are performed.'	'The GC/MS must meet DFTPP criteria prior to the ICAL.' 'Daily analysis of the GC/MS tune check solution is no longer required as part of the CCV. The analyst should, however, closely monitor chromatography as well as target and IS responses in the CCV for deterioration in the system.' NOTE: In an effort to monitor the system on an on-going basis, the laboratory will continue to run the tune check every 12-hour shift. The twelve-hour time will start with the injection of the last ICAL standard in sequences where an ICAL is performed and at the injection of the CCV in sequences when the ICAL is not necessary.
Tune requirements	See Table 5	See Table 8270E-2

Topic	8270D	8270E
Minimum RF	…'It is also recommended that a minimum RF for the most common target analytes, as noted in Table 4 [SW-846 8270D], be demonstrated for each individual calibration level as a means to ensure that these compounds are behaving as expected.'	…'Because the minimum RFs in Table 4 [SW-846 8270E] were determined using specific ions and instrument conditions that may vary, it is neither expected nor required that all analytes meet these minimum RFs. The information is provided as guidance only. The laboratory should establish procedures in its SOP (e.g., laboratory established minimum RFs, signal-to-noise (S/N) checks, etc.) to ensure that the instrument is working properly and that the calibration standards were correctly prepared.' See Table 8270E-1 in this Appendix.

Table 8270E - I: Guidance Response Factor Criteria for Initial Calibration Using the Suggested Ions from Table 1 [SW-846 8270E Rev.6, Update VI, June 2018]

Semi-volatile Compound	Guidance Min Response Factor (RF)
Acenaphthene	0.9
Acenaphthylene	0.9
Acetophenone	0.01
Anthracene	0.7
Atrizine	0.01
Benzaldehyde	0.01
Benze (a)anthracene	0.8
Benzo(a)pyrene	0.7
Benzo(b)fluoranthene	0.7
Benzo(g,h,i)perylene	0.5
Benzo(k)fluoranthene	0.7
1,1'-Biphenyl	0.01
Bis(2-chloroethoxy)methane	0.3
Bis(2-chloroethyl)ether	0.7
Bis-(2-ethylhexyl)phthalate	0.01
4-Bromophenyl-phenyl ether	0.1
Butyl benzyl phthalate	0.01
Caprolactam	0.01
Carbazole	0.01
4-Chloroaniline	0.01
4-Chloro-3-methylphenol	0.2
2-Chloronaphthalene	0.8
2-Chlorophenol	0.8
4-Chlorophenyl-phenyl ether	0.4
Chrysene	0.7
Dibenz(a,h)anthracene	0.4
Dibenzofuran	0.8
Di-n-butyl phthalate	0.01
3,3'-Dichlorobenzidine	0.01
2,4-Dichlorophenol	0.2
Diethyl phthalate	0.01
Dimethyl phthalate	0.01

Semivolatile Compound	Guidance Min Response Factor (RF)
2,4-Dimethylphenol	0.2
4,6-Dintro-2-methylphenol	0.01
2,4-Dinitrophenol	0.01
2,4-Dintrotoluene	0.2
2,6-Dinitrotoluene	0.2
Di-n-octyl-phthalate	0.01
Fluoranthene	0.6
Fluorene	0.9
Hexachlorobenzene	0.1
Hexachlorobutadiene	0.01
Hexachlorocyclopentadiene	0.05
Hexachloroethane	0.3
Indeno(1,2,3-cd)pyrene	0.5
Isophorone	0.4
2-Methylnaphthalene	0.4
2-Methylphenol	0.7
4-Methylphenol	0.6
Naphthalene	0.7
2-Nitroaniline	0.01
3-Nitroaniline	0.01
4-Nitroaniline	0.01
Nitrobenzene	0.2
2-Nitrophenol	0.1
4-Nitrophenol	0.01
N-nitroso-di-n-propylamine	0.5
N-nitrosodiphenylamine	0.01
2,2’-Oxybis-(1-chloropropane)	0.01
Pentachlorophenol	0.05
Phenanthrene	0.7
Phenol	0.8
Pyrene	0.6
1,2,4,5-Tetrachlorobenzene	0.01
2,3,4,6-Tetrachlorophenol	0.01
2,4,5-Trichlorophenol	0.2
2,4,6-Trichlorophenol	0.2

TABLE 8270E-II. DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
68	< 2% of m/z 69
69	Present
70	< 2% of m/z 69
197	< 2% of m/z 198
198	Base peak or present
199	5 – 9% of m/z 198
365	> 1% of m/z 198 (Base Peak)
441	<150% of m/z 443
442	Base Peak or present
443	15 – 24% of m/z 442

TABLE 8270E-III: Summary of QC Criteria for use with 8270E

Quality Control Type	Minimum Frequency	Specification	Suggested Acceptance Criteria
Instrument performance check (tune)	Prior to initial calibration	≤50 ng Decafluorotriphenylphosphine (DFTPP) injected	Meet ion ration criteria for reference compound: DFTPP, or alternative documented criteria; Tailing factor ≤ 2 and degradation ≤ 20%
Initial calibration (ICAL)	Prior to analyzing samples, and as needed if continuing performance criteria cannot be met	5 point minimum for response factor (RF) and linear regressions (LR), 6 points minimum for quadratic regression (QR) >90% of reported target analytes meet ICAL criteria	For average RF calibration model: ≤20% relative standard deviation (RSD) of RFs For LR or QR model: R≥0.995, R^2≥099. Independent of calibration model: Low standard recalculation (refit) should be ±50% of true value; other standards > lower limit quantitation (LLOQ) are recommended to be ± 30% of true value. Or, relative standard error (RSE) ≤ 20%
Initial Calibration Verification (ICV)	After each ICAL and prior to analyzing samples	Prepared from different source of target analytes than ICAL standards	Calculated concentrations of target analytes are ± 30% of true value
Continuing Calibration Verification (CCV)	Once at least every 12 hours	>80% of target analytes meet CCV criteria	Targets are ≤ 20% difference or drift; IS responses are within 50% to 200% of midpoint of ICAL or average of ICAL ISs; and retention times for ISs have not shifted > 30 seconds relative to ICAL
Blanks	One method blank (MB) per preparation	NA	Target analyte concentrations in blank are

Quality Control Type	Minimum Frequency	Specification	Suggested Acceptance Criteria
	batch of 20 or fewer samples; Instrument blanks as needed		< ½ LLOQ, or ≤ 10% of concentration in field sample
Laboratory Control Sample (LCS)	One per preparation batch of 20 or fewer samples	NA	Meets recovery criteria established by laboratory
Duplicates and Matrix Spikes	A duplicate and matrix spike, or matrix spike/matrix spike duplicate per preparation of 20 or fewer samples (not required per batch)	NA	Performance-based or project-defined recovery criteria for matrix spikes; Relative percent difference (RPD) criteria between measured concentrations in sample and laboratory duplicate or in matrix spike and matrix spike duplicate
Surrogates	Added to each sample	NA	Performance-based recovery criteria established by the laboratory or criteria chosen for the project
Internal Standards (IS)	Added to each sample	NA	IS response is within 50-200% of the response of the same IS in the midpoint ICAL standard (or average of ICAL) or most recent CCV
Qualitative Analyte Identification	Each target analyte	NA	RT in sample is within ± 10 seconds of RT in midpoint ICAL or CCV standard) or within ± 10 seconds relative to the shift of the associated IS (delta RT of the IS ± 10 seconds). Characteristic ion(s) within ± 30% of expected ion ratio in reference spectrum; or match to reference library spectra ≥ 0.8 (only for full

Quality Control Type	Minimum Frequency	Specification	Suggested Acceptance Criteria
			mass range acquisition modes)

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ENVIRONMENTAL SCIENCES

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Kelly M. Nance
Quality Manager

1/13/2022 9:31:57 AM
Bradley E. Belding
Operations Manager

1/7/2022 3:56:35 PM
Kristina P. Bouknight
Environmental Health and Radiation Safety Officer

1/14/2022 6:14:51 PM
Maria S. Gonzalez
Supervisor

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: pH by Electrometric Measurement / pH Paper Method
METHOD: SM4500-H B-2011 / 9040C / 150.1 / 9041A / 9045D
ISSUER: Pace ENV - Local Quality - WCOL

Table of Contents

1.0 Scope And Application

This standard operating procedure (SOP) describes the laboratory procedure for the determination of pH by electrometric measurement and paper method.

1.1 Target Analyte List

Type of Sample	Method
Aqueous	EPA 150.1 / SM 4500-H+ B-2011 / 9040C
Non-Aqueous	9041A
Solid	9040C / 9045D

Methods 4500-H+ B-2011 and EPA Method 150.1 are applicable to the measurement of pH in drinking, surface, and saline waters, domestic and industrial wastes, and acid rain (atmospheric deposition).

EPA Method 150.1 is approved for the analysis of samples for compliance monitoring under the Safe Drinking Water Act.

Standard Method 4500-H+ B-2011 is approved for the analysis of samples for compliance monitoring under the Safe Drinking Water Act and Clean Water Act.

EPA SW-846 Method 9040C is used to measure the pH of aqueous wastes and those multiphase wastes where the aqueous phase constitutes at least 20% of the total volume of waste.

EPA SW-846 Method 9040C is approved for the analysis of samples for solid and hazardous waste compliance monitoring.

EPA SW-846 Method 9041A may be used to measure pH as an alternative to Method 9040C in cases where pH measurements by Method 9040C are not possible (oily samples), however:

Method 9041A is not applicable to wastes that contain components that may mask or alter the pH paper color change.

pH paper is not considered to be as accurate a form of pH measurement as pH meters. For this reason, pH measurement taken with Method 9041A cannot be used to define a waste as corrosive or non-corrosive.

EPA SW-846 Method 9045D is used in measuring pH in soils and waste samples. Wastes may be solids, sludges, or non-aqueous liquids. If water is present, it must constitute less than 20% of the total volume of the sample.

EPA SW-846 Method 9045D is approved for the analysis of samples for solid and hazardous waste compliance monitoring.

The corrosivity of concentrate acids and bases, or of concentrated acids and bases mixed with inert substances, cannot be measured. The pH measurement requires some water content.

pH is reported as standard units (SU) or pH units. There is no reporting limit or method detection limit applicable with pH analysis.

2.0 Summary of Method

2.1 EPA Method 150.1, Standard Method 4500-H+ B-2011, and EPA SW-846 Method 9040C: The pH of the sample is determined electrometrically using a combination electrode. The measuring device is calibrated using a series of solutions of known pH.

2.2 EPA SW-846 Method 9041A: The approximate pH of the waste is determined with wide-range pH paper. Then a more accurate pH determination is made using “narrow-range” pH paper whose accuracy has been determined using a series of buffers or by comparison with a calibrated pH meter.

2.3 EPA SW-846 Method 9045D: In method 9045D, 20 g of sample is mixed with 20 mL of reagent water and the pH of the resulting aqueous solution is then determined electrometrically using a combination electrode.

NOTE: If sample absorbs the water, add more reagent water in increments of 10 mLs until there is sufficient free liquid to analyze the pH.

3.0 Interferences

3.1 EPA Method 150.1, Standard Method 4500-H+ B-2011, EPA SW-846 Method 9040C, and EPA SW-846 Method 9045D

The glass electrode, in general, is not subject to solution interferences from color, turbidity, colloidal matter, oxidants, reductants, or high salinity.

Samples with very low or very high pH may give incorrect readings on the meter. For samples with a true pH of > 10, the measured pH may be incorrectly low. This error can be minimized by using a “low sodium error” electrode. Strong acid solutions, with a true pH of < 1, may give incorrectly high pH measurements.

Coating of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gentle wiping or detergent washing, followed by reagent water rinsing. An additional treatment with hydrochloric acid (10%) may be necessary to remove any remaining film. The electrode can also be cleaned per the manufacturer’s instructions.

Temperature effects on the electrometric measurement of pH arise from two sources. The first is caused by the change in electrode output at various temperatures. This interference can be controlled with instruments having temperature compensation or by calibration the electrode-instrument system at the temperature of the samples. The second source is the change of pH inherent in the sample at various temperatures. This error is sample dependent

and cannot be controlled; it should therefore be noted by reporting both the pH and temperature at the time of analysis.

3.2 EPA SW-846 Method 9041A

Certain wastes may inhibit or mask changes in the pH paper. This interference can be determined by adding small amounts of acid or base to a small aliquot of the waste and observing whether the pH paper undergoes the appropriate changes.

CAUTION: The addition of acid or base to wastes may result in violent reactions or the generation of toxic fumes (e.g., hydrogen cyanide). These tests should be performed in a well-ventilated hood when dealing with unknown samples.

4.0 Definitions

Refer to the Laboratory Quality Manual [QAMP ME0012K] for a glossary of common lab terms and definitions.

5.0 Health and Safety

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

6.0 Sample Collection, Preservation, Holding Time, And Storage

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory may perform sample collection for sample to be analyzed by this SOP in accordance with the *Field Services* SOP [FS SOP ME001BS]. Refer to this SOP for these instructions.

The laboratory will provide containers for the collection of samples upon client request for analytical services. Bottle kits are prepared in accordance with the *Sample Container Shipping* SOP [AD SOP ME001DS]. For this test method, immediately after sample collection, samples should be checked for X and X and field treated. The bottle kits provided by the laboratory should include field test kits and treatment reagent.

Requirements for container type, preservation, and field quality control (QC) for the common list of test methods offered by Pace are listed in the *Pace-WCOL Analytical Methods List* [ME002BS].

This is a field parameter and pH measurements should be done at the time of sampling. Maximum sample holding time for these methods is 15 minutes. Every effort must be made to analyze pH samples as soon as possible from sample receipt. Sample data analyzed outside holding time criteria is addressed in the report narrative.

General Requirements

Matrix	Routine Container	Min. Sample Amount[1]	Preservation	Holding Time
Aqueous	250 mL plastic	50mL	Thermal: ≤ 6°C Chemical: None	15 mins.
Solid	2 oz glass Teflon-lined lid	50g	Thermal: ≤ 6°C Chemical: None	15 mins.

[1]*Minimum amount needed for each discrete analysis.*

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with the *Sample Receiving* SOP [AD SOP ME0013H]. Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are stored at 4°C until sample preparation. Prepared samples (extracts, digestates, distillates, other) are stored at 4°C until sample analysis.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 30 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

7.0 Equipment And Supplies

7.1 Equipment

7.1.1 **Analytical balance** – Capable of accurately weighing to the nearest 0.0001 g

7.1.2 **pH Meter** – Commercially available from any approved vendor. pH meter should be accurate and reproducible to 0.1 pH unit with a range of 0 to 14 SU and equipped with a temperature compensation adjustment. Thermo Fisher Orion Star A111; VWR SB70P or equivalent.

NOTE: Refer to the Major Operational Equipment List [QA Control Log ME001PM] for specific details regarding the equipment utilized during this procedure.

7.2 Supplies

7.2.1 Combination electrode with silver/silver chloride references accurate to ±0.05 SU.

7.2.2 Thermometer – temperature sensor attached to pH meter

7.2.3 Wide-range pH paper

7.2.4 Narrow-range pH paper – With a distinct color change for every 0.5 pH unit (e.g., Alkacid Full-Range pH Kit, Fisher Scientific or equivalent)

7.2.5 Kimwipes or other equivalent soft tissue

7.2.6 Polypropylene snap-cups, 70mL and 120mL capacity

7.2.7 Magnetic stirrer and Teflon-coated stir bars

8.0 Reagents And Standards

8.1 Reagents

8.1.1 **Reagent water –** PAS-WCOL employs a series of in-house deionized (DI) tanks to purify the incoming water. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks. See the Deionized Water System SOP [QA SOP ME0012S] for further information.

8.1.2 **Hydrochloric acid (HCl),** concentrated – Reagent grade or better. May be purchased from any approved vendor that can provide a certificate of analysis. Follow manufacturer expiration date.

8.1.3 **Hydrochloric acid solution (1:4)** – Carefully add 20mL of concentrated HCl to approximately 70mL of dilution water. Allow to cool to room temperature and dilute to 100mL. Follow manufacturer expiration date of stock material.

8.1.4 **Sodium hydroxide (NaOH), 10N** – May be purchased from any approved vendor that can provide a certificate of analysis. Follow manufacturer expiration date.

8.1.5 **Sodium hydroxide (NaOH) solution (0.1 N)** – Add 1 mL of 10N NaOH to approximately 70 mL of dilution water. Dilute to 100mL and invert to mix. Follow manufacturer expiration date of stock material.

8.2 Standards

8.2.1 **Certified pH buffers** – pH 1, 4, 7, 10 and 12 buffers. May be purchased from any approved vendor that can provide a certificate of analysis. Follow manufacturer expiration date.

NOTE: pH buffer certificates of analysis must be obtained online or the pH buffer labels must be removed and retained for standard traceability.

9.0 Procedure

NOTE: Automatic temperature compensator (ATC) verifications are performed as required by the *Equipment and Instrumentation* SOP [QA SOP ME002JT].

NOTE: The balance is verified at the beginning of each analytical day using a certified weight set. Refer to the *Equipment and Instrumentation* SOP [QA SOP ME002JT] for balance verification procedures and acceptance criteria.

9.1. Equipment Preparation

9.1.1 Support Equipment

9.1.2 Instrument

9.1.2.1 Routine Instrument Operating Conditions

9.2. Initial Calibration

9.2.1 Calibration Design

3-point calibration of the meter is performed prior to sample analysis:

Refer to appendix B and C for calibration instructions specific to the meter being used. Obtain fresh buffer to be used for calibration and for all calibration verifications.

Rinse the electrode with reagent water and blot dry with a soft tissue between measurements.

For corrosivity characterization (EPA SW-846 Methods 9040C and 9045D), the calibration of the pH meter should include a buffer of pH 2 for acidic wastes and a pH 12 buffer for caustic wastes; also, for corrosivity characterization, the sample must be measured at 25 ± 1 °C if the pH of the waste is above 11.0 SU.

Each batch of narrow-range pH paper must be calibrated versus certified pH buffers or by comparison with a pH meter which has been calibrated with certified pH buffers. If the incremental reading of the narrow-range pH paper is within 0.5 pH units, then the agreement between the buffer and the calibrated pH meter with the paper must be within 0.5 pH units.

9.2.2 ICAL Evaluation

9.2.3.1 **Calibration slope** - The slope must be 92-102%.

Record all data in the pH Meter Calibration for INM [INM Form ME0014P].

9.2.3.2 **Initial Calibration Verification (ICV**) - A second source 7.0 SU buffer is analyzed immediately following each calibration. This buffer result must agree within ± 0.05 pH unit of the true value before sample analysis begins. If it fails to meet this criterion, recalibrate the meter

9.2.3.3 Buffers are single use only. Buffers must be changed after each use

9.2.3 **Continuing Calibration Verification (CCV)** - 1 level of standard (4, 7, or 10 pH buffer) is run at the end of every 10 samples and at the end of the analytical day to verify that run is in control. CCV must be within ± 0.05 pH unit of its true value. If CCV result falls outside of its acceptance range, two consecutive CCVs must be analyzed and show results within ± 0.05 pH unit of the true value before continue with sample analysis. Otherwise, recalibrate the meter. In all cases, when the initial CCV falls outside of acceptance limit, the 10 samples above that CCV must be reanalyzed.

9.2.3.1 Buffers are single use only. Buffers must be changed after each use

9.3. Sample Preparation

9.3.1 Sample Preparation of Soils:

Weigh 20g of soil into a snap-cup and add 20mL of reagent water.

Place a magnetic stir-bar into the snap-cup, close the snap-cup lid, and place snap-up on magnetic stirrer.

Continuously stir the suspension for 5 minutes. Record the stirring time.

NOTE: Additional water may be added if working with hygroscopic soils and salts or other problematic matrices. Add additional water in 10 mL increments until there is enough free liquid to measure the pH.

Remove snap-cup from the magnetic stirrer and allow soil suspension to settle for 1 hour to allow most of the suspended clay to settle out from the suspension. Record the settling time. Alternatively, centrifuge or filter off the aqueous phase for pH measurement.

9.3.2 Sample Preparation of Waste Materials:

Weigh 20g of waste material into a snap-cup and add 20mL of reagent water.

Place a magnetic stir-bar into the snap-cup, close the snap-cup lid, and place the snap-cup on a magnetic stirrer.

Continuously stir the suspension for 5 minutes. Record the stirring time.

Remove the snap-cup from the magnetic stirrer and allow the suspension to settle for 15 minutes to allow most of the suspended waste to settle out from the suspension. Alternatively, centrifuge or filter off the aqueous phase for pH measurement.

NOTE: If the waste is hygroscopic and absorbs all of the reagent water, repeat the preparation procedures using 20g of waste and 40mL of reagent water. Additional water may be added if working with hygroscopic soils and salts or other problematic matrices. Add additional water in 10 mL increments until there is enough free liquid to measure the pH.

NOTE: If the supernatant is multi-phasic, decant the oily phase and measure the pH of the aqueous phase. The electrode may need to be cleaned if it becomes coated with an oily material.

9.4. Analysis

9.1.1 EPA Method 150.1, EPA SW-846 Method 9040C, and Standard Method 4500 H+ B-2011

Transfer a well-mixed aliquot of sample into a snap-cup, using a sufficient volume to cover the electrode-sensing element tip and to give adequate space for the magnetic stirring bar. Do not dilute the sample.

Put a stir bar in the snap-cup and place on magnetic stirrer. Stir gently at a constant rate in order to mix the sample well.

With the meter set to pH mode, immerse the sensing element tip of the pH electrode and thermometer (ATC) directly into the sample. Allow time for the meter reading to stabilize. Record the sample pH and temperature.

Repeat measurements on successive aliquots of sample (fresh portions of the same sample) until values differ by less than 0.1 SU.

Rinse the pH electrode and the thermometer (ATC) between samples with reagent water and blot dry with a soft tissue.

If the pH of the sample falls outside the calibrated range of the meter, i.e. >10.0 or <4.0, an additional known buffer must be read to bracket the value of the sample. This buffer must read within ±0.05 SU. If the buffer value falls outside of this range the sample must be put in a separate batch with the meter calibrated with buffers bracketing the value of the sample.

Record data using the pH (SM 4500H+ B-2011, SW-846 Method 9040C, 150.1) form [INM Form ME001O0].

9.1.2 EPA SW-846 Method 9045D:

9.1.2.1 Sample Preparation of Soils:

Weigh 20g of soil into a snap-cup and add 20mL of reagent water.

Place a magnetic stir-bar into the snap-cup, close the snap-cup lid, and place snap-up on magnetic stirrer.

Continuously stir the suspension for 5 minutes. Record the stirring time.

NOTE: Additional water may be added if working with hygroscopic soils and salts or other problematic matrices. Add additional water in 10 mL increments until there is enough free liquid to measure the pH.

Remove snap-cup from the magnetic stirrer and allow soil suspension to settle for 1 hour to allow most of the suspended clay to settle out from the suspension. Record the settling time. Alternatively, centrifuge or filter off the aqueous phase for pH measurement.

9.1.2.2 Sample Preparation of Waste Materials:

Weigh 20g of waste material into a snap-cup and add 20mL of reagent water.

Place a magnetic stir-bar into the snap-cup, close the snap-cup lid, and place the snap-cup on a magnetic stirrer.

Continuously stir the suspension for 5 minutes. Record the stirring time.

Remove the snap-cup from the magnetic stirrer and allow the suspension to settle for 15 minutes to allow most of the suspended waste to settle out from the suspension. Alternatively, centrifuge or filter off the aqueous phase for pH measurement.

NOTE: If the waste is hygroscopic and absorbs all of the reagent water, repeat the preparation procedures using 20g of waste and 40mL of reagent water. Additional water may be added if working with hygroscopic soils and salts or other problematic matrices. Add additional water in 10 mL increments until there is enough free liquid to measure the pH.

NOTE: If the supernatant is multi-phasic, decant the oily phase and measure the pH of the aqueous phase. The electrode may need to be cleaned if it becomes coated with an oily material.

9.1.2.3 pH Measurement of Soils and Waste Materials:

Immerse the electrode just below the surface of the suspension. Record the sample pH and temperature.

Rinse the pH electrode and the thermometer (ATC) between samples with reagent water and blot dry with a soft tissue.

If the pH of the sample falls outside the calibrated range of the meter, i.e. >10.0 or <4.0, an additional known buffer must be read to bracket the value of the sample. This buffer must read within ±0.05 SU. If the buffer value falls outside of this range the sample must be put in a separate batch with the meter calibrated on buffers bracketing the value of the sample.

Record data using the pH (Soil) form [INM Form ME001SX].

9.1.2.4 EPA CLP SOW ISM 02.4 and SOM02.4:

Weigh 20g of sample into a snap-cup and add 20mL of reagent water.

NOTE: Additional water may be added if working with hygroscopic soils and salts or other problematic matrices. Add additional water in 10 mL increments until there is enough free liquid to measure the pH.

Place a magnetic stir-bar into the snap-cup, close the snap-cup lid, and place the snap-cup on a magnetic stirrer.

Continuously stir the suspension for 1 hour. Record the stirring time.

Remove the snap-cup from the magnetic stirrer and allow the suspension to settle for at least 1 hour to allow most of the suspended waste to settle out from the suspension. Difficult samples may be filtered or centrifuged to separate the aqueous layer for pH determination.

NOTE: If the supernatant is multi-phasic, decant the oily phase and measure the pH of the aqueous phase. The electrode may need to be cleaned if it becomes coated with an oily material.

Immerse the electrode just below the surface of the suspension. Record the sample pH and temperature.

Rinse the pH electrode and the thermometer (ATC) between samples with reagent water and blot dry with a soft tissue.

If the pH of the sample falls outside the calibrated range of the meter, i.e. >10.0 or <4.0, an additional known buffer must be read to bracket the value of the sample. This buffer must read within ±0.05 SU. If the buffer value falls outside of this range the sample must be put in a separate batch with the meter calibrated on buffers bracketing the value of the sample.

Record data using the pH (Soil) form [INM Form ME001SX].

9.1.3 EPA SW-846 Method 9041A (pH paper method):

A representative aliquot of the waste must be tested first with wide-range pH paper to determine the approximate pH.

The appropriate narrow-range pH paper is chosen and the pH of a second aliquot of the waste is determined. This measurement should be performed in duplicate. Place one or two drops of sample on the pH paper and record the pH of the sample.

Identification of interference - Certain wastes may inhibit or mask changes in the pH paper. This interference can be determined by adding small amounts of acid or base to a small aliquot of the sample and observing whether the pH paper undergoes the appropriate changes:

Take a third aliquot of the waste, approximately 2 mL in volume, and add diluted HCl (7.5) dropwise until a pH change is observed. Note the color change.

Add NaOH solution (7.6) to a fourth aliquot and note the color change. (Wastes that have a buffering capacity may require additional acid or base to result in a measurable pH change.)

The observation of the appropriate color change is a strong indication that no interferences have occurred.

CAUTION: Addition of acid or base to samples may result in violent reactions or the generation of toxic fumes. These tests should be performed in a well-ventilated hood when dealing with unknown samples.

Record all data using the pH (Paper) form [INM Form ME001RF].

10.0 Data Analysis And Calculations

10.1 Quantitative Identification

10.1.1 Methods SM 4500-H+ B-2011, EPA 150.1, and EPA SW-846 9040C

Sample results are read directly from the meter and are reported to the nearest 0.1 pH unit; the last measured sample aliquot is reported.

Sample temperature, at the time of analysis, must be reported in conjunction with pH reporting. Temperature measurements are read directly from the meter and are reported in °C.

10.1.2 Method EPA SW-846 9045D

Sample results are read directly from the meter and are reported to the nearest 0.1 pH unit; the last measured sample aliquot is reported.

Soil samples are reported as “soil pH measured in water at __°C”, where “__°C” is the temperature at which the test was conducted.

Waste samples are reported as “waste pH measured in water at __°C”, where “__°C” is the temperature at which the test was conducted.

10.1.3 Method EPA SW-846 9041A

Sample results are reported directly from the pH strip color comparison guide, which is printed on the pH strip box. Results are reported as whole numbers.

10.2 Calculations

See the Laboratory Quality Assurance Manual [QAMP ME0012K] for equations for common calculations.

The difference between duplicate sample results is calculated as follows:

Difference = X1 – X2 Where: X1 = the sample pH

X2 = the duplicate pH

11.0 Quality Control and Method Performance

11.1 Quality Control

The following QC samples are prepared and analyzed with each batch of samples. Refer to Appendix B for acceptance criteria and required corrective action.

QC Item	Frequency
Sample Duplicate	At least 1 per batch

11.2 Instrument QC

The following Instrument QC checks are performed. Refer to Appendix B for acceptance criteria and required corrective action.

QC Item	Frequency
Initial Calibration	Every day samples are analyzed
Initial Calibration Verification	After calibration
Continuing Calibration Verification	After every 10 samples and at end of analytical day

11.3 Method Performance

11.3.1. **Initial Calibration Verification (ICV)** - A second source 7.0 SU buffer is analyzed immediately following each calibration. This buffer result must agree within ± 0.05 pH

unit of the true value before sample analysis begins. If it fails to meet this criterion, recalibrate the meter.

11.3.2. **Sample Duplicates** – SM4500 H+ requires sample duplicates to be performed at a frequency of at least one per matrix type per batch of 20 samples or fewer. This procedure requires repeat measurements on successive aliquots of sample (fresh portions of the same sample) until readings vary by < 0.1 pH units. This satisfies the sample duplicate requirement specified in SM 4500 H+. Sample duplicates are still required for EPA SW-846 9045D and 9041A at the frequency stated above. 9045D duplicate measurements must not vary by more than 0.2 SU. 9041A duplicate measurements must not vary by more than 0.5 SU.

11.3.3. **Continuing Calibration Verification (CCV**) – 1 level of standard (4, 7, or 10 pH buffer) is run at the end of every 10 samples and at the end of the analytical day to verify that run is in control. CCV must be within ± 0.05 pH unit of its true value. If CCV result falls outside of its acceptance range, two consecutive CCVs must be analyzed and show results within ± 0.05 pH unit of the true value before continuing with sample analysis. Otherwise, recalibrate the meter. In all cases, when the initial CCV falls outside of acceptance limit, the 10 samples above that CCV must be reanalyzed.

11.3.4. **Check buffers** –If any sample read has a pH outside the calibrated range of the meter (4.0-10.0) another buffer (1.0 or 12) must be read that as nearly as possible encompasses the pH value of the sample. These buffers must read within ± 0.05 pH unit of the true value before sample analysis continues. If the buffer does not read within the specified range the meter must be recalibrated using the buffers necessary to bracket the sample pH. Consult the group leader or a more experienced analyst if needed for assistance in this procedure.

11.3.4.1. Buffers are single use only. Each aliquot of buffer must be changed after each use.

11.5 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to the *Demonstration of Capability* SOP [QA SOP ME001F2] for more information.

12.0 Data Review And Corrective Action

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical

record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employee's complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to the *Data Review* SOP [QA SOP ME003LP] for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

Refer to Appendix B for a complete summary of QC, acceptance criteria, and recommended corrective actions for QC associated with this test method.

13.0 Pollution Prevention And Waste Management

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

14.0 Modifications

14.1 A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

15.0 Responsibilities

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 Attachments

16.1 APPENDIX A: QC SUMMARY

16.2 APPENDIX B: CALIBRATION INSTRUCTIONS FOR ORION STAR A111 PH METER

16.3 APPENDIX C: CALIBRATION INSTRUCTIONS FOR VWR SB70P PH METER

17.0 References

NOTE: Where references exclude a date or edition, the latest edition of the referenced document adopted/recognized by the laboratory's accreditation bodies applies. Refer to the Quality Assurance Management Plan [QAMP ME0012K] for details.

17.1 Standard Methods for the Examination of Water and Wastewater, 22nd edition, 2011 – pH Value 4500 H+ B-2011.

17.2 SW-846, Test Method for Evaluating Solid Waste, Third Edition – pH Electrometric Measurement, Method 9040 C, Revision 3, November 2004.

17.3 SW-846, Test Method for Evaluating Solid Waste, Third Edition – pH Paper Method, Method 9041 A, Revision 1, July 1992.

17.4 SW-846, Test Method for Evaluating Solid Waste, Third Edition – Soil and Waste pH, Method 9045 D, Revision 4, November 2004.

17.5 Standard Methods for the Examination of Water and Wastewater, 22nd edition, 2011 – 4020 Quality Assurance/Quality Control.

17.6 Methods for the Chemical Analysis of Water and Wastes (MCAWW) (EPA/600/4-79/020), pH, Electrometric Method, Method 150.1, Issued 1971; Editorial Revision 1978 and 1982.

17.7 USEPA Contract Laboratory Program SOW for Organic Analysis, SOM02.4, September 2016

17.8 USEPA Contract Laboratory Program SOW for Inorganic Analysis, ISM02.4, September 2016.

18.0 Revision History

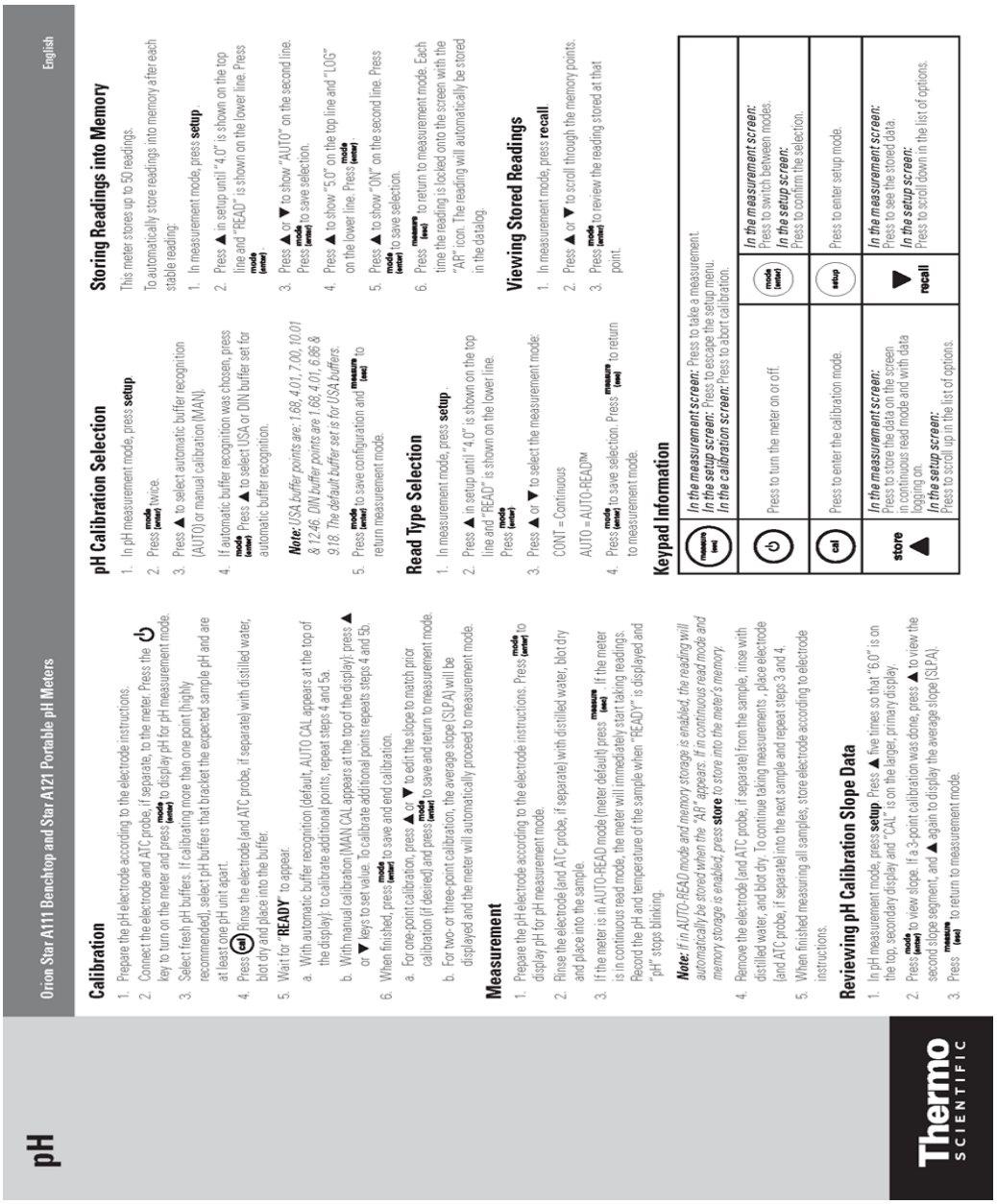
Revision #	Revision Date	Section Modified	Modification	Reason for Modification
-09	**01/21/2022**	11.3.4.1	Added section to denote that buffers are single use	Compliance audit (Qualtrax WF # 31911)

Appendix A: QC Summary

QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
ICAL	At instrument set up, prior to use and daily prior to analysis	Slope: 92% to 106$	Identify and correct source of problem, repeat	None. Do not proceed with analysis
ICV	After each ICAL.	± 0.05 SU of true value	Re-analyze. If repeat failure, repeat ICAL.	None.
CCV	After every 10 samples, and at end of analytical batch.	± 0.05 SU of true value	Re-analyze two consecutive passing CCVs with results within ± 0.05 SU of true value. Otherwise, recalibrate the meter. Repeat analysis on last 10 samples.	None.
Sample Duplicate	At least one per batch.	SM4500-H B-2011 - < 0.1 SU difference 9041A - < 0.5 SU difference All other methods: < 0.2 SU difference	NA	Quality duplicate out of criteria.

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: pH by Electrometric Measurement / pH Paper Method
METHOD: SM4500-H B-2011 / 9040C / 150.1 / 9041A / 9045D
ISSUER: Pace ENV - Local Quality - WCOL

Appendix B: Calibration Instructions for Orion Star A111 pH Meter



pH

Orion Star A111 Benchtop and Star A121 Portable pH Meters

English

Calibration

1. Prepare the pH electrode according to the electrode instructions.
2. Connect the electrode and ATC probe, if separate, to the meter. Press the ⏻ key to turn on the meter and press mode (enter) to display pH for pH measurement mode.
3. Select fresh pH buffers. If calibrating more than one point (highly recommended), select pH buffers that bracket the expected sample pH and are at least one pH unit apart.
4. Press cal. Rinse the electrode (and ATC probe, if separate) with distilled water, blot dry and place into the buffer.
5. Wait for "**READY**" to appear.
   a. With automatic buffer recognition (default, AUTO CAL appears at the top of the display): to calibrate additional points, repeat steps 4 and 5a.
   b. With manual calibration (MAN CAL appears at the top of the display): press ▲ or ▼ keys to set value. To calibrate additional points repeats steps 4 and 5b.
6. When finished, press mode (enter) to save and end calibration.
   a. For one-point calibration, press ▲ or ▼ to edit the slope to match prior calibration (if desired) and press mode (enter) to save and return to measurement mode.
   b. For two- or three-point calibration, the average slope (SLPA) will be displayed and the meter will automatically proceed to measurement mode.

Measurement

1. Prepare the pH electrode according to the electrode instructions. Press mode (enter) to display pH for pH measurement mode.
2. Rinse the electrode (and ATC probe, if separate) with distilled water, blot dry and place into the sample.
3. If the meter is in AUTO-READ mode (meter default) press measure (esc). If the meter is in continuous read mode, the meter will immediately start taking readings. Record the pH and temperature of the sample when "READY" is displayed and "pH" stops blinking.

   Note: *If in AUTO-READ mode and memory storage is enabled, the reading will automatically be stored when the "AR" appears. If in continuous read mode and memory storage is enabled, press* **store** *to store into the meter's memory.*
4. Remove the electrode (and ATC probe, if separate) from the sample, rinse with distilled water, and blot dry. To continue taking measurements , place electrode (and ATC probe, if separate) into the next sample and repeat steps 3 and 4.
5. When finished measuring all samples, store electrode according to electrode instructions.

Reviewing pH Calibration Slope Data

1. In pH measurement mode, press **setup**. Press ▲ five times so that "6.0" is on the top, secondary display and "CAL" is on the larger, primary display.
2. Press mode (enter) to view slope. If a 3-point calibration was done, press ▲ to view the second slope segment, and ▲ again to display the average slope (SLPA).
3. Press measure (esc) to return to measurement mode.

pH Calibration Selection

1. In pH measurement mode, press **setup**
2. Press mode (enter) twice.
3. Press ▲ to select automatic buffer recognition (AUTO) or manual calibration (MAN).
4. If automatic buffer recognition was chosen, press mode (enter). Press ▲ to select USA or DIN buffer set for automatic buffer recognition.

   Note: *USA buffer points are: 1.68, 4.01, 7.00, 10.01 & 12.46. DIN buffer points are 1.68, 4.01, 6.86 & 9.18. The default buffer set is for USA buffers.*
5. Press mode (enter) to save configuration and measure (esc) to return measurement mode.

Read Type Selection

1. In measurement mode, press **setup**
2. Press ▲ in setup until "4.0" is shown on the top line and "READ" is shown on the lower line. Press mode (enter).
3. Press ▲ or ▼ to select the measurement mode:

   CONT = Continuous

   AUTO = AUTO-READ™
4. Press mode (enter) to save selection. Press measure (esc) to return to measurement mode.

Storing Readings into Memory

This meter stores up to 50 readings.

To automatically store readings into memory after each stable reading:

1. In measurement mode, press **setup**
2. Press ▲ in setup until "4.0" is shown on the top line and "READ" is shown on the lower line. Press mode (enter).
3. Press ▲ or ▼ to show "AUTO" on the second line. Press mode (enter) to save selection.
4. Press ▲ to show "5.0" on the top line and "LOG" on the lower line. Press mode (enter).
5. Press ▲ to show "ON" on the second line. Press mode (enter) to save selection.
6. Press measure (esc) to return to measurement mode. Each time the reading is locked onto the screen with the "AR" icon. The reading will automatically be stored in the datalog.

Viewing Stored Readings

1. In measurement mode, press **recall**
2. Press ▲ or ▼ to scroll through the memory points.
3. Press mode (enter) to review the reading stored at that point.

Keypad Information

measure (esc)	***In the measurement screen:*** Press to take a measurement. ***In the setup screen:*** Press to escape the setup menu. ***In the calibration screen:*** Press to abort calibration.
⏻	Press to turn the meter on or off.
mode (enter)	***In the measurement screen:*** Press to switch between modes. ***In the setup screen:*** Press to confirm the selection.
cal	Press to enter the calibration mode.
setup	Press to enter setup mode.
store ◀	***In the measurement screen:*** Press to store the data on the screen in continuous read mode and with data logging on. ***In the setup screen:*** Press to scroll up in the list of options.
▶ recall	***In the measurement screen:*** Press to see the stored data. ***In the setup screen:*** Press to scroll down in the list of options.

Thermo SCIENTIFIC

Appendix C: Calibration Instructions for VWR SB70P pH Meter

pH Calibration

1. Prepare the electrode according to the electrode user guide.
2. In the setup mode, select the buffer set (USA or EUro) that will be used for the automatic buffer recognition feature.
3. In the measurement mode, press (≡) until the arrow icon points to the top line, press (△) until the **pH** icon is shown and press (↙) to begin the calibration.
4. Rinse the electrode and ATC probe with distilled water and place into the buffer.
5. Wait for the **pH** icon to stop flashing.
   a. Automatic buffer recognition – When the **pH** icon stops flashing the meter will display the temperature-corrected pH value for the buffer.
   b. Manual calibration – When the **pH** icon stops flashing the meter will display the actual pH value read by the electrode. Press (↵) until the first digit to be changed is flashing, press (△) / (▽) to change the value of the flashing digit and continue to change the digits until the meter displays the temperature-corrected pH value of the buffer. Once the pH buffer value is set, press (↵) until the decimal point is in the correct location.
6. Press (↙) to proceed to the next calibration point and repeat steps 4 and 5 or press (i) to save and end the calibration.
7. The actual electrode slope, in percent, will be displayed in the main field and SLP will be displayed in the lower field.
   a. For a one point calibration, press (↵) and (△) / (▽) to edit the slope and press (i) to return to the measurement mode.
   b. For a two or more point calibration, the meter will automatically proceed to the measurement mode after the slope is displayed.

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Document Title: Herbicides by Gas Chromatographic Analysis

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Date Information

Effective Date: Thursday, September 23, 2021

Notes

Document Notes:

All Dates and Times are in Eastern Standard Time Zone.

Signature Manifest

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All dates and times are in Eastern Standard Time Zone.

ME00157-09

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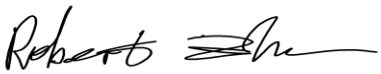
Daniel J. Wright
General Manager 1

8/17/2021 4:52:38 PM
Kelly M. Nance
Quality Manager



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Robert Zhu
Technical Specialist

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Bradley E. Belding
Operations Manager

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Kristina P. Bouknight
Environmental Health and Radiation Safety Officer

8/24/2021 8:30:10 AM
James C. Geiger
Supervisor

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Herbicides by Gas Chromatographic Analysis
METHOD: EPA SW-846 Method 8151A
ISSUER: Pace ENV - Local Quality - WCOL

Table of Contents

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Herbicides by Gas Chromatographic Analysis
METHOD: EPA SW-846 Method 8151A
ISSUER: Pace ENV - Local Quality - WCOL

1.0 Scope and Application

This standard operating procedure (SOP) describes the laboratory procedure for the determination of organic analytes by Gas Chromatography (GC). The procedures are based on SW-846 methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA). Individual analytes and methods are described in the appendices.

North Carolina and Department of Defense-Quality Systems Manual (DoD-QSM) requirements discussed in section 17 and the appendices of this SOP supersede the requirements of normal procedures.

2.0 Summary of Method

After the initial preparation step, (*Extraction of Chlorinated Herbicides* [EXT SOP ME001IM]), the sample is introduced to the GC and concentrations of target analytes are measured by the detector response within a defined retention time window, relative to the response to standard concentrations. Internal or external standardization procedures are used as specified in the method appendices.

3.0 Interferences

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. If interference is detected, it is necessary to determine if the source of interference is in the preparation and/or cleanup of the samples; then take corrective action to eliminate the problem.

3.2 The use of high purity reagents, solvents, and gases helps to minimize interference problems. Refer to ME001J8, the Solvent Purity Check policy, for the procedure for testing the purity of solvents used for extraction.

3.3 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample.

3.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples.

3.5 Co-elution of target analytes with non-targets can occur, resulting in false positives or biased high results.

4.0 Definitions

Refer to the *Laboratory Quality Manual* [QAMP ME0012K] for a glossary of common lab terms and definitions.

4.1 ***Non-conformance Memo (NCM)*** - A form used to document a non-conforming event. An analyst must document a non-conformance memo when a non-conforming event occurs. A non-conforming event may include the reporting of analytical data outside of method or SOP criteria, or when there

is a deviation from a written policy or procedure. Information in an NCM may be used by project managers to flag data in the report narrative, or by the quality department to track trends and initiate corrective actions, where applicable. Additional information on the NCM policy and procedure is located in the *Complaints and Nonconformances* SOP [QA SOP ME001BO].

5.0 Health and Safety

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

6.0 Sample Collection, Preservation, Holding Time, and Storage

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory performs samples collection for samples to be analyzed by this SOP in accordance with the *Field Services* SOP [FS SOP ME001BS]. Refer to this SOP for these instructions.

The laboratory will provide containers for the collection of samples upon client request for analytical services. Bottle kits are prepared in accordance with the *Sample Container Shipping* SOP [AD SOP ME001DS]. For this test method, immediately after sample collection, samples should be checked for X and X and field treated. The bottle kits provided by the laboratory should include field test kits and treatment reagent.

Requirements for container type, preservation, and field quality control (QC) for the common list of test methods offered by Pace are listed in the *Pace-WCOL Analytical Methods List* [ME002BS].

General Requirements

Matrix	Routine Container	Min. Sample Amount[1]	Preservation	Holding Time
Aqueous	250 mL Amber Glass (2X)	250 mL	Thermal: ≤ 6°C Chemical: Hydrochloric Acid (HCl)	Collection to Prep: 7 days Prep to Analysis: 40 days
Soil	4 oz. Glass with Teflon-lined lid	50 g	Thermal: ≤ 6°C Chemical: None	Collection to Prep: 14 days Prep to Analysis: 40 days

[1]Minimum amount needed for each discrete analysis.

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with the *Sample Receiving* SOP [AD SOP ME0013H]. Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are stored at X°C until sample preparation. Prepared samples (extracts, digestates, distillates, other) are stored at X°C until sample analysis.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 28 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

7.0 Equipment and Supplies

NOTE: Refer to the *Major Operational Equipment List* [QA Control Log ME001PM] for specific details regarding the equipment utilized during this procedure.

7.1 Equipment

7.1.1 Gas Chromatograph/Electron Capture Detect (GC/ECD) – An analytical system complete with a temperature-programmable gas chromatograph suitable for injection port(s), and electron capture detectors (ECD)

7.1.2 Data Acquisition System –HP Chemstation 6. Data rate is 20Hz.

7.1.3 Data Analysis System – Target 4.14 by Thruput.

7.2 Supplies

7.2.1 Column – 30 m x 0.28 mm I.D. x 0.25 µm film thickness silicon-coated fused-silica capillary column (Restek MXT-1 or equivalent). Alternate columns are acceptable if they provide acceptable performance.

7.2.2 Syringe – 10-µL, 25-µL, 50-µL, 100-µL, 1-mL Hamilton Laboratory grade syringes or equivalent.

7.2.3 Gases for carrier and make-up: Hydrogen and Helium

8.0 Reagents and Standards

NOTE: Other volumes of standards and reagents may be prepared to account for expected usage. As long as all ratios are kept constant, this is not considered a deviation from the approved procedure.

NOTE: All stored reagents and standards must be labeled as required by the Preparation and *Documentation of Laboratory Standards and Reagents* SOP [QA SOP ME001HG], the *Contingency and Emergency Preparedness Plan* [HS SOP ME0012D], the *Safety Manual* [Corp Manual COR-MAN-HSE], and the *Laboratory Quality Manual* [QAMP ME0012K].

8.1 Reagents

8.1.1 ***Reagent water*** – Pace employs a series of in-house deionized (DI) tanks to purify the incoming water. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks. See the *Deionized Water System* SOP [QA SOP ME0012S] for further information.

8.2 Standards

8.2.1 ***Stock Standards*** – Stock standards are purchased as certified solutions or prepared from pure solutions.

Stock standards are stored according to manufacturer recommendations, either 4°C ± 2°C, –10 to –20°C, or ambient temperature, depending on the media the standards were prepared with. All stock standards must be protected from light. Stock standard solutions should be brought to room temperature before using.

Purchased standards, if unopened, expire on the date specified by the manufacturer. Opened stock standards and all working standards expire 6 months from the date opened or prepared or on the expiration date of their parent standards, whichever is sooner. All standards' expiration dates are checked prior to use and disposed of if not within that expiration date.

8.2.2. ***Calibration Standards*** – Semivolatile calibration standards are prepared as dilutions of the stock standards. Surrogates standards are used as specified in the method appendices. Prepared semivolatile calibration solutions must be kept refrigerated at 4 ± 2 °C and protected from light. The standards must be replaced at least every six months or sooner if comparison with check standards indicates a problem.

8.2.3. ***Gases for carrier and make-up*** - Hydrogen and Nitrogen.

8.2.4. ***Quality Control (QC) Standards*** – QC standards (LCS and matrix spiking standards) are prepared and stored in the same way as calibration standards. They must be made from a stock independent from the calibration standards.

Note: The following QC are made from a second source stock independent from the calibration standards: ICV, LCS, and the MS/MSD.

9.0 Procedure

9.1 Equipment Preparation

9.1.1 Instrument

9.1.1.1 Gas Chromatograph/Flame Ionization Detector (GC/FID) – An analytical system complete with a temperature-programmable gas chromatograph suitable for direct injection or split/splitless injection, including analytical columns, and gases.

9.2 Initial Calibration

9.2.1 All herbicide calibration standards must go through the esterification process. These standards are called procedural standards. Follow the procedure provided in the Extraction of Chlorinated Herbicides [EXT SOP ME001IM].

9.2.2 If external calibration is used, prepare standards containing each analyte of interest at a minimum of five concentration levels. Analyze from low to high concentration. The low- level standard should be at or below the LOQ. The other standards define the working range of the detector. Recommended calibration levels are given in the appendices.

9.2.3 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 new columns, replacing the ECD detector, etc. A new calibration is not required after clipping the column, replacing the septum or syringe, or other minor maintenance. All maintenance must be recorded in the instrument's maintenance log with the date and initials of the analyst performing the maintenance.

9.2.4 With the exception of Section 10.5 below, it is not acceptable to remove points from a calibration curve for the purpose of meeting criteria, unless the points are the highest or lowest of the curve and the reporting limit and/or linear range is adjusted accordingly. In any event, at least 5 points must be included in the calibration curve.

9.2.5 A level may be removed from the calibration if the reason can be clearly documented, for example, a broken vial. A minimum of five levels must remain in the calibration. The documentation must be retained with the initial calibration. Alternatively, if the analyst believes that a point on the curve is inaccurate, the point may be reanalyzed and the reanalysis used for the calibration. All initial calibration points must be analyzed without any changes to instrument conditions; all points must be analyzed within 24 hours.

9.3 ***External Standard Calibration*** – Quantitation by the external standard method assumes a proportional relationship between the calibration run and the analyte in the sample. To use this approach, introduce each calibration standard into the GC using the technique that will be used for samples. The ratio of the peak height or area response to the mass or concentration injected may be used to prepare a calibration curve.

$$CF = A / X$$

Where:

CF = Calibration Factor
A = Peak Area (or Height) of the compound in the standard
X = Concentration of standard (µg/mL)

Some data systems may use the inverse of this formula. This is acceptable so long as the same formula is used for standards and samples. However, if matrix interferences would make quantitation using peak area inaccurate for a particular sample, then peak height may be used as a substitute.

9.4 ***Calibration curve fits*** – Average calibration factor/response factor or linear regression may be used to fit the data. Average calibration factor/response factor may be used if the % RSD of the calibration factor of the analyte in the calibration standard is ≤ 20 %. Linear regression may be used if the % RSD is > 20 %. The correlation coefficient for the fit must be ≥ 0.995. The Target data system uses R2 to measure the fit, R2 must be ≥ 0.990. The ICAL cannot be forced through zero. No sample analysis may be performed unless these criteria are met on both columns. See section 10.7.3 for additional methods used to determine calibration function acceptability.

9.4.1 *Average Calibration Factor/Response Factor* – The equation for average calibration factor is:

$$\overline{CF} = \frac{\sum_{i=1}^{n} CFi}{n}$$

Where:

CF = Average calibration factor

n = Number of calibration levels

$\sum_{i=1}^{n} CFi$ = Sum of response factors for each calibration level

9.4.2 *Linear Regression* – The response must increase with increasing concentration. The linear fit uses the following functions:

9.4.3 *External Standard*

$$y = ax + b \text{ or } x = (y-b)/a$$

Where:

y = Instrument response
x = Concentration
a = Slope
b = Intercept

9.4.4 Either of the two methods described below in sections 10.7.2.1 and 10.7.2.2 may be used to determine calibration function acceptability for linear curves. Both procedures refit the

calibration data back to the calibration model and evaluates the difference between the measured and the true amounts or concentrations used to create the model.

9.4.5 *% Error*

$$\% \text{ Error} = \frac{X_i - X'_i}{X_i} \times 100$$

Where:

x'i = Measured amount of analyte at calibration level i, in mass or concentration units.
xi = True amount of analyte at calibration level i, in mass or concentration units.

Percent error between the calculated and expected amounts of an analyte should be ≤ 30% for all standards. For some data uses, ≤50% may be acceptable for the lowest calibration point.

9.4.6 *Relative Standard Error (RSE)* – RSE

$$RSE = \sqrt{\sum_{i=1}^{n}\left[\frac{x'_i - x_i}{x_i}\right]^2 \Big/ (n-p)}$$

Where:

x'i = Measured amount of analyte at calibration level i, in mass or concentration units.
xi = True amount of analyte at calibration level i, in mass or concentration units.
p = Number of terms in fitting equation (average = 1, linear = 2)
n= Number of calibration points.

The RSE acceptance limit criterion for the calibration model is the same as the RSD limit (Section 9.3).

9.4.7 Non-standard analytes are sometimes requested. For these analytes, it may be acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five-point initial calibration. This action must be with client approval. If the analyte is detected in any of the samples, a five-point initial calibration must be generated and the sample(s) reanalyzed for quantitation.

9.4.8 ***Initial Calibration Verification (ICV)/Second Source Calibration Verification*** – The ICAL is verified by analyzing an ICV. The ICV is a standard obtained from a source independent of the source of standards for the initial calibration. Its concentration should be at or near the middle of the calibration range. The ICV is analyzed immediately after the initial calibration. The value of the second source must agree within 30 % of the expected value of the ICAL before sample analysis can begin. If the ICV fails, repeat. If the second ICV fails, maintenance must be performed and two consecutive ICVs must pass or a new ICAL must be analyzed before sample analysis can begin.

9.4.9 ***Continuing Calibration Verification (CCV)***, also known as the Instrument Performance Check.

9.4.10 *Opening CCV* – The ICAL must be verified at the beginning of each analytical sequence, after every 10 field samples, and at the end of the sequence. The opening CCV must pass all analytes of interest within + 20 %. Any CCV that has samples reported after it is considered an opening CCV, even if it is analyzed in the middle of a sequence.

If the opening CCV fails, repeat once. If the second analysis fails, instrument maintenance must be performed and two consecutive CCVs must pass or a new ICAL must be performed before sample analysis can occur.

The center of each retention time window is updated with each 12-hour CCV. The widths of the windows will remain the same (refer to Section 11.6 for details).

After any major maintenance (e.g. column change), the RT window must be calculated for each analyte and surrogate.

9.4.11 *Closing CCV* – Any CCV analyzed after samples is considered a closing CCV for those samples.

If the samples preceding the closing CCV are non-detect and the closing CCV is >120 %, the data may be reported.

If the closing CCV < 80 %, all associated samples must be reanalyzed.

If the samples preceding the closing CCV have a concentration > LOQ, the closing CCV must agree within + 20 %. If the closing CCV fails, all associated samples must be reanalyzed.

A single CCV may be considered both an opening and a closing CCV if the CCV is in the middle of a sequence. At no time can there be more than 12 hours between bracketing CCVs.

Non-detect results: A single sample may be reported from a combination of front and back column results. If only one column passes the CCV criteria, only ND results may be reported for that column, and that analyte.

When a concentration above the LOQ (or MDL if “J” flags are required) is detected and confirmed, the lower of the two results is reported. In this case, both front and back columns must pass the opening and closing CCV. (Some client specific programs may require that the lower or both results will be reported.)

Samples quantitated by external standard methods must be bracketed by calibration verification standards that meet the criteria listed above.

9.4.12 *% Difference Calculation*

9.4.12.1 <u>% Difference for External Standard Method</u>

$$\%D = \frac{CF_c - \overline{CF}}{\overline{CF}} \times 100$$

Where:

% D = Percent difference
CFc = Calibration factor from the continuing calibration
CF = Average calibration factor from the initial calibration

9.4.13 *% Drift Calculation* - % Drift is used for comparing the continuing calibration to a linear curve. The criteria for % drift are the same as for % difference.

$$\% \text{ Drift} = \frac{\text{Calc. Conc.} - \text{Theoretical Conc.}}{\text{Theoretical Conc.}} \text{ X } 100$$

9.4.14 The retention times of all the analytes in the continuing calibration verification standards must be within the retention time windows as determined in the initial calibration.

9.5 Sample Preparation

9.5.1 See SOPs for the preparation procedure for aqueous and solid samples.

9.5.2 ***Cleanup*** – Cleanup procedures are referenced in the appendices.

9.6 Analysis

9.6.1 ***Gas Chromatography*** – Chromatographic conditions for individual methods are presented in the appendices.

9.6.2 ***Sample Introduction*** – In general, semi-volatile analytes are introduced by direct injection of the extract. Samples, standards, and QC must be introduced using the same procedure.

9.6.3 ***Analytical Sequence*** – An analytical sequence starts with an initial calibration or a daily calibration. Refer to the individual method appendices for method specific details of daily calibrations and analytical sequences.

The daily calibration includes analyses of standards containing all target analytes and updating the retention time windows.

If there is a break in the analytical sequence of greater than 12 hours, a new analytical sequence must be started with a daily calibration.

9.7 ***Retention Time Windows***

9.7.1 Retention time windows must be determined for all analytes and surrogates. Make an injection of all analytes of interest each day over a 72-hour period. Record the retention time for each single component analyte and surrogate to three decimal places. Calculate the mean and standard deviation of the three absolute retention times for each analyte and surrogate.

9.7.2 If the standard deviation of the retention times for a target compound is 0.000 (i.e., no difference between the absolute retention times), the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes.

9.7.3 The width of the retention time window for each analyte and surrogate is defined as plus or minus three (± 3) times the standard deviation of the mean absolute retention time established

during the 72-hour period. If the default standard deviation in Section 11.6.2 is used, the width of the window will be ± 0.03 minutes.

9.7.4 The centers of the windows are updated with the mid-point of the initial calibration or the CCV that starts the analytical sequence. The centers of the windows must be updated every 12 hours. The widths of the windows will remain the same until new windows are generated following the installation of a new column.

9.7.5 For DOD, the laboratory must calculate new retention time windows each time a new column is installed. The new windows must be generated before performing initial calibrations or analyzing samples.

9.8 *Corrective Action for Retention Times* – The retention times of all compounds in the continuing calibration that starts the sequence must be within the RT windows established during the initial calibration. If this condition is not met, a new initial calibration must be performed. Each subsequent continuing calibration must be within the retention time windows established by the CCV that started the 12-hour analytical sequence. If this condition is not met, all samples analyzed after the last compliant standard must be reanalyzed unless the following conditions are met for any compound that elutes outside the retention time window:

9.9 Daily Retention Time Windows – The center of the retention time windows are determined in Section 11.6.4. The retention time windows must be updated by the CCV at the beginning of each analytical sequence, but not for any other calibration verification standards.

9.10 Percent Moisture – Analytical results may be reported as dry or wet weight, as required by the client. Percent moisture must be determined if results will be reported as dry weight. Refer to the percent moisture SOP ME0013F for determination of percent moisture.

9.11 Procedural Variations – For compliance samples, all procedures outlined in this SOP must be followed without exception. In the event that a deviation from this SOP cannot be avoided (i.e., demonstrated and uncorrectable matrix interference, non-compatible matrix, insufficient sample amount due to client or laboratory error) it is imperative that an NCM is completed that clearly documents the anomaly. This anomaly must be communicated to the client such that corrective action activity including sample recollection can be performed. Based upon the type and severity of the anomaly, the results may not be appropriate for compliance reporting. Discussions between the client, the appropriate regulatory authority, and Shealy will be necessary to resolve certain anomalies. In any case, it is essential that any anomaly be documented with an NCM.

10.0 Data Analysis and Calculations

10.1 Qualitative Identification

10.1.1 Tentatively Identified Compounds (TICS)

Tentative identification occurs when a peak is found within the retention time window for an analyte, at a concentration above the reporting limit, or above the MDL if “J” flags are required. Confirmation is required on a second column. Refer to the appendices for test specific requirements for confirmation. Identification is confirmed if a peak is also present in the retention time window for that analyte on the confirmatory column, at a concentration greater than the reporting limit (MDL if “J” flags required). For confirmed results, the lower of the two results is reported.

If the relative percent difference (RPD) between the responses on the two columns is > 40 %, or if in the opinion of an experienced analyst the complexity of the matrix is resulting in false positives, the confirmation is suspect and the results are qualified. RPD is calculated using the following formula:

$$RPD = \frac{|X1 - X2|}{\frac{(X1 + X2)}{2}} x\ 100$$

Where: X = Result

The experience of the analyst should weigh heavily in the interpretation of the chromatogram. For example, sample matrix or laboratory temperature fluctuation may result in variation of retention times.

10.1.2 Manual Integration - Manual changes to automated integration is called manual integration. Manual integration is sometimes necessary to correct inaccurate automated integrations but must never be used to meet QC criteria or to substitute for proper instrument maintenance and/or method set-up. To assure that all manual integrations are performed consistently and are ethically justified, all manual integrations must be performed, reviewed, and recorded in accordance with corporate SOP ENV-SOP-CORQ-0006, Manual Integration.

10.1.3 ***Calibration Range*** – If concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half of the calibration range. It may be necessary to dilute samples due to matrix. In this case, an NCM is required.

10.1.4 ***Dilutions*** – Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hit or hits below 20 % of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50 % of the calibration range.

10.1.5 *Guidance for dilutions due to matrix* – If the sample is initially run at a dilution and only minor matrix peaks are found, then the sample should be reanalyzed at a more concentrated dilution. Analyst judgment is required to determine the most concentrated dilution that will not result in instrument contamination.

10.1.6 *Reporting Dilutions* – The most concentrate dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

10.2 ***Interferences*** – If peak detection is prevented by interferences, further cleanup should be attempted. If no further cleanup is reasonable, then elevation of reporting levels and/or lack of positive identification must be addressed in the case narrative.

10.3 Quantitative Identification

10.3.1 Sum the area of all peaks eluting between n-decane (C10) and n-octacosane (C-28). Subtract the surrogate area from the total area of DRO when calculating a sample result. This area is

used to calculate the DRO concentration. This area is generated by projecting a horizontal baseline between the retention times of C_{10} and C_{28}.

10.3.2 For samples showing chromatographic patterns similar to #2 diesel fuel, calculations will be based on the initial calibration curve for diesel and reported as such.

10.3.3 For samples showing chromatographic patterns other than #2 diesel fuel, calculations will be based on #2 diesel fuel initial calibration curve and reported as #2 diesel fuel. Qualifiers can be added to LIMS 4 upon sample upload for further pattern identification (See section 17).

10.3.4 Analyst shall establish a chromatographic library containing reference chromatograms such as motor oil, JP-4 jet fuel, kerosene, #5 fuel oil and other fuel types to allow for fingerprinting.

10.4 Calculations

Capabilities of individual data systems may require the use of different formulas than those presented here. When this is the case, the calculations used must be shown to be equivalent and must be documented in an appendix attached to this document.

See the *Laboratory Quality Assurance Manual* [QAMP ME0012K] for equations for common calculations.

10.3.1. *External Standard Calculations*

10.3.1.1. Aqueous Samples

$$\text{Conc. } (\mu L) = \frac{Ax \times Vt \times DF}{CF \times Vo}$$

Where:

Ax = Response for the analyte in the sample
DF = Dilution factor
Vt = Volume of total extract, µL
Vo = Volume of sample extracted or purged, mL
CF = Calibration factor, area or height/µg/mL, Section 9.3

If linear regression is used the following calculation is used:

$$\text{Conc. } = \frac{(y - b) \times Vt \times DF}{a \times Vo}$$

Where:

y = Instrument response
a = Slope
b = Intercept

10.3.1.2. Non-aqueous and Solid Samples

$$\text{Conc. (mg/kg)} = \frac{Ax \times Vt \times DF}{CF \times W \times D}$$

Where:

W = Weight of sample extracted, g

$$D = \frac{100 - \%\text{moisture}}{100}$$

(D = 1 if wet weight is required)

If linear regression is used the following calculation is used:

$$\text{Conc.} = \frac{(y - b) \times Vt \times DF}{a \times W \times D}$$

Where:

y = Instrument response
x = Concentration
a = Slope
b = Intercept

10.3.2. *Surrogate Recovery* – Concentrations of surrogate compounds are calculated using the same equations as for the target compounds. The response factor from the initial calibration is used. Surrogate recovery is calculated using the following equation:

$$\%\ \text{Recovery} = \frac{X1}{X2} x\ 100$$

Where:

X1 = Concentration (amount) found
X2 = Concentration (amount) spiked

11.0 Quality Control and Method Performance

11.1 Quality Control

The following QC samples are prepared and analyzed with each batch of samples. Refer to Appendix B for acceptance criteria and required corrective action.

QC Item	Frequency
Method Blank (MB)	1 per batch
Laboratory Control Sample (LCS)	1 per batch
Laboratory Control Sample Duplicate (LCSD)	As needed
Matrix Spike (MS)	1 per 10 samples
Matrix Spike Duplicate (MSD)	1 per 10 samples
Sample Duplicate –	1 per batch

11.1.1. ***Quality Control Batch*** – The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The quality control batch must contain a matrix spike/spike duplicate (MS/MSD), a laboratory control sample (LCS), and a method blank (MB). Laboratory generated QC samples (MB, LCS, and MS/MSD) do not count towards the maximum 20 samples in a batch. If there is insufficient sample to analyze an MS/MSD or MS and sample duplicate an LCSD may be performed to document precision and bias. Field QC samples are included in the batch count but cannot be used for matrix spikes. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate.

11.1.2. ***Control Limits*** – In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS/LCSD). These limits are maintained by QA, reviewed regularly and updated as needed. The recovery limits are mean recovery ± 3 standard deviations. Control limits for compliance with the DoD-QSM can be found in DoD tables for each determinative method.

All surrogate, LCS/LCSD, and MS/MSD recoveries must be entered into LIMS (when available) or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

Refer to the *Quality Assurance Management Plan* [QAMP, ME0012K], for further details of control limits.

11.1.3. ***Method Blank (Laboratory Reagent Blank)*** – One method blank must be processed with each preparation and/or analytical batch. The method blank consists of a similar matrix to the batch of associated samples in which no target analytes or interferences are present at concentrations that impact the analytical results. The method blank is to contain all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. Refer to Appendix DoD for method blank acceptance criteria for samples analyzed for the Department of Defense.

The method blank must not contain any analyte of interest less than ½ the LOQ or less than the project-specific requirements. If the method blank contains an analyte of interest less than ½ the LOQ or project-specific limits, then the method blank and associated samples must be reanalyzed. If the method blank contamination is confirmed, the entire batch must be re-prepared and reanalyzed unless the samples are not affected. Where permitted by the program area or client, the following exceptions apply. Any method blank that does not meet acceptance criteria, is flagged on the data report with a B flag.

- The contamination is not detected in the samples.
- The sample concentration is ≥ 10X the blank concentration.

To clarify the compounds of interest that are associated with each sample the LIMS generated worksheet printout for each sample will be contained in the batch data file. This worksheet printout will list the required target compounds and the reporting limits.

The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, corrective action should be taken. The method blank should be reanalyzed if the analyst feels that the failure was attributed to instrument problems. If the failure is due to a poor extraction, the entire batch must be sent for re-extraction.

11.1.4. ***Instrument Blank*** – Instruments must be evaluated for contamination during each 12-hour analytical run. This may be accomplished by analysis of a method blank. If a method blank is not available, an instrument blank must be analyzed. The solvent used for the instrument blank may vary with instrumentation. Surrogate standards may be added to the instrument blank for additional quality control. The instrument blank is evaluated in the same way as the method blank.

11.1.5. ***Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD) are also known as Laboratory Fortified Blank and Laboratory Fortified Blank Duplicate*** – Analyze an LCS for each batch of samples. In the unlikely event that an MS/MSD pair cannot be performed, an LCS/LCSD pair must be performed. The LCS and LCSD are prepared in the same manner. Both the LCS and LCSD contain all or a majority of the analytes of interest, and must contain the same analytes as the matrix spike(s). The LCS/LCSD is of a different source than the ICAL. If any analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur. The initial corrective action will be to check all calculations. If the calculations are correct, the ensuing corrective action will be the re-preparation and reanalysis of the batch.

The LCS does require confirmation; therefore, each analyte is required to pass on both columns for the LCS and the CCV. Any analyte reported for the LCS must be bracketed by passing CCVs on that column.

Refer to individual test appendices for LCS compounds and surrogate recovery criteria for the LCS.

Some clients require additional analytes for spiking in the LCS. The added compounds must be statistically evaluated to determine if the in-house recovery limits are achievable and realistic.

11.1.6. ***Matrix Spike (MS) and Matrix Spike Duplicate (MSD) are also known as the Laboratory Fortified Matrix and Laboratory Fortified Matrix Duplicate*** – For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and concentrations are the same as the LCS/LCSD. Compare the percent recovery and relative percent difference (RPD) to those in the laboratory specific, historically generated limits.

If any individual recovery falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check all calculations. If the calculations are correct, check the recovery of that analyte in the laboratory control sample (LCS). If the recovery of the analyte in the LCS is within limits, then matrix interference has been demonstrated and the laboratory operation may proceed.

If the recovery for any component is outside QC limits for the matrix spike/spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action may include re-preparation and reanalysis of the batch. MS/MSD failures are flagged in the report.

The matrix spike/spike duplicate must be analyzed at the same dilution as the parent sample (the un-spiked sample).

11.1.7. ***Surrogates*** – Every sample, blank, and QC sample is spiked with at least one surrogate compound. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits. The compounds routinely included in the surrogate spiking solutions, along with recommended standard concentrations, are listed in the appendices for each analysis.

If any surrogate is outside the acceptance limits, an NCM must be written and the following corrective actions must take place (except for dilutions > 5x).

Check all calculations for error.

Ensure that instrument performance is acceptable. If the system is demonstrated to be out of control, all steps taken to return the system to control must be fully documented as part of the corrective action.

Recalculate the data and/or reanalyze the extract if either of the above checks reveals a problem.

If the above corrective actions have taken place and the result is a surrogate that is outside of the limits, re-extract and reanalyze the sample. If the re-extract is outside of limits, flag the data as “Estimated Concentration” due to demonstrated matrix effect.

It is only necessary to re-extract/reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

11.1.8. ***LOQ Verification*** - A verification of the LOQ is performed at least annually, and whenever significant changes are made to the preparation and/or analytical procedure.

The verification is performed by the extraction and analysis of reagent water spiked at 0.5-2 times the established LOQ.

The LOQ check is carried through the same preparation and analytical procedures as environmental samples and other QC samples.

The LOQ verification is performed after the initial calibration and should be performed on every instrument where data is reported annually.

Recovery of the target analytes in the LOQ verification should be within ± 20 % of the LCS criteria, until the laboratory has enough data to establish acceptance limits for the LOQ. This practice acknowledges the potential for greater uncertainty at the low end of the calibration curve.

When reporting concentrations below the LOQ, the results are qualified as estimated with a J flag on the analytical report

11.1.9. ***Quality Assurance Summaries*** – Certain clients may require specific project or program QC which may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

11.1.10. ***PAS-SC QC Program*** – Further details of QC and corrective action guidelines are presented in the Quality Assurance Management Plan [QAMP ME0012K]. Refer to this document if the samples are from a state other than South Carolina, or if the samples are for non-regulatory reporting.

11.2 Instrument QC

The following Instrument QC checks are performed. Refer to Appendix B for acceptance criteria and required corrective action.

QC Item	Frequency
Initial Calibration	Every day samples are analyzed
Reporting Limit Check Solution (RLCS)	After calibration
Initial Calibration Blank	After calibration
Initial Calibration Verification	After calibration. 7196A: after every 15 samples
Continuing Calibration Blank	After every 10 samples and at end of analytical day
Continuing Calibration Verification	After every 10 samples and at end of analytical day

11.3 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to the Demonstration of Capability SOP [QA SOP ME001F2] for more information.

Initial and Continuing Demonstrations of Capability (IDOC and CDOC) – To establish the ability of an analyst to generate acceptable accuracy, each analyst must make a satisfactory initial demonstration of capability prior to using any method and at any time there is a change in instrument type or method. Thereafter, a continuing demonstration of capability is required annually. Refer to the *Demonstration of Capability* SOP [QA SOP ME001F2] for additional information.

Each analyst must make an initial demonstration of capability (IDOC) / initial demonstration of proficiency (IDP) for each individual method. Demonstration of capability for both solid and water matrices is required. This requires analysis of QC check samples containing a representative list of analytes for the method.

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 at 2500 µg/mL for #2 diesel fuel standard.

Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these results with the acceptance criteria given in Table V.

An IDOC must be performed for new analysts being trained on the method or when major change in sample preparation occurs, such as change in solvent. The IDOC must document that the new analyst is capable of performing the method or portion of the method, of which the analyst is responsible.

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.

A continuing demonstration of capability (CDOC) is required annually for all analysts performing the method.

12.0 Data Review and Corrective Action

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employee's complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to the Data Review SOP [QA SOP ME003LP] for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in

the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

13.0 Pollution Prevention

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

14.0 Modifications

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 Method Validation and Instrument Verification for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

Modifications from Reference Method – Chapter 1 of SW-846 states that the Method Blank should not contain any analyte of interest at or above the Method Detection Limit. This SOP states that the Method Blank must not contain any analyte of interest at or above ½ the reporting limit. Common lab contaminants are allowed to be up to 5 times the reporting limit in the blank following consultation with the client.

Troubleshooting and Maintenance – Refer to the instrument manufacturer's instructions for troubleshooting and maintenance. Maintenance procedures can also be found in the Equipment and Instrumentation SOP [QA SOP ME002JT].

Requirements for North Carolina Compliance samples:

Method Blank must be < ½ LOQ

On each day North Carolina compliance samples are analyzed, the laboratory must analyze a standard at the lowest reported concentration (LOQ) to verify the instrument can detect analytes at the LOQ. The analytes of interest must be detected to verify quantitation at the LOQ.

15.0 Responsibilities

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 Attachments

16.1 APPENDIX A: HERBICIDES BY METHOD 8151A
16.2 TABLE A-1. Standard Analyte List, Method 8151A
16.3 TABLE A-2. Recommended Instrumental Conditions, Method 8151A
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16.11 TABLE DoD-A. Chlorinated Herbicide Target Analyte List for DoD
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17.0 References

Note: Where references exclude a date or edition, the latest edition of the referenced document adopted/recognized by the laboratory's accreditation bodies applies. Refer to the *Quality Assurance Management Plan* [QAMP ME0012K] for details.

17.1 *Consolidated Quality System Manual (QSM) for Environmental Laboratories*. Department of Defense (DoD) / Department of Energy (DoE).

17.2 *General Requirements for the Competence of Testing and Calibration Laboratories*. International Standard ISO/IEC 17025.

17.3 *Laboratory Accreditation Standards*. TNI Standard. The NELAC Institute.

17.4 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Update V, July 2014, *Method 8000D*.

17.5 EPA Method 8151A, SW846, *Test Method for Evaluating Solid Waste*, Revision 1, December 1996.

18.0 Revision History

Revision #	Revision Date	Section Modified	Modification	Reason for Modification
-08	**09/23/2021**	Title Page	Update QM to Kelly Nance and EHSO to Kristina Bouknight	Personnel change
		All	Update formatting	Pace compliance

APPENDIX A: HERBICIDES BY METHOD 8151A

1. SCOPE AND APPLICATION

1.1. This method is applicable to the gas chromatographic determination of Chlorinated phenoxy acid herbicides, and includes all extracts derived from solid, aqueous, or non-aqueous matrices that have been prepared according to SOP ME001IM. The herbicides listed in Table A-1 are routinely analyzed. Other chlorinated acids may be analyzed by this method if the quality control criteria in Section 9 and the initial demonstration of method performance in Section 13 are met.

2. SUMMARY OF METHOD

2.1. This method presents conditions for the analysis of prepared extracts of phenoxy acid herbicides by gas chromatography. The herbicides, as their methyl esters, are injected onto the column, separated, and detected by electron capture detectors. Quantitation is by the external standard method. Final results are reported as acid.

3. DEFINITIONS

3.1. Refer to the QAMP for definitions of terms used in this document. Definitions may also be found in the main body of this SOP.

4. INTERFERENCES

4.1. Refer to the main body of this SOP for general information regarding chromatographic interferences.

4.2. Chlorinated acids and phenols cause the most direct interference with this method.

4.3. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups for the herbicide extracts are described in the sample preparation SOP.

5. SAFETY

5.1. Refer to Section 5 of the method 8000D section of this SOP for general safety requirements.

6. EQUIPMENTS AND SUPPLIES

6.1. Refer to Section 6 of the main body of this SOP. A 63Ni electron capture detector is required.

6.2. Refer to Table A-2 for analytical columns.

6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Refer to Section 7 of the main body of this SOP for general information on reagents and standards.

7.1.1. Instrument calibration standards are purchased from vendor in acid form. These standards must undergo esterification procedures; refer to prep SOP, ME001IM Section 11.7.

7.1.2. Unopened standards have a manufacturer determined expiration date.

7.1.3. Opened standards have a two-month expiration date from date of being opened to date of esterification.

7.1.4. After esterification, the calibration standards have an expiration date of 6 months.

7.2. Refer to Table A-3 and C-4 for details of calibration and other standards.

8. SAMPLE PREPARATION, PRESERVATION AND STORAGE

8.1. Refer to Section 8 of the method 8000D section of this SOP.

9. QUALITY CONTROL

9.1. Refer to Section 9 of the main body of this SOP for quality control requirements except surrogate information.

9.2. ***Surrogates*** – Every sample, blank, and QC sample (excluding instrument blanks) is spiked with a surrogate compound. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

9.2.1. The current surrogate recovery limits are located in the LIMS. A hard copy is kept in the laboratory, and is available upon request.

9.2.2. *QC*: The surrogate must pass for a single column for the MB and the LCS. If this criterion is not met, all effected samples must be re-prepared and reanalyzed.

9.2.3. *Samples*: The surrogate must pass recovery limits for at least one of the two columns.

9.2.3.1. PAS-SC policy is to report the lower result between the two columns. If the surrogate failed for the reported column, the surrogate from both columns must be reported to show that at least one of the surrogates passed (documenting that the prep procedure was performed within performance limits).

9.2.3.2. If the surrogate fails for both columns, the following corrective actions must take place (except for >5x dilutions):

9.2.3.3. Check all calculations for error.

9.2.3.4. Ensure that instrument performance is acceptable.

9.2.3.5. Recalculate the data and/or reanalyze the extract if either of the above checks reveals a problem.

9.2.4. If the results are confirmed, re-prepare and reanalyze the sample once. Re-preparation is not necessary if there is obvious chromatographic interference. In any case, an NCM must be filed to document the specifics.

9.2.4.1. If the reanalysis shows failing surrogate recoveries, matrix interference has been demonstrated and an NCM must be written so that the data can be reported with a flag.

9.2.4.2. If the reanalysis shows a passing surrogate recovery, the re-prep results are reported.

9.2.5. If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate then matrix effect has been demonstrated for that sample and re-preparation is not necessary. If only one is out of control, then re-preparation or flagging of the data is required. Each scenario requires an NCM.

9.2.6. Refer to the *Quality Assurance Management Plan* [QAMP] for further details of the corrective actions.

9.3. Refer to Table A-5 for minimum performance criteria for the initial demonstration of capability.

9.4. Refer to Table A-5 for the components and levels of the LCS and MS mixes.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to Section 10 of the main body of this SOP for general calibration requirements.

10.2. External standard calibration is used for this method. A five-point calibration curve is run from standards prepared from stock standards which have undergone the esterification process. These standards are called procedural standards. Follow the procedure provided in Section 11.7 of the prep SOP (ME001IM).

10.3. A mid-level standard is used for the continuing calibration. The center(s) of retention time windows for any analytes included in the daily calibration are updated. The widths of the windows will remain the same. See Section 11.6 of the main body of this SOP and method 8000D for details.

10.4. Refer to Table A-2 for details of GC operating conditions.

11. PROCEDURE

11.1. Refer to the main body of this SOP for procedural requirements.

11.2. ***Extraction*** – The extraction procedure is described in SOP ME001IM.

11.3. ***Cleanup*** – The alkaline hydrolysis and subsequent extraction of the basic solution described in the extraction procedure provides an effective cleanup.

11.4. Analytical sequence – The analytical sequence starts with an initial calibration of at least five points, or a daily calibration that meets % difference criteria from an existing initial calibration.

11.4.1. The daily calibration consists of mid-level standards of all analytes of interest. The center of retention time windows must be updated with the daily calibration.

11.4.2. A continuing calibration verification (CCV) is run after every 10 field samples or 12 hours, whichever comes first.

11.5. ***Gas Chromatography*** – Chromatographic conditions are listed in Table A-2.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the main body of this SOP for identification and quantitation of single component analytes.

12.2. All standards must go through the esterification process. These standards are called procedural standards. Follow the procedure provided in Section 11.7 of the prep SOP [ME001IM].

13. METHOD PERFORMANCE

13.1. Multiple laboratory performance data has not been published by the SOP for this method. Table A-5 lists minimum performance standards required by Shealy for the four replicate initial demonstration of capability (required by Section 13.2 of the main body of this SOP) for this method. The spiking level should be equivalent to a mid-level calibration.

14. POLLUTION PREVENTION

14.1. Pollution prevention encompasses any technique used to reduce the volume or toxicity of a waste at the point of generation. Reagents and standards should be purchased and/or prepared in volumes consistent with laboratory use to minimize the production of hazardous waste from unused and/or expired surplus chemicals.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Waste Manager should be contacted if additional information is required.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Update V, July 2014, *Method 8000D*.

16.2. EPA Method 8151A, SW846, *Test Method for Evaluating Solid Waste*, Revision 1, December 1996.

17. MISCELLANEOUS

17.1. ***Modifications from Reference Method*** – Refer to the main body of this SOP for modifications from the reference method.

17.2. Tables DoD-A and B have been added to Appendix C for LCS, matrix spike, and surrogate control limits for all DoD samples.

TABLE A-1. Standard Analyte List, Method 8151A

Compound	CAS Number	Reporting Limit		
		Aqueous (µg/L)	Soil (µg/Kg)	TCLP (mg/L)
2,4,5-T	93-76-5	0.5	10	NA
2,4,5-TP (Silvex)	93-72-1	0.5	10	0.02
2,4-D	94-75-7	2.0	40	0.005
2,4-DB	94-82-6	4.0	80	NA
Dalapon	75-99-0	5.0	100	NA
Dicamba	1918-00-9	1.0	20	NA
Dichloroprop	120-36-5	2.0	40	NA
Dinoseb	88-85-7	2.0	40	NA
MCPA	94-74-6	200	4000	NA
MCPP	93-65-2	200	4000	NA
Pentachlorophenol	87-86-5	1.0	20	NA

- The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Volume	Final Volume	Dilution Factor
Ground Water	1000 mL	10 mL	10
Low-level Soil	50 g	10 mL	10
TCLP	100 mL	10 mL	10
Non-aqueous Waste	1 g	10 mL	10

- Specific reporting limits are highly matrix dependent. The reporting limits listed above are provided for guidance only and may not always be achievable. For special projects, the extracts may be analyzed without any dilution, resulting in reporting limits 10 times lower than those in Table A-1.

TABLE A-2. Recommended Instrumental Conditions, Method 8151A

Parameter	Recommended Conditions
Injection port temperature	200°C
Detector temperature	325°C
Temperature program	50°C hold 2min, 30°C/min to 300°C, hold 0.5min
Column 1	DB-XLB 25m x 0.32mm x 0.25µm Capillary
Column 2	DB-35MS 25m x 0.32mm x 0.25µm Capillary
Injection	1 µL
Carrier gas	Hydrogen
Make up gas	Nitrogen
Instrument configuration	Single injection using a y-split, dual detector, dual column

- Recommended conditions should result in resolution of all analytes listed in Table A-1.

TABLE A-3. Calibration Standard Preparation, Method 8151A
8151A Calibration Stock Standards

Vendor	Catalog #	Analyte(s)	Concentration (µg/mL)	Final Concentration (µg/mL) *
Supelco	21603357	Dalapon	250	25.0
		Dicamba	50.0	5.0
		Dichloroprop	100	10.0
		MCPA	10000	1000
		MCPP	10000	1000
		Pentachlorophenol	10.0	1.0
		2,4-D	100	10.0
		2,4-DB	200	20.0
		2,4-DCPA	100	10.0
		2,4,5-T	25.0	2.5
		2,4,5-TP	25.0	2.5
Accustandard	P-144S	Dinoseb	100	10.0

* 1 mL of each standard is esterified and brought to a volume of 10.0 mL to achieve final concentration. Details of the esterification process can be found in ME001IM.

Initial Calibration Standard Preparation

Calibration Level	µL of Each Esterified Standard	Final Volume (mL) in Hexane
ICAL-1	10.0	10.0
ICAL-2	40.0	10.0
ICAL-3	80.0	10.0
ICAL-4	200	10.0
ICAL-5	1000	10.0

TABLE A-4. Calibration Levels, Free Acids1 (µg/L), Method 8151A

Compound	Level 1	Level 2	Level 3	Level 4	Level 5
2,4,5-T	2.5	10	20	50	250
2,4,5-TP (Silvex)	5	10	20	50	250
2,4-D	10	40	80	200	1000
2,4-DB	20	80	160	400	2000
DCAA (Surrogate)	10	40	80	200	1000
Dalapon	25	100	200	500	2500
Dicamba	5	20	40	100	500
Dichloroprop	10	40	80	200	1000
Dinoseb	10	40	80	20	1000
MCPA	1000	4000	8000	20000	100000
MCPP	1000	4000	8000	20000	100000
Pentachlorophenol	1	4	8	20	100

[1] The reporting limits listed in Table A-1 will be achieved with these calibration levels and a 10- fold dilution of the sample extract. Lower reporting limits can be achieved with lesser dilutions of the sample extract.

TABLE A-5. LCS/Matrix Spike and Surrogate Spike Levels, Method 8151A

Compound	Aqueous (µg/L)	Soil (µg/Kg)	Waste (µg/Kg)	TCLP (mg/L)
2,4,5-T	10	200	10000	N/A
2,4,5-TP (Silvex)	10	200	10000	0.20
2,4-D	10	200	10000	0.20
2,4-DB	10	200	10000	N/A
Dalapon	10	200	10000	N/A
Dicamba	10	200	10000	N/A
Dichloroprop	10	200	10000	N/A
Dinoseb	10	200	10000	N/A
MCPA	1000	20000	1000000	N/A
MCPP	1000	20000	1000000	N/A
Pentachlorophenol	10	N/A	N/A	N/A
DCAA (Surrogate)	50	1000	50000	0.5

TABLE A-6. Aqueous LCS, MS, MSD, Surrogate, and Initial Demonstration of Capability Recovery Limits, Method 8151A

Compound	% Recovery Limits	South Carolina % Recovery Limits	% RSD Limits
2,4,5-T	70-130	70-130	40
2,4,5-TP (Silvex)	70-130	70-130	40
2,4-D	70-130	70-130	40
2,4-DB	70-130	70-130	40
Dalapon	31-126	70-130	40
Dicamba	64-113	70-130	40
Dichloroprop	70-130	70-130	40
Dinoseb	31-128	NA	40
MCPA	70-130	70-130	40
MCPP	60-159	70-130	40
Pentachlorophenol	70-130	70-130	40
DCAA (Surrogate)	50-112	62-117	NA

TABLE A-7. Solid LCS, MS, MSD, Surrogate, and Initial Demonstration of Capability Recovery Limits, Method 8151A

Compound	% Recovery Limits	South Carolina % Recovery Limits	% RSD Limits
2,4,5-T	63-121	70-130	40
2,4,5-TP (Silvex)	58-135	70-130	40
2,4-D	63-119	70-130	40
2,4-DB	39-135	70-130	40
Dalapon	40-96	70-130	40
Dicamba	70-130	70-130	40
Dichloroprop	66-104	70-130	40
Dinoseb	10-100	NA	40
MCPA	70-130	70-130	40
MCPP	70-130	70-130	40
DCAA (Surrogate)	44-114	44-114	NA

APPENDIX – DoD QSM Requirements

Sections found in this appendix supersede and/or supplement the existing sections of the SOP. These requirements must be met when analyzing samples for the Department of Defense as stipulated in the DOD Quality Systems Manual.

Table B-1. Organic Analysis by Gas Chromatography (GC)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Breakdown check (Endrin/DDT Method 8081 only)	Before sample analysis and at the beginning of each 12-hour shift.	Degradation of DDT and Endrin must each be ≤ 15%.	Correct problem, then repeat breakdown checks.	Flagging is not appropriate.	No samples shall be run until degradation of DDT and Endrin is each ≤ 15%.
Initial Calibration (ICAL) for all analytes (including surrogates)	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	ICAL must meet one of the three options below: Option 1: RSD for each analyte ≤ 20%; Option 2: linear least squares regression for each analyte: r2 ≥ 0.99; Option 3: non-linear least squares regression (quadratic) for each analyte: r2 ≥ 0.99.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. Quantitation for multicomponent analytes such as chlordane, toxaphene, and Aroclors must be performed using a 5-point calibration. Results may not be quantitated using a single point. No samples shall be analyzed until ICAL has passed.

Table B-1. Organic Analysis by Gas Chromatography (GC)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Calculated for each analyte and surrogate.
Retention Time (RT) window width	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from the 72-hour study or 0.03 minutes, whichever is greater.	NA.	NA.	Calculated for each analyte and surrogate. Only applicable if internal standard calibration is not used.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within established RT windows. All reported analytes within ± 20% of true value.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

Table B-1. Organic Analysis by Gas Chromatography (GC)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence with the exception of CCVs for Pesticide multi- component analytes (i.e., Toxaphene, Chlordane and Aroclors other than 1016 and 1260), which are only required before sample analysis.	All reported analytes and surrogates within established RT windows. All reported analytes and surrogates within ± 20% of true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without valid CCVs. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-1. Organic Analysis by Gas Chromatography (GC)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal Standards (IS)	If employed, every field sample, standard, and QC sample.	Retention time within ± 0.06 RRT UNITS from retention time of the midpoint standard in the ICAL; Internal standard signal (area or height) within -50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV can be used.	Inspect GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the Case Narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	NA.
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid Method Blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-1. Organic Analysis by Gas Chromatography (GC)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project- specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error).

Table B-1. Organic Analysis by Gas Chromatography (GC)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. RPD ≤ 30% (between MS and MSD or sample and MD).	Examine the project- specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.

Table B-1. Organic Analysis by Gas Chromatography (GC)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project if available; otherwise use DoD/DOE QSM Appendix C limits or in- house LCS limits if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch if sufficient sample material is available. If obvious chromatographic interference is present, reanalysis may not be necessary, but the client must be notified prior to reporting data, and the failures must be discussed in the Case Narrative.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the Case Narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Confirmation of positive	All results > the DL must be confirmed	Calibration and QC criteria for second	NA.	Apply J-flag if RPD > 40%. Discuss in	Use project-specific reporting

Non-linear regression – The COD is presented in the Target data system as R^2. Six points shall be used for the second order. The coefficient of determination (COD) R^2 must be ≥ 0.99. The non-linear fit uses the following functions:

External Standard

$y = ax^2 + bx + c$

Where:
y = Instrument response
x = Concentration
a = Slope
b = Intercept

Coefficient of Determination

$$COD = \frac{\sum_{i-1}^{n}\left(Y_{obs} - \bar{y}\right)^2 - \left(\frac{n-1}{n-p}\right)\sum_{i-1}^{n}\left(Y_{obs} - Y_i\right)^2}{\sum_{i-1}^{n}\left(Y_{obs} - \bar{y}\right)^2}$$

Where:
Y_{obs} = Observed response (area) for each concentration from the initial calibration standard
$\bar{Y}$ = Mean observed response from the initial calibration
Y_i = Calculated (or predicted) response at each concentration from the original calibration(s)
n = Total number of calibration points (i.e., 6 for a quadratic model)
p = Number of adjustable parameters in the polynomial equation (i.e., 2 for a second order polynomial)

TABLE DoD-A. Chlorinated Herbicide Target Analyte List for DoD

Compound	CAS Number	Reporting Limit	
		Aqueous (µg/L)	Soil (µg/Kg)
2,4,5-T	93-76-5	0.5	10
2,4,5-TP (Silvex)	93-72-1	0.5	10
2,4-D	94-75-7	2.0	40
2,4-DB	94-82-6	2.0	40
Dalapon	75-99-0	1.0	20
Dicamba	1918-00-9	1.0	20
Dichloroprop	120-36-5	2.0	40
Dinoseb	88-85-7	0.5	10
MCPA	94-74-6	200	4000
MCPP	93-65-2	200	4000

TABLE DoD-B. LCS/MS Control Limits for Chlorinated Herbicides for DoD

Compound	Aqueous		Solid	
	Upper	Lower	Upper	Lower
2,4,5-T	42	147	31	138
2,4,5-TP (Silvex)	51	134	43	129
2,4-D	45	152	28	144
2,4-DB	35	153	34	142
Dalapon	19	139	40	96
Dicamba	50	141	38	132
Dichloroprop	46	159	28	155
Dinoseb	31	128	10	100
MCPA	35	144	28	135
MCPP	33	157	35	143
Pentachlorophenol	56	139	70	130

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Kelly M. Nance
Quality Manager

2/10/2022 8:38:25 AM
Bradley E. Belding
Operations Manager

2/4/2022 4:12:27 PM
Kristina P. Bouknight
Environmental Health and Radiation Safety Officer

2/7/2022 9:44:51 AM
Maria S. Gonzalez
Supervisor

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Flash Point by Pensky-Marten Closed Cup Tester
METHOD: 1010 A/B
ISSUER: Pace ENV - Local Quality - WCOL

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TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Flash Point by Pensky-Marten Closed Cup Tester
METHOD: 1010 A/B
ISSUER: Pace ENV - Local Quality - WCOL

1.0 Scope and Application

This standard operating procedure (SOP) describes the laboratory procedure for the determination of Flashpoint by Pensky-Marten Closed Cup Tester.

This method is used to determine the flash point of fuel oils, lube oils, suspensions of solids, liquids that tend to form a surface film under test conditions, and other liquids.

No specific test for determining the flash point of solids is required by RCRA regulations. Wastes, solid in matrix, may only be characterized as an ignitable waste by meeting the narrative regulatory definition located in 40 CFR 261.21(a)(2). The result obtained by this method cannot be used to directly classify a solid-matrix waste as ignitable, nor can the results be used to definitively classify a waste as non-hazardous; however, result may provide useful information that can be applied to a generator's knowledge of a waste in determining whether a waste meets the regulatory definition.

2.0 Summary of Method

The test cup is filled to the filling mark with sample. The sample is then heated at a slow constant rate with continual stirring (for solid samples, no stirring is required). An ignition source is directed into the test cup at regular intervals with simultaneous interruption of stirring. The flash point is the lowest sample temperature at which application of the ignition source cause the vapor above the sample to ignite

3.0 Interferences

3.1 Tests are to be performed in a draft-free room or compartment. Tests made in a laboratory hood or in any location where drafts occur are not reliable.

NOTE: Analysis may be performed in a fume hood provided that the hood's blower (or fan) is shut off during the analysis.

3.2 Acetone must be used to thoroughly clean all parts of the test cup and its accessories. Thoroughly dry all parts of the test cup and its accessories to ensure the removal of the acetone used to clean the apparatus.

4.0 Definitions

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

5.0 Health and Safety

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the PAS-SC Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

A fire extinguisher must be placed within reaching distance from where this test is performed. At all times, refrain from bending over the cup when the test is being performed. Unexpected flash can cause serious injury to exposed body parts in close proximity to the cup.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

6.0 Sample Collection, Preservation, Holding Time, and Storage

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

Requirements for container type, preservation, and field quality control (QC) for the common list of test methods offered by PAS-SC are included in the PAS-SC-WCOL Analytical Methods List [Admin Form ME002BS].

General Requirements

Matrix	Routine Container	Minimum Sample Amount[1]	Preservation	Holding Time
Aqueous	250mL glass	50mL	Thermal: ≤6°C Chemical: None	28 days
Solid/Waste	4oz glass	10g	Thermal: ≤6°C Chemical: None	28 days

[1]*Minimum amount needed for each discrete analysis.*

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with the Sample Receiving SOP [Admin SOP ME0013H].

After receipt, samples are stored at 4 ± 2°C until sample analysis.

When possible, samples logged in for multiple analyses that includes flash point testing, flash point should be collected in a separate container or be the first test to be performed on a sample. Do not open containers unnecessarily to prevent loss of volatile material or possible introduction of moisture, or both.

7.0 Equipment and Supplies

7.1 **Pensky-Martens Closed Cup Apparatus (manual)** – Consists of the test cup, test cover and shutter, stirring device, heating source, ignition source device, air bath, and top plate.

7.2 **Thermometer** – ASTM Pensky-Martens Low Range Thermometer, having a range from 20 to 230 °F (ASTM #9F thermometer).

7.3 **Ignition source** – Bottled gas flame.

7.4 **Flame-Ignition Device** – Any flame-ignition device that has a tip with an opening of 0.69 – 0.79 mm (0.027 – 0.031 in.) in diameter is qualified. This tip can be made of stainless steel or other suitable metals.

7.5 **Barometer** – NIST traceable, capable of determining ambient pressure

8.0 Reagents and Standards

8.1 **Reagent water** – PAS-SC employs a series of in-house deionized (DI) tanks to purify the incoming water. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks. See the Deionized Water System SOP [QA SOP ME0012S] for further information.

8.2 **Calibration fluid [p-xylene, flash point = 81°F]** – May be purchased from any approved vendor that can provide a certificate of analysis. Note manufacturer's expiration date.

8.3 **Cleaning solvent [acetone]** – Used to clean testing cup and accessories

9.0 Procedure

9.1 Equipment Preparation

9.1.1. Thermometers are maintained and verified as required by the Equipment and Instrumentation SOP [QA SOP ME002JT].

9.1.2. Instrument Performance Check - The performance of the manual apparatus is verified each day by the ignition of the calibration fluid (p-Xylene). **The flash point obtained must be 81 ± 1°F**. When the flash point obtained is not within that limit, check the condition and operation of the apparatus, especially with regard to tightness of the lid. After any adjustment, repeat the test using a fresh portion of p-Xylene. The p-Xylene must flash within criteria before sample analysis can begin.

9.2 Analysis

9.2.1. Thoroughly clean and dry all parts of the cup and lid before beginning the analysis; care must be taken to ensure cleaning solvent (acetone) is removed.

9.2.2. Fill the test cup with the sample to be tested to the level indicated by the filling mark inside of the test cup. For solid samples, fill the test cup to the mark without packing the sample.

9.2.3. Place the lid on the test cup and place the assembly into the apparatus. Be sure that the locking device is properly engaged.

9.2.4. Insert the thermometer into its holder.

9.2.5. Light the test flame and adjust until the flame is approximately 4 mm in diameter.

9.2.6. Adjust the heating mantle setting to supply heat at such a rate that the temperature, as indicated by the thermometer, increases at the rate specified below:

- Aqueous Samples: 9 – 11°F/min.
- Samples containing suspended solids: 2-3°F/min.

- Highly viscous materials: 2-3°F/min.

9.2.7. Turn the stirring device to the setting indicated below:

- Aqueous Samples: 105 rpm
- Samples containing suspended solids: 250 rpm
- Highly viscous materials: 250 rpm
- Solid samples: No stirring

9.2.8. Document the initial temperature of the sample and the ambient barometric pressure in the Flash point spreadsheet [INM Form ME00192].

9.2.9. Apply the test flame by operating the mechanism on the cover which controls the shutter and test flame burner so that the flame is lowered into the vapor space of the cut in 0.5s, left in its lowered position for 1s, and quickly raised to its high position. Do not stir the sample while applying the test flame. Continue applying the test flame at intervals of 2°F.

9.2.10. Record the observed flash point as the reading on the thermometer at the time the ignition source application causes a distinct flash in the interior of the test cup. Do not confuse the true flash point with the bluish halo that sometimes surrounds the test flame at applications preceding the one that causes the actual flash. If the initial temperature of the sample was not at least 30°F below the observed flashpoint, the sample must be cooled to 30°F - 50°F below the sample's expected flashpoint and the flashpoint must be re-determined.

9.2.10.1. The sample is deemed to have flashed when a flame appears and instantaneously propagates itself over the entire surface of the sample.

9.2.10.2. When the ignition source is a test flame, the application of the test flame may cause a blue halo or an enlarged flame prior to the actual flash point. This is not a flash point and shall be ignored.

9.2.10.3. Any samples that result in flash point of < 140°F will also be analyzed in duplicate.

9.2.11. If there is no flash after reaching 140°F, stop and report the flash point as > 140°F. If the sample flashes during the first application of the ignition source, results must be recorded as ≤ the observed temperature.

9.3 Procedural Variations – For compliance samples, all procedures outlined in this SOP must be followed without exception. In the event that a deviation from this SOP cannot be avoided (i.e., demonstrated and uncorrectable matrix interference, non-compatible matrix, insufficient sample amount due to client or laboratory error) it is imperative that an NCM is completed that clearly documents the anomaly. This anomaly must be communicated to the client such that corrective action activity including sample recollection can be performed. Based upon the type and severity of anomaly, the results may not be appropriate for compliance reporting. Discussions between the client, the appropriate regulatory authority, and PAS-SC will be necessary to resolve certain anomalies. In any case, it is essential that any anomaly be documented on an NCM.

10.0 Data Analysis and Calculations

10.1 Record the ambient barometric pressure at the time of the analysis. If the pressure is greater than or lower than 760 mm Hg, then correct the flash point as follows:

Corrected flash point = F + 0.06 (760 – P)

Where:
F = observed flash point, °F,
P = ambient barometric pressure, mm Hg

Report the corrected flash point to the nearest degree in °F

10.2 Relative Percent Difference of sample duplicates

$$RPD = \frac{|X1 - X2|}{\frac{(X1 + X2)}{2}} \times 100$$

Where:
X1 = the sample flash point temperature (corrected)
X2 = the duplicate flash point temperature (corrected)

11.0 Quality Control and Method Performance

11.1 Quality Control

The following QC samples are prepared and analyzed with each batch of samples.

QC Item	Frequency
Method Blank (MB)	1 per batch of 20
Sample Duplicate	1 per 10 samples

11.1.1. **Initial and Continuing Demonstrations of Capability (IDOC and CDOC)** – To establish the ability of an analyst to generate acceptable accuracy, each analyst must make a satisfactory initial demonstration of capability prior to using any method and at any time there is a change in instrument type or method. Thereafter, a continuing demonstration of capability is required annually. Refer to the Demonstration of Capability SOP [QA SOP ME001F2] for additional information.

11.1.2. **Method Blank (MB)** - One method blank must be performed per batch (no more than 20 samples). The method blank consists of approximately 100 mL of DI water.

11.1.3. **Sample Duplicates** - Sample duplicates are performed at a frequency of 10% and the relative percent difference for the sample and its duplicate must be within ±10%.

NOTE: Any samples that result in flash point of < 140°F will also be analyzed in duplicate.

11.1.4. **Initial Calibration Verification (ICV)** - A second source of p-Xylene is ignited after the MB to verify that the instrument performance check standard is acceptable. The flash point obtained must be 81 ± 1°F.

11.1.4.1. When the flash point obtained is not within that limit, check the condition and operation of the apparatus, especially with regard to tightness of the lid. After any adjustment, repeat the test using a fresh portion of p-Xylene. The p-Xylene must flash within criteria before sample analysis can begin.

11.1.5. **Continuing Calibration Verification (CCV)** - Samples analyzed for the Department of Energy (DOE) must also include an instrument performance check at the end of the analytical batch. This check is performed using the p-Xylene source used for the instrument performance verification. The flash point obtained must be 81 ± 1°F.

11.2 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed training for this version of the SOP in EQLIMS. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to Demonstration of Capability SOP [QA SOP ME001F2] for more information.

The department supervisor has the responsibility to ensure that this procedure is performed by an employee who has been properly trained in its use and has the required experience.

12.0 Data Review and Corrective Action

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employee's complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

12.2 Correction Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Refer to Appendix B for a complete summary of QC, acceptance criteria, and recommended corrective actions for QC associated with this test method.

13.0 Pollution Prevention and Waste Management

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

14.0 Modifications

This method is an SW-86 method-defined parameter and may not be modified when used for RCRA testing.

15.0 Responsibilities

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 Attachments

16.1 Appendix A: DoD/DoE Method Specific Quality Control Requirements

17.0 References

Note: Where references exclude a date or edition, the latest edition of the referenced document adopted/recognized by the laboratory's accreditation bodies applies.

17.1 *Consolidated Quality System Manual (QSM) for Environmental Laboratories*. Department of Defense (DoD) / Department of Energy (DoE).

17.2 *General Requirements for the Competence of Testing and Calibration Laboratories*. International Standard ISO/IEC 17025.

17.3 *Laboratory Accreditation Standards*. TNI Standard. The NELAC Institute.

17.4 SW-846, Test Method for Evaluating Solid Waste, Third Edition – Pensky-Martens Closed-Cup Method for Determining Ignitability, Method 1010A, Revision 1, November 2004.

17.5 SW-846, Test Methods for Flash Point by Pensky-Martens Closed Cup Tester, Method 1010B

17.6 ASTM D 93-80 Method A and Method B– Standard Test Method for Flash Point by Pensky-Martens Closed Cup Tester.

18.0 Revision History

This document supersedes the following document(s):

Revision #	Revision Date	Section Modified	Modification	Reason for Modification
-11	**02/10/2022**	Signature Page	Removed Dan Wright as General Manager	Personnel Update

Appendix A: DoD – DoD/DoE Method Specific Quality Control Requirements

Sections found in this appendix supersede and/or supplement the existing sections of this SOP. In addition to the general method performance criteria, these requirements must be met when analyzing samples for the Department of Defense (DoD) and the Department of Energy (DOE) as stipulated in the DoD/DOE Quality System Manual.

QC Parameter	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comment(s)
Method Blank (MB)	One per preparatory and/or analytical batch	Flashpoint > 140°F	Reprocess affected samples in a subsequent preparation batch	If reanalysis cannot be performed (limited sample volume), data must be qualified	Flagging is only appropriate in cases where the samples cannot be reanalyzed
LoQ Verification (LoQv)	NA	See comments	See comments	NA	Refer to *Method Validation* [QA Policy ME003BF]
LoD Verification (LoDv)	NA	See comments	See comments	NA	Refer to *Method Validation* [QA Policy ME003BF]

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Daniel J. Wright
General Manager 1

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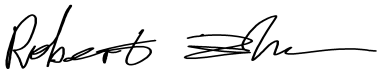
Kelly M. Nance
Quality Manager



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Robert Zhu
Technical Specialist

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Bradley E. Belding
Operations Manager

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Kristina P. Bouknight
Environmental Health and
Radiation Safety Officer

12/3/2021 1:47:36 PM
Fran Stone
Supervisor

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Mercury Analysis by Cold-Vapor Atomic Absorption
METHOD: Methods 245.1 / 7470A and 7471B
ISSUER: Pace ENV - Local Quality - WCOL

TABLE OF CONTENTS

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes the laboratory procedure for the determination of mercury by Cold Vapor Atomic Absorption.

The procedures outlined in this SOP apply to Pace Analytical Services, LLC – West Columbia (PAS-WCOL). It is the responsibility of all employees to ensure they adhere to the procedures herein.

1.1. Target Analyte List and Limits of Quantitation (LOQ)

The target analytes and the normal LOQ that can be achieved with this procedure are provided in the table below:

Type of Sample	Method	Limit of Quantitation
Aqueous	245.1/7470A	0.00020 mg/L
TCLP Extract	7470A	0.00020 mg/L
Solid	7471B	0.083 mg/Kg

LOQ are established in accordance with Pace policy and SOPs for method validation and for the determination of detection limits (DL) and quantitation limits (LOQ). DL and LOQ are routinely verified and updated when needed.

The reporting limit (RL) is the value to which analytes are reported as detected or not detected in the final report. When the RL is less than the lower limit of quantitation (LLOQ), all detects and non-detects at the RL are qualitative. The LLOQ is the lowest point of the calibration curve used for each target analyte.

DL, LOQ, and RL are always adjusted to account for actual amounts used and for dilutions.

Method 245.1/7470A/7471B is a cold-vapor atomic absorption procedure approved for determining the concentration of mercury (organic and inorganic) (CAS # 7439-97-6). Organic mercury compounds will not respond to the cold-vapor atomic absorption technique unless they are first broken down and converted to mercuric ions. Potassium permanganate oxidizes many of these compounds, but a number of organic mercury compounds, including phenyl mercuric acetate and methyl mercuric chloride, are only partially oxidized by this reagent. Therefore, potassium persulfate is added to ensure that organo-mercury compounds, if present, will be oxidized to the mercuric ion before measurement. A heat step is required for methyl mercuric chloride when present in, or spiked to, a natural system.

Method 7470A is used for the analysis of mercury in ground water, aqueous samples, TCLP and other extracts.

Method 245.1 is used for the analysis of mercury in drinking, surface, and saline waters, domestic and industrial waste.

Method 7471B is a cold-vapor atomic absorption procedure approved for determining the

concentration of total mercury (organic and inorganic) in soils, sediments, and sludge type materials.

All samples must be subjected to appropriate digestion steps prior to analysis.

2.0 SUMMARY OF METHOD

All samples are digested manually prior to being analyzed.

This method is a procedure based on the absorption of radiation at 253.7 nm by mercury vapor. The mercury is reduced to the elemental state and is mixed with a reducing agent (usually stannous chloride) to form elemental mercury vapor. Argon or nitrogen gas is introduced to the mixture to carry the mercury vapor. The mercury bearing gas then passes through the drying tube. The drying tube dehumidifies the gaseous mixture. The dry vapor then enters the optical cell. A mercury lamp controlled by the error signal of the reference beam delivers a stable source of emission at 254 nm. Absorbance by the mercury cold-vapor is measured using a solid-state detector with a wide dynamic range.

Refer to the appropriate SOPs, *Digestion of Aqueous Samples and TCLP/SPLP Extracts for Mercury Analysis by Cold-Vapor Atomic Absorption Spectroscopy* [IM SOP ME001II] and *Digestion of Solid and Semisolid Wastes for Mercury Analysis by Cold-Vapor Atomic Absorption* [IM SOP ME001J6], for sample preparation methods.

3.0 INTERFERENCES

3.1. Possible interference from sulfide is eliminated by the addition of potassium permanganate during the digestion process (see *Digestion of Aqueous Samples and TCLP/SPLP Extracts for Mercury Analysis by Cold-Vapor Atomic Absorption Spectroscopy* [IM SOP ME001II]). Concentrations as high as 20 mg/L (or 20 mg/Kg) of sulfide, as sodium sulfide, do not interfere with the recovery of added inorganic mercury in reagent water.

3.2. Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L (or 10 mg/Kg) has no effect on recovery of mercury from spiked samples.

3.3. Seawaters, brines, and industrial effluents high in chlorides may result in falsely high mercury because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253.7 nm. This is evident when the sample does not maintain purple or brown color after the addition of potassium permanganate. This problem can be eliminated by using a reduced initial volume. When this occurs, the initial volume will be noted in the preparation batch. This information is used in the calculation of the final result.

4.0 DEFINITIONS

Note: Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

4.1. **Nonconformance Memo (NCM)**- A form used to document a non-conforming event. An analyst must document a non-conformance memo when a non-conforming event occurs. A non-conforming event may include the reporting of analytical data outside of method or SOP criteria,

or when there is a deviation from a written policy or procedure. Information in an NCM may be used by project managers to flag data in the report narrative, or by the quality department to track trends and initiate corrective actions, where applicable. Additional information on the NCM policy and procedure is located in the Complaints and Nonconformances SOP [QA SOP ME001BO].

5.0 HEALTH AND SAFETY

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.
The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure. Additional health and safety information can be obtained from safety data sheets (SDS) maintained electronically in the public directory.

Analysts must ensure that the proper type of glassware is selected for each application. Discard chipped or broken glassware to prevent injury. All glassware must be discarded into a broken glass container; chipped or broken non-volumetric glassware may be sent off-site for repair as an alternative to disposal.

The preparation of reagents must be conducted in a fume hood or well-ventilated area. Any and all accidents and spills must be reported to a laboratory supervisor and/or the Environmental Health and Safety (EHSO) Officer. Always carry bulk concentrated acid bottles in appropriate impact proof containers. Acid spills must be neutralized immediately, flushed with water and cleaned up using appropriate spill kits.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

Procedures must be carried out in a manner protective of the health and safety of all PAS-WCOL personnel. All work must be stopped in the event of a known or potential compromise to the health and safety of a PAS-WCOL employee. The situation must be reported immediately to the Environmental Health and Safety Officer (EHSO).

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory performs samples collection for samples to be analyzed by this SOP in accordance with the *Field Services SOP* [Field Services SOP ME001BS]. Refer to this SOP for these instructions.

The laboratory will provide containers for the collection of samples upon client request for analytical services. Bottle kits are prepared in accordance with the *Sample Container Shipping SOP* [Admin SOP ME001DS]. For this test method, aqueous sample mays be collected and shipped without acid preservation:

- For method 245.1, acid must be added before analysis to dissolve any metals that adsorb to the container walls. Following acidification at the laboratory and if collected the same day, the sample must be mixed, held for 16 hours (drinking water) or 24 hours (non-potable water), and then verified to be pH < 2 just before analysis or prep (if prep required). If pH is not < 2, add more acid and hold for 16 (drinking water) or 24 (non-potable water) more hours until verified to be pH < 2.

- For method 7470A, if the sample was collected the day of receipt, the 24-hour waiting period is not required after preservation. If the sample was not collected the day of receipt, follow the procedure in 8.2.1.

Requirements for container type, preservation, and field quality control (QC) for the common list of test methods offered by Pace are included in the laboratory's quality manual.

General Requirements

Matrix	Routine Container	Minimum Sample Amount[1]	Preservation	Holding Time
Aqueous	250mL plastic	100mL	Thermal: <6°C Chemical: HNO_3	Collection to Analysis: 28 days
Solid	2oz glass Teflon-lined lid	10g	Thermal: <6°C Chemical: None	Collection to Analysis: 28 days

[1]*Minimum amount needed for each discrete analysis.*

Additional volume is required for MS/MSD and field duplicate, if requested. A full routine container is sufficient volume to analyze field and matrix QC.

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with the *Sample Receiving SOP* [Admin SOPME0013H]. Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples may be stored at room temperature until sample preparation. Prepared samples (extracts, digestates, distillates, other) are stored at room temperature until sample analysis.

7.0 EQUIPMENT AND SUPPLIES

Note: Refer to the Major Operational Equipment List [QA Control Log ME001PM] for specific details regarding the equipment and data processing software utilized during this procedure.

7.1. **Mercury Analysis System** (Leeman Labs, Inc. Model # Hydra II or equivalent)

7.2. **Waste bottle** or other provision for waste analysis materials.

7.3. **Adjustable mechanical pipettes**.

7.4. **Bottle top Dispensers, repeater pipettes**.

7.5. **Class A volumetric flasks**.

7.6. **Class A graduated cylinders**.

7.7. **Balance** – capable of accurately weighing to the nearest 0.0001 g.

8.0 Reagents And Standards

Note: Volumes of standards and reagents may differ from those described in the following sections to account for expected usage. As long as all ratios are kept constant, this is not considered a deviation from the approved procedure.

Note: All stored reagents and standards must be labeled as required by the *Preparation and Documentation of Laboratory Standards and Reagents* SOP [QA SOP ME001HG], the *Comprehensive Chemical Hygiene, Safety, and Hazard Communication Plan* [HS SOP ME0012D], and the *Quality Assurance Management Plan* [QAMP ME0012K].

Note: When preparing diluted acid solutions or standards/reagents that contain acids, always add acid to water. If water is added to acid, a violent reaction may occur.

8.1. **Reagent water** – A series of in-house deionized (DI) tanks is employed to purify the incoming water. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks. See the *Deionized Water System* SOP [QA SOP ME0012S] for further information.

8.2. **Rinse reagent (10% hydrochloric acid)** – Dilute 100 mL of concentrated HCl to 1 L with reagent water.

8.3. **Stannous chloride solution (SnCl2)** – Add 50 g stannous chloride and 25 mL of concentrated HCl and bring to a final volume of 500 mL with DI water.

8.4. **Argon or Nitrogen** (99.995% purity)

9.0 Procedure

Note: All blanks, standards, and samples must be appropriately prepared prior to analysis.

9.1. **Equipment Preparation**

9.1.1. Set up the instrument using the parameters recommended by the manufacturer.

9.1.2. Fill the rinse tray with 10% HCl rinse reagent. Fill the stannous chloride (SnCl2) bottle with 10% SnCl2.

9.1.3. Allow the system to warm up by running reagent blank for approximately 15 minutes.

9.1.4. Start the calibration process:

9.1.4.1. Place the calibration standards and blank in the standard rack.

9.1.4.2. Input all required information into the data system and start the instrument calibration.

9.1.4.3. Calibration Evaluation – Accept or reject the calibration based on the requirements outlined in Section 9.2.3.

9.1.4.4. Calibrate the instrument as described below. The calibration curve is valid for 24 hours from actual calibration time (i.e. the last calibration standard must be analyzed within 24 hours of the end time indicated on the calibration curve prep batch).

9.1.4.5. After the calibration is accepted, input all information for the sample rack into the data system.

9.1.4.6. Set up the sample rack starting in order from right to left. All preparation batch QC samples are included in the sample rack.

9.1.4.7. The ICB, ICV, CCB and CCV are set up on the check standard rack.

9.1.4.8. Refer to the manufacturer's instrument manual for specific directions on autosampler set up.

9.1.4.9. After all samples are analyzed, it is recommended to run reagent blank through all pump tubes for approximately 10 minutes.

9.2. **Initial Calibration**

9.2.1. **Calibration Design** - A 6-point calibration curve consisting of 5 standards and a calibration blank is utilized.

9.2.1.1. For methods 245.1/7470A, the calibration curve consists of 0.2, 0.5, 1.0, 2.0, and 5.0 ug/L standards. The 2.0 ug/L standard is also used as the continuing calibration verification standard.

9.2.1.2. For method 7471B, the calibration curve consists of 1, 5, 10, 15, and 20 µg/L standards. The 10 µg/L standard is also used as the continuing calibration verification standard.

9.2.2. **Calibration Sequence** - Calibration standards are analyzed in sequence from lowest to highest concentration to minimize the chance of carryover from a higher concentration standard.

9.2.3. ICAL Evaluation

9.2.3.1. The calibration curve is obtained by plotting the instrument response against standard concentration values. A calibration curve may be fitted using the calibration standard concentration/response data via the instrument software. This is a linear regression curve and the "r" value (correlation coefficient) must be ≥ 0.995 for the curve to be acceptable.

9.2.3.2. Relative error (%RE) is evaluated minimally at the concentration of the lowest and mid-point standards. Recovery must be ≤50% for the lowest standard and ≤30% for the mid-point standard. If this criterion is not met, re-prepare the standards and recalibrate.

9.2.3.3. The initial calibration verification (ICV) has a mid-level concentration and is analyzed after calibration to verify the calibration curve. This verification must be from a separate source from the calibration standards.

9.2.3.4. Calibration accuracy is monitored throughout the analytical run through the analysis of a known mid-range standard (continuing calibration verification (CCV) at least every 10 samples.

9.3. **Suggested analytical sequence**:

- Instrument Calibration
- ICV
- ICB
- LOQ (LLCCV)
- CCV
- CCB
- MB
- LCS
- 10 samples (or less, batch QC included as samples)
- CCV
- CCB
- 10 samples (or less)
- CCV
- CCB

10.0 Data Analysis And Calculations

Note: Refer to *Quality Assurance Management Plan* [QAMP ME0012K] for further information regarding data reduction.

10.1. Samples results (in µg/L) are taken directly from the computer printout or computer-generated pdf.

10.2. Sample results must be reported in mg/L or mg/Kg with up to two significant figures.

10.3. The ***final concentration determined in digested solid samples when reported on a dry weight basis*** is calculated as follows:

$$\text{mg/Kg, dry weight} = \frac{C \times D \times V}{W \times S}$$

Where:
C = Concentration from instrument readout, in µg/L
D = Instrument dilution factor
V = Final volume after sample preparation, in mL
W = Weight of wet sample digested, in g
S = Percent solids in decimal form

Note: A percent solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. Refer to the appropriate SOP for the determination of percent solid (*Percent Solids and Percent Moisture in Solid and Semi-solids* [Wet Chem SOP ME0013F]). If the results are to be reported on wet weight basis, the "S" factor is omitted from the above equation.

10.4. Sample result in mg/L:

$$\text{mg/L} =$$

10.5. ICV or CCV Percent Recovery:

$$\%\ \text{Recovery} = \quad \times 100$$

Where:
X_1 = observed ICV (or CCV) concentration
X_2 = true ICV (or CCV) concentration

10.6. LCS Percent Recovery:

$$\%\ \text{Recovery} = \quad \times 100$$

Where:
X = observed concentration
t = spike concentration

10.7. MS or MSD Percent Recovery:

$$\%\ \text{Recovery} = \quad \times 100$$

Where:
X = observed concentration of un-spiked sample
X_S = observed concentration of spike sample
t = concentration of added spike

10.8. Relative Percent Difference between MS and MSD or LCS and LCSD:

$$RPD = \quad \times 100$$

Where:

X_1 = the first detected concentration
X_2 = the second detected concentration

10.9 Relative Error (%RE)

$$\%RE = \frac{x_2 - x_1}{x_1} \times 100$$

Where:

X_1 = true value of the calibration standard
X_2 = measured concentration of the calibration standard

11.0 Quality Control And Method Performance

11.1. Quality Control

The following QC samples are prepared and analyzed with each batch of samples.

QC Item	Frequency
Method Blank (MB)	1 per batch of 20 or fewer samples.
Laboratory Control Sample (LCS)	1 per batch of 20 or fewer samples.
Laboratory Control Sample Duplicate (LCSD)	As needed and in absence of MS/MSD
Matrix Spike (MS)	245.1: 1 per 10 samples. 1 additional MS if >10 samples. 7470A and 7471B: 1 per 20 or fewer samples
Matrix Spike Duplicate (MSD)	1 per batch of 20 or fewer samples.
Sample Duplicate	As needed

11.2. Instrument QC

The following Instrument QC checks are performed. Details are listed below the table.

QC Item	Frequency
Initial Calibration	At instrument set up, after significant change in instrument response, prior to every analytical run
Initial Calibration Verification	After each ICAL
Initial Calibration Blank	After ICV

Continuing Calibration Verification	Before sample analysis, after every 10 (or fewer) samples, and at the end of sample analysis
Continuing Calibration Blank	After each CCV

11.2.1. **Initial and Continuing Demonstrations of Capability (IDOC and CDOC)** – Prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to the *Demonstration of Capability* SOP [QA SOP ME001F2] for more information.

11.2.2. **Linear Dynamic Range (LDR)** must be determined initially (i.e., at instrument setup) or whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient standards to ensure that the resulting curve is linear. The verification of linearity must use a minimum of one blank and 3 standards. If any verification data exceeds the initial values by ±10%, linearity must be re-established. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

11.2.3. **Preparation Batch** – A group of up to 20 samples that are of the same matrix and are processed together within 24 hours using the same procedures and reagents. The batch must contain a method blank (MB), a laboratory control sample (LCS) and a matrix spike/matrix duplicate (MS/MSD) pair, or a laboratory control sample duplicate (LCSD) if it is not possible to prepare the MS/MSD. Method 245.1 requires one MS/MSD per 10 samples and an additional MS for batches greater than 10 samples. In some cases, at the request of the client, it may be appropriate to process a matrix spike and a sample duplicate (DUP) in place of the MS/MSD. If the client specifies certain samples for the MS/MSD, the batch may contain multiple MS/MSD pairs. Laboratory generated QC samples (MB, LCS or LCS/LCSD, MS/MSD, DUP) are not counted towards the maximum 20 samples in a prep batch. Field QC samples are included in the prep batch count.

11.2.4. **Method Blank (MB)** – One method blank must be processed with each batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure (digestion and analysis). The method blank is used to identify any system and process interferences or contamination that may lead to the reporting of elevated analyte concentrations or false positive data.

11.2.4.1. For a method blank to be acceptable for use with the accompanying samples, the concentration in the blank must not exceed the LOQ (however, see the appendices for state or program specific requirements). If the MB concentration exceeds the LOQ, re-analyze the method blank. If still unacceptable, re-preparation and re-analysis of all samples associated with a contaminated method blank is required unless the following conditions apply:

11.2.4.2. If the concentration of the MB exceeds the LOQ, but the sample results are

non-detect (ND), then results may be reported with the completion of an NCM. If results are estimated ("J" flagged), then results may be reported with the completion of an NCM if the project manager deems this to be acceptable. If the concentration of the MB is less than 10% of the regulatory limit or less than 10% of the sample concentration, then sample results may be reported with the completion of an NCM.

11.2.4.3. If the client requests that results be reported with estimated ("J") values and there is a "J" value for mercury in the MB, then all sample analyses with a positive result for that analyte must be flagged. Samples must be reprepared and reanalyzed if conditions in the preceeding sections are not met.

11.2.4.4. If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. Such action must result in the completion of an NCM. Refer to Section 11.16 Procedural Variations, for more detail.

11.2.5. **LOQ Check (LLCCV)** – A standard with a concentration at the LOQ (0.20 µg/L for 245.1 and 7470A; 1.0 ug/L for 7471B)) is analyzed after the calibration. The recommended acceptance criteria is ±50% (or refer to the appendices for state or program specific acceptance criteria). If the standard does not fall within the specified criteria, the analytical run is evaluated to determine if the associated samples can be reported. If the LOQ Check fails, it may be reanalyzed prior to sample analysis. If it fails again, recalibration may be necessary.

11.2.5.1. If the instance where the LOQ Check recovery is greater than the upper control limit and the sample results are ND, then results may be reported with the completion of an NCM. If sample results are estimated ("J" flagged), then results may be reported with the completion of an NCM if the project manager deems this to be acceptable.

11.2.6. **Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD)** - *One aqueous LCS must be processed with each preparation batch. The LCS must contain* a known concentration of mercury and be carried through the entire analytical procedure (digestion and analysis). The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing within acceptable accuracy and precision guidelines. An LCSD must be prepared if insufficient sample is available to prepare an MS/MSD. The acceptable control limits for the LCS are as follows: 85 – 115% recovery of the true value (i.e. spiked concentration) recovery for method 245.1; 80-120% recovery of the true value for methods 7470A and 7471B. If the LCSD is prepared and analyzed, the RPD must be ≤ 20%. See appendices for state or program specific requirements.

11.2.6.1. LCS spiking levels (true values) are 2 µg/L for aqueous/TCLP extract and 10 µg/L for solid extracts. Solid results will be converted to mg/Kg as the final step before reporting.

11.2.6.2. If the LCS does not meet the acceptance criteria, it may be re-analyzed once. If the LCS results are still unacceptable, re-prep and re-analysis of all samples associated with the LCS is required unless the following condition

applies:

11.2.6.3. In the instance where the LCS recovery is greater than the upper control limit and the sample results are ND, then results may be reported with the completion of an NCM. If sample results are estimated ("J" flagged), then results may be reported with the completion of an NCM is the PM deems this to be acceptable.

11.2.7. **Matrix Spike/Matrix Spike Duplicate (MS/MSD)** – One MS/MSD pair must be processed with each batch of samples for methods 7470A and 7471B. Method 245.1 requires one MS/MSD per 10 samples and an additional MS for batches containing greater than 10 samples. The MS is a field sample to which a known concentration of analyte has been added. An MSD is a second aliquot of the same sample prepared and analyzed along with the sample and MS. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. The MS/MSD must be analyzed at the same dilution as the parent sample (the un-spiked sample) so that a direct relationship and matrix effect can be evaluated. **Samples identified as field blanks cannot be used for MS/MSD analysis.** The MS/MSD acceptance criteria are as follows: 70-130% recovery of the true value for method 245.1 with an RPD of 20% or less; 80-120% recovery of the true value for 7470A and 7471B with an RPD of 20% or less. See appendices for state or program specific requirements.

11.2.7.1. If the percent recovery or RPD falls outside the acceptable range, check all calculations. If the calculations are correct, check the recovery of the analyte in the LCS. If the recovery of the analyte in the LCS is within limits, then matrix interference has been demonstrated and the laboratory operation may proceed. MS/MSD recoveries that fall outside of the acceptance criteria are flagged in the report.

11.2.7.2. MS and/or MS/MSD samples that have sample analyte concentrations greater than 4 times the spiked analyte amount are not required to meet the percent recovery criteria.

11.2.7.3. Matrix spike levels are 2 µg/L for aqueous/TCLP extract and 10 µg/L for solid extracts. Solid results are converted from µg/L to mg/Kg as the final step before reporting.

11.2.8. **Initial Calibration Verification (ICV/QCS)** – The ICV is analyzed after the calibration to verify the calibration curve. The concentration of the ICV is mid-level of the calibration curve. The true value concentration is 2 µg/L for methods 7470A/245.1 and 10 µg/L for method 7471B. The ICV standard must be prepared using a stock standard that is a separate source from the calibration standards. The ICV is digested for methods 7470A and 7471B but is not digested for method 245.1 (refer to the mercury preparation SOPs ME001J6 and ME001II).

11.2.8.1. The ICV acceptance criteria is ±10% of the true value. If the ICV fails this criteria, it may be immediately re-analyzed one time. If the re-analyzed ICV does not meet acceptance criteria, the cause must be determined and the

instrument recalibrated. See appendices for state or program specific requirements.

11.2.8.2. In the instance where the ICV recovery is greater than the upper control limit and the sample results are ND, then results may be reported with the completion of an NCM. If sample results are estimated ("J" flagged), then results may be reported with the completion of an NCM if the project manager deems this to be acceptable.

11.2.9. **Initial Calibration Blank (ICB)** – One ICB must be analyzed immediately after the ICV to check the system cleanliness. The ICB consists of reagent grade DI water and is prepared as per the mercury preparation SOPs (refer to ME001J6 and ME001II). The ICB must be < LOQ (refer to appendices for state or program specific requirements). If the ICB is outside acceptance criteria, then the analysis must be terminated, the problem corrected, and the instrument recalibrated (which may include reprep of the curve and associated run QC) unless the following situation applies.

11.2.9.1. In the instance where the ICB concentration is greater than the LOQ, and the sample results are ND, then results may be reported with the completion of an NCM. If sample results are estimated ("J" flagged), then results may be reported with the completion of an NCM if the project manager deems this to be acceptable.

11.2.10. **Continuing Calibration Verification (CCV)** – Calibration accuracy is monitored throughout the analytical run via the analysis of a CCV. The CCV is a mid-range standard that is analyzed at least every 10 samples. The CCV true value concentration is 2 µg/L for methods 7470A/245.1 and 10 µg/L for method 7471B. The CCV is digested for methods 7470A and 7471B but is not digested for method 245.1. See appendices for state or program specific requirements.

11.2.10.1. Method 245.1: The CCV immediately following calibration must be within ±5% of the true value. Subsequent CCVs must fall within ±10% of the true value.

11.2.10.2. Methods 7470A and 7471B: All CCVs must fall within ±20% of the true value.

11.2.10.3. In the instance where the CCV recovery is greater than the upper control limit and the samples areND, then results may be reported with the completion of an NCM. If sample results are estimated ("J" flagged), then results may be reported with the completion of an NCM if the project manager deems this to be acceptable.

11.2.11. **Continuing Calibration Blank (CCB)** – A CCB is analyzed immediately following each CCV in order to check the system cleanliness. The CCV consists of reagent grade DI water and is prepared as per mercury preparation SOPs. The CCB concentration must be <LOQ (refer to appendices for state or program specific requirements) . If the CCB falls outside acceptance criteria, then the analysis must be terminated, the problem corrected, and the instrument recalibrated unless the following condition applies:

11.2.11.1. In the instance where the CCB concentration is greater than the LOQ, and the sample results are ND, then results may be reported with the completion of an NCM. If sample results are estimated ("J" flagged), then results may be reported with the completion of an NCM if the project manager deems this to be acceptable.

11.3. **Method Performance**

11.3.1. **Method Validation** - Prior to institution of any method for which data will be used for compliance reporting, the method must be validated; routine quality control procedures are utilized to monitor the validity of the method. Refer to the Quality Assurance Management Plan [QAMP; ME0012K] for information regarding method performance and data quality objectives.

11.3.1.1. **Method Detection Limit** – Detection limits (DL) and limits of quantitation (LOQ) are established at initial method setup and verified on an on-going basis thereafter. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 Method Validation and Instrument Verification and to the *Method Validation Policy* [QA Policy ME003BF] for these procedures.

11.4. **Analyst Qualifications and Training** - Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. The group leader has the responsibility to ensure that this procedure is performed by an employee who has been properly trained in its use and has the required experience.

12.0 Data Review And Corrective Action

12.1. **Data Review**

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employees complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to Data Review SOP [QA SOP ME003LP] for specific instructions and requirements for

each step of the data review process.

12.2. **Corrective Action**

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

13.0 Pollution Prevention And Waste Management

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual. Pollution prevention encompasses any technique used to reduce the volume or toxicity of a waste at the point of generation. Reagents and standards should be purchased and/or prepared in volumes consistent with laboratory use to minimize the production of hazardous waste from unused and/or expired surplus chemicals.

14.0 Modifications

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 Method Validation and Instrument Verification for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

For compliance samples, all procedures outlined in this SOP must be followed without exception. In the event that a deviation from this SOP cannot be avoided (i.e., demonstrated and uncorrectable matrix interference, non-compatible matrix, insufficient sample amount due to client or laboratory error) it is

imperative that an NCM is completed that clearly documents the anomaly. This anomaly must be communicated to the client such that corrective action activity including sample recollection can be performed. Based upon the type and severity of the anomaly, the results may not be appropriate for compliance reporting. Discussions between the client, the appropriate regulatory authority, and the laboratory will be necessary to resolve certain anomalies. In any case, it is essential that any anomaly be documented on an NCM.

15.0 RESPONSIBILITIES

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 ATTACHMENTS

16.1 Appendix A – North Carolina Requirements

16.2 Appendix B – DoD QSM Requirements

17.0 REFERENCES

Note: Where references exclude a date or edition, the latest edition of the referenced document adopted/recognized by the laboratory's accreditation bodies applies. Refer to the *Quality Assurance Management Plan* [QAMP ME0012K] for details.

17.1. *Consolidated Quality System Manual (QSM) for Environmental Laboratories*. Department of Defense (DoD) / Department of Energy (DoE).

17.2. *General Requirements for the Competence of Testing and Calibration Laboratories.* International Standard ISO/IEC 17025.

17.3. *Laboratory Accreditation Standards*. TNI Standard. The NELAC Institute.

17.4. *40 CFR Part 136, Appendix B, Determination of Method Detection Limits*.

17.5. SW-846, Test Method for Evaluating Solid Waste, Third Edition – *Mercury in Liquid Waste by Cold-vapor Atomic Absorption Procedure, Method 7470A*, Revision 1, September 1994.

17.6. SW-846, Test Method for Evaluating Solid Waste– *Mercury in Solid or Semisolid Waste by Cold-vapor Atomic Absorption Procedure, Method 7471A/B*, Revision 2, February 2007.

17.7. EPA/600/4-79-020 – *Mercury, Cold Vapor Technique, Manual, Method 245.1*, Revision 3.0 1994

18.0 REVISION HISTORY

Revision #	Revision Date	Section Modified	Modification	Reason for Modification
-09	12/09/2021	1.1	Updated LOQs	Match LIMS reporting
		3.3	Updated to match prep SOP	Consistency
		9.2.3.2	Added %RE	Pace corporate policy
		11.2.4	Updated method blank criteria and reporting requirements	Clarification

APPENDIX A – North Carolina Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be ≤ 1/2 LOQ	Terminate analysis; Correct the problem; Recalibrate.
CCB	Immediately following each CCV.	The result must be ≤ 1/2 LOQ	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB.
Method Blank	One per sample preparation batch of up to 20 samples.	The result must be ≤ 1/2 LOQ	Re-digest and reanalyze samples.
LOQ Verification	After each calibration.	±50% recovery	Reanalyze; If it fails again, recalibrate.

APPENDIX B – DoD QSM Requirements

Sections found in this appendix replace existing sections and apply solely to analyses for the Department of Defense (DoD) and Department of Energy (DOE). Some sections in this appendix may contain information to be added to existing main sections addressing any additional requirements as stipulated in the DOD Quality Systems Manual as referenced in the QAMP [ME0012K].

Table B-7. Inorganic Analysis by Atomic Absorption Spectrophotometry (AA)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL) for all analytes	Daily ICAL prior to sample analysis.	R2 ≥ 0.99.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	FLAA and GFAA: minimum three standards and a Calibration Blank. CVAA/Mercury: minimum 5 standards and a Calibration Blank. No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 10% of the true value.	Correct problem. Rerun ICV. If that fails, rerun ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

Table B-7. Inorganic Analysis by Atomic Absorption Spectrophotometry (AA)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Low-level Calibration Check Standard (LLCCV)	Daily.	All reported analytes within ± 20% of the true value.	Correct problem and repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed without a valid Low-Level Calibration Check Standard (LLCCV). LLCCV should be less than or equal to the LOQ. If the concentration of the lowest calibration standard is less than or equal to the LOQ, the lowest standard may be re-quantified against the calibration curve as a LLCCV. Otherwise, a separate standard must be analyzed as the LLCCV prior to the analysis of any samples.

Table B-7. Inorganic Analysis by Atomic Absorption Spectrophotometry (AA)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Continuing Calibration Verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All reported analytes within ± 10% of the true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.	Results may not be reported without valid CCVs. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-7. Inorganic Analysis by Atomic Absorption Spectrophotometry (AA)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch.	The absolute values of all analytes must be < ½ LOQ or < 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	Correct problem. If required, re-prep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid Method Blank. Flagging is only appropriate in cases where the samples cannot be re-prepped or reanalyzed. Non-detects associated with positive blank infractions may be reported. Sample results > 10X the LOQ associated with negative blanks may be reported.

Table B-7. Inorganic Analysis by Atomic Absorption Spectrophotometry (AA)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Initial and Continuing Calibration Blank (ICB/CCB)	Immediately after the ICV and immediately after every CCV.	The absolute values of all analytes must be < ½ LOQ or < 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	ICB: Correct problem and repeat ICV/ICB analysis. If that fails, rerun ICAL. All samples following the last acceptable Calibration Blank must be reanalyzed. CCBs may not be re-analyzed without re-analysis of the associated samples and CCV(s).	Flagging is not appropriate.	Results may not be reported without valid Calibration Blanks Non-detects associated with positive blank infractions may be reported. Sample results > 10X the LOQ associated with negative blanks may be reported. For CCB, failures due to carryover may not require an ICAL
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not	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.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-7. Inorganic Analysis by Atomic Absorption Spectrophotometry (AA)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to the source of difference (i.e., matrix effect or analytical error).
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	The data shall be evaluated to determine the source of difference. For Sample/MD: %Recovery and RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.

Table B-7. Inorganic Analysis by Atomic Absorption Spectrophotometry (AA)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Dilution Test (Flame AA and GFAA only)	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within ± 10% of the original measurement.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	Only applicable for samples with concentrations > 50 X LOQ (prior to dilution). Use along with MS/MSD or PDS data to confirm matrix
Post-Digestion Spike (PDS) Addition (Flame AA and GFAA only)	One per preparatory batch if MS or MSD fails.	Recovery within 80-120%.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	Criteria apply for samples with concentrations < 50 X LOQ prior to dilution.
Method of Standard Additions (MSA)	When dilution or post digestion spike fails and if the required by project.	NA.	NA.	NA.	Document use of MSA in the Case Narrative.

Table C-11. Method 7470 - 7471 Series Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
7439-97-6	Mercury	6471	102	7.5	80	124

Table C-12. Method 7470 - 7471 Series Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
7439-97-6	Mercury	10530	100.5	6.3	82	119

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Daniel J. Wright
General Manager 1

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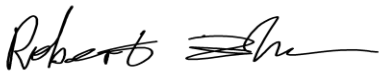
Kelly M. Nance
Quality Manager



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Robert Zhu
Technical Specialist

9/25/2021 8:12:15 PM
Bradley E. Belding
Operations Manager

9/28/2021 8:38:52 AM
James C. Geiger
Supervisor

9/23/2021 1:06:03 PM
Kristina P. Bouknight
Environmental Health and Radiation Safety Officer

Table of Contents

1.0 Scope and Application

This standard operating procedure (SOP) describes the laboratory procedure for the determination of organic analytes by Gas Chromatography (GC).

The procedures are based on SW-846 methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA). Individual analytes and methods are described in the appendices.

Note: Refer to appendices for state and/or program specific method performance criteria, which supersede and/or supplement the general method performance criteria prescribed in this SOP.

1.1 Target Analyte List and Limits of Quantitation (LOQ)

The target analytes and the normal LOQ that can be achieved with this procedure are provided in Tables A-1 and B-1, Appendix A.

LOQ are established in accordance with Pace policy and SOPs for method validation and for the determination of detection limits (DL) and quantitation limits (LOQ). DL and LOQ are routinely verified and updated when needed. The current LOQ for each target analyte that can be determined by this SOP as of the effective date of this SOP is provided in Table 1, Appendix A.

The reporting limit (RL) is the value to which analytes are reported as detected or not detected in the final report. When the RL is less than the lower limit of quantitation (LLOQ), all detects and non-detects at the RL are qualitative. The LLOQ is the lowest point of the calibration curve used for each target analyte.

DL, LOQ, and RL are always adjusted to account for actual amounts used and for dilution.

1.2 Applicable Matrices

This method is applicable to the determination of the concentration of semi volatile analytes in solid, non-aqueous, and aqueous matrices

2.0 Summary of Method

In general, semi-volatile analytes in aqueous samples are prepared for analysis using continuous liquid/liquid extraction [*Continuous Liquid-Liquid Extraction* - EXT SOP ME00155]. Solid samples are prepared using ultrasonic disruption [*Ultrasonic Extraction* - EXT SOP ME00154], microwave extraction [*Microwave Extraction* - EXT SOP ME00156], or soxhlet extraction [*Soxhlet Extraction* - EXT SOP ME001LX].

After the initial preparation step, the sample is introduced to the GC and concentrations of target analytes are measured by the detector response within a defined retention time window, relative to the response of standard concentrations. Internal or external standardization procedures are used as specified in the method appendices.

3.0 Interferences

NOTE: Test/Method specific interferences are addressed in each method specific appendix.

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. If interference is detected, it is necessary to determine if the source of interference is in the preparation and/or cleanup of the samples and then take corrective action to eliminate the problem.

3.2 The use of high purity reagents, solvents, and gases helps to minimize interference problems. Refer to the Solvent Purity Check policy [QA Policy ME001J8] for the procedure for testing the purity of solvents used for extraction.

3.3 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample.

3.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples.

3.5 Co-elution of target analytes with non-targets can occur, resulting in false positives or biased high results.

4.0 Definitions

Refer to the *Laboratory Quality Manual* [QAMP ME0012K] for a glossary of common lab terms and definitions.

4.1 Non-conformance Memo (NCM) - A form used to document a non-conforming event. An analyst must document a non-conformance memo when a non-conforming event occurs. A non-conforming event may include the reporting of analytical data outside of method or SOP criteria, or when there is a deviation from a written policy or procedure. Information in an NCM may be used by project managers to flag data in the report narrative, or by the quality department to track trends and initiate corrective actions, where applicable. Additional information on the NCM policy and procedure is located in the Complaints and Nonconformances SOP [QA SOP ME001BO].

5.0 Health and Safety

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

6.0 Sample Collection, Preservation, Holding Time, and Storage

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory performs samples collection for samples to be analyzed by this SOP in accordance with the *Field Services* SOP [FS SOP ME001BS]. Refer to this SOP for these instructions.

The laboratory will provide containers for the collection of samples upon client request for analytical services. Bottle kits are prepared in accordance with the *Sample Container Shipping* SOP [AD SOP ME001DS].

Requirements for container type, preservation, and field quality control (QC) for the common list of test methods offered by Pace are listed in the *Pace-WCOL Analytical Methods List* [ME002BS].

General Requirements

Matrix	Routine Container	Min. Sample Amount[1]	Preservation	Holding Time
Aqueous	2 x 250 mL amber glass Teflon-lined lid	250 mL	Thermal: <6°C Chemical: None	Collection to Prep: Pest 7 days/ PCB 1 year Prep to Analysis: Pest 40 days/ PCB 1 year
Non-Aqueous	4 oz glass Teflon-lined lid	50 g	Thermal: <6°C Chemical: None	Collection to Prep: Pest 14 days/ PCB 1 year Prep to Analysis: Pest 40 days/ PCB 1 year
Solid	4 oz glass Teflon-lined lid	50 g	Thermal: <6°C Chemical: None	Collection to Prep: Pest 14 days/ PCB 1 year Prep to Analysis: Pest 40 days/ PCB 1 year

[1]Minimum amount needed for each discrete analysis.

PCB wipes – Routine container is gauze + 4mL hexane in a 4 oz glass jar with a Teflon-lined lid.

Additional volume is required for MS/MSD and field duplicate, if requested. For aqueous samples, 2 x 250 mL amber glass Teflon-lined lid are required to analyze field and matrix QC. For solid samples, a full routine container is sufficient volume to analyze field and matrix QC.

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with the *Sample Receiving* SOP [AD SOP ME0013H]. Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are stored at 4 ± 2°C until sample preparation. Prepared samples (extracts, digestates, distillates, other) are stored at 4 ± 2°C until sample analysis.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 28 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

7.0 Equipment and Supplies

NOTE: Method specific equipment and supplies are listed in each method appendix.

NOTE: Refer to the *Major Operational Equipment List* [QA Control Log ME001PM] for specific details regarding the equipment utilized during this procedure.

7.1 Equipment

7.1.1 An analytical system consisting of a gas chromatograph (GC) with dual Electron Capture Detectors (ECD) is required.

8.0 Reagents and Standards

NOTE: Other volumes of standards and reagents may be prepared to account for expected usage. As long as all ratios are kept constant, this is not considered a deviation from the approved procedure.

NOTE: Reagents may be purchased from any approved vendor that can provide a certificate of analysis. All standards must be prepared using certified reference materials. Follow manufacturer expiration date unless stated otherwise below.

NOTE: All stored reagents and standards must be labeled as required by the Preparation and *Documentation of Laboratory Standards and Reagents* SOP [QA SOP ME001HG], the *Contingency and Emergency Preparedness Plan* [HS SOP ME0012D], the *Safety Manual* [Corp Manual COR-MAN-HSE], and the *Laboratory Quality Manual* [QAMP ME0012K].

8.1 Reagents

8.1.1 Reagent water – A series of in-house deionized (DI) tanks is employed to purify the incoming water. Reagent water must be free of the analytes of interest as demonstrated

through the analysis of method blanks. See the *Deionized Water System* SOP [QA SOP ME0012S] for further information.

8.1.2 Gases for carrier and make-up - Hydrogen and Nitrogen.

8.2 Standards

8.2.1 Stock Standards – Stock standards are purchased as certified solutions.

8.2.1.1 Stock standards are stored according to manufacturer recommendations, either 4°C ± 2°C, –10°C to –20°C or ambient temperature, depending on the media the standards were prepared with. All stock standards must be protected from light. Stock standard solutions must be brought to room temperature before using.

8.2.1.2 Purchased standards, if unopened, expire on the date specified by the manufacturer. Opened stock standards and all working standards expire 6 months from the date opened or prepared or on the expiration date of their parent standards, whichever is sooner. All standards' expiration dates are checked prior to use and disposed of if not within that expiration date.

8.2.2 Calibration Standards – Semi-volatile calibration standards are prepared as dilutions of the stock standards. Surrogates standards are used as specified in the method appendices. Prepared semi-volatile calibration solutions must be kept refrigerated at 4 ± 2 °C and protected from light. The standards must be replaced at least every six months or sooner if comparison with check standards indicates a problem. See appendices and tables for calibration standard preparation information.

8.2.3 Quality Control (QC) Standards – QC standards (LCS and matrix spiking standards) are prepared and stored in the same way as calibration standards.

8.2.3.1 The following QC are made from a second source stock independent from the calibration standards: ICV, LCS, and the MS/MSD.

9.0 Procedure

9.1 Equipment Preparation

9.1.1 Support Equipment

9.1.1.1 Incubators, water baths, refrigerator units, freezer units, bottle top dispensers, pipettes, thermometers, and ovens are maintained and verified as required by the *Equipment and Instrumentation* SOP [QA SOP ME002JT].

9.1.1.2 The balance is verified at the beginning of each analytical day using a certified weight set. Refer to *the Equipment and Instrumentation* SOP [QA SOP ME002JT] for balance verification procedures and acceptance criteria.

9.1.2 Instrument

9.1.2.1 Routine Instrument Operating Conditions – Refer to Tables A-2 and B-2, Appendix A for recommended instrument conditions.

9.2 Initial Calibration

9.2.1 Prepare standards containing each analyte of interest at a minimum of five concentration levels. Analyze from low to high concentration. The low-level standard must be at or below the LOQ. The other standards define the working range of the detector. Recommended calibration levels are given in the appendices.

9.2.2 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 new columns, replacing the ECD detector, etc. A new calibration is not required after clipping the column, replacing the septum or syringe, or other minor maintenance. All maintenance must be recorded in the instrument's maintenance log with the date and initials of the analyst performing the maintenance.

9.2.3 It is not acceptable to remove points from a calibration curve for the sole purpose of meeting criteria, unless the points are the highest or lowest of the curve and the reporting limit and/or linear range is adjusted accordingly. In any event, at least five-points must be included in the calibration curve. The following exception applies:

9.2.3.1 A level may be removed from the calibration if the reason can be clearly documented, for example, a broken vial. A minimum of five levels must remain in the calibration. The documentation must be retained with the initial calibration. Alternatively, if the analyst believes that a point on the curve is inaccurate, the point may be reanalyzed and the reanalysis used for the calibration. All initial calibration points must be analyzed without any changes to instrument conditions; all points must be analyzed within 24 hours.

9.2.4 Non-standard analytes are sometimes requested. For these analytes, it may be acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five-point initial calibration. This action can be taken but only with client approval. If the analyte is detected in any of the samples, a five-point initial calibration must be generated and the sample(s) reanalyzed for quantitation.

9.2.5 Aroclor analysis typically includes a five-point calibration for the Aroclor 1016/1260 mix with single points analyzed for pattern identification purposes. If an Aroclor other than 1016 or 1260 is detected in a sample, the extract will be re-analyzed by a single point.

9.2.6 External Standard Calibration – Quantitation by the external standard method assumes a proportional relationship between target compound response and calibration standard response. The ratio of the peak height or area response to the mass or concentration injected is defined as the calibration factor (CF) and may be used to prepare a calibration curve. Calibration standards and samples are introduced into the instrument by the same technique and at the same volume.

9.2.6.1 Some data systems may use the inverse of the formula provided in section 10. This is acceptable so long as the same formula is used for standards and samples. However, if matrix interferences would make quantitation using peak area inaccurate for a particular sample, then peak height may be used as a substitute.

9.2.7 ICAL Evaluation

9.2.7.1 Curve Fit - Average calibration factor (CF) or linear regression, may be used to fit the data.

9.2.7.1.1 Average Calibration Factor - If the %RSD of variation in CFs is <20%, average calibration factor is used. To evaluate linearity of the ICAL, the mean CF, the standard deviation (SD), and the relative standard deviation (RSD) are calculated as denoted in section 10. RSD calculations are performed within Target data system; results are summarized in an ICAL Summary Form (Form 6).

9.2.7.1.2 Linear Regression – If the %RSD of variation in CFs is >20%, linear regression is used. The correlation coefficient (r) for the fit must be ≥ 0.995. The Target data system uses a coefficient of determination (r2) to measure the fit, r2 must be ≥ 0.990. The ICAL cannot be forced through zero. No sample analysis may be performed unless these criteria are met on both columns. Regression formulas are provided in section 10. Calculations are performed within the Target data system; results are summarized in an ICAL Summary Form (Form 6).

9.2.7.1.3 Weighting of Data Points – In linear fits, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason, it is preferable to increase the weighting of the lower concentration points. 1/concentration weighting (often called 1/ X2 weighting) will improve accuracy at the low end of the curve and should be used.

9.2.7.2 Relative Error

9.2.7.2.1 Either calculation of % error or calculation of relative standard error (RSE) may be used to determine calibration function acceptability. Both procedures refit the calibration data back to the calibration model and evaluates the difference between the measured and the true amounts or concentrations used to create the model. Formulas are provided in section 10. Calculations are performed within the Target data system; results are summarized in an ICAL Summary Form (Form 6).

9.2.7.2.1.1. Percent Error - % error between the calculated and expected amounts of an analyte must be ≤ 30% for all standards. For

some data uses, ≤50% may be acceptable for the lowest calibration point.

9.2.7.2.1.2. Relative Standard Error (RSE) - RSE acceptance limit criterion for the calibration model is the same as the RSD limit for average CF (<20%).

9.2.7.3 Initial Calibration Verification (ICV)/Second Source Calibration Verification

9.2.7.3.1 The ICAL is verified by analyzing an ICV. The ICV is a standard obtained from a source independent of the source of standards used for the initial calibration (different manufacturer or different lot). Its concentration must be at or near the middle of the calibration range. The ICV is analyzed immediately after the initial calibration. The value of the second source must agree within 30% of the expected value of the ICAL before sample analysis can begin. If the ICV fails, repeat. If the second ICV fails, maintenance must be performed and two consecutive ICVs must pass or a new ICAL must be analyzed before sample analysis can begin.

9.2.8 Continuing Calibration Verification (CCV)/Instrument Performance Check

9.2.8.1 The ICAL must be verified at the beginning of each analytical sequence, after every 20 injections, and at the end of the sequence. Injections of method blank extracts, matrix spike samples, and other non-standards are counted in the total. Solvent blanks, injected as a check on cross-contamination, need not be counted in the total.

9.2.8.2 Opening CCV – Any CCV that has samples reported after it is considered an opening CCV, even if it is analyzed in the middle of a sequence.

9.2.8.2.1 The opening CCV must pass all analytes of interest within + 20% difference for those compounds using average CF calibration and ± 20% drift for those compounds using a linear fit. If the opening CCV fails, repeat once. If the second analysis fails, instrument maintenance must be performed and two consecutive CCVs must pass or a new ICAL must be performed before sample analysis can occur.

9.2.8.3 Closing CCV – Any CCV analyzed after samples is considered a closing CCV for those samples.

9.2.8.3.1 The closing CCV must pass all analytes of interest within + 20% difference for those compounds using average CF calibration and ± 20% drift for those compounds using a linear fit.

9.2.8.4 A single CCV may be considered both an opening and a closing CCV if the CCV is in the middle of a sequence. At no time can there be more than 12 hours between bracketing CCVs.

9.2.8.5 Samples must be bracketed by CCV standards that meet the above criteria. When confirmation is made on a second column, CCV standards must meet the above criteria on the column from which the reported result is taken.

9.2.8.5.1 If an opening or closing CCV fails high for a specific compound, samples that are ND (or J flag if requested) for that compound are acceptable.

9.2.8.6 Multi-component compound CCVs (toxaphene, chlordane and aroclors) are calculated and evaluated from the average of the quantitation peaks of the compound.

9.2.8.7 The center of each retention time window is updated with each 12-hour CCV. The widths of the windows will remain the same. After any major maintenance (e.g. column change), the RT window must be calculated for each analyte and surrogate.

9.2.8.8 The retention times of all the analytes in the continuing calibration verification standards must be within the retention time windows as determined in the initial calibration

9.3 Sample Preparation

9.3.1 Extraction – Extraction procedures are referenced in the appendices.

9.3.2 Cleanup – Cleanup procedures are referenced in the appendices.

9.4 Analysis

9.4.1 Gas Chromatography – Chromatographic conditions for individual methods are presented in the appendices.

9.4.2 Sample Introduction – In general, semi-volatile analytes are introduced by direct injection of the extract. Samples, standards, and QC must be introduced using the same procedure

9.4.3 Example Analytical Sequence - An analytical sequence starts with an initial calibration or a daily calibration. Refer to the individual method appendices for method specific details of daily calibrations and analytical sequences.

9.4.3.1 The daily calibration includes analyses of standards containing all target analytes and updating the retention time windows.

9.4.3.2 If there is a break in the analytical sequence of greater than 12 hours, a new analytical sequence must be started with a daily calibration.

9.4.4 Retention Time Windows

9.4.4.1 Retention time windows must be determined for all analytes and surrogates. Make an injection of all analytes of interest each day over a 72-hour period. Record the retention time for each single component analyte and surrogate to three decimal

places. Calculate the mean and standard deviation of the three absolute retention times for each analyte and surrogate. For multi-response analytes (e.g., Aroclors), choose the major peaks and calculate the mean and standard deviation of those peaks.

9.4.4.2 If the standard deviation of the retention times for a target compound is 0.000 (i.e., no difference between the absolute retention times), the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes.

9.4.4.3 The width of the retention time window for each analyte, surrogate, and major constituent in multi-component analytes is defined as plus or minus three (± 3) times the standard deviation of the mean absolute retention time established during the 72-hour period. If the default standard deviation in Section 9.4.4.2 is used, the width of the window will be 0.03 minutes.

9.4.4.4 The centers of the windows are updated with the mid-point of the initial calibration or the CCV that starts the analytical sequence. The widths of the windows will remain the same until new windows are generated following the installation of a new column.

9.4.4.5 The laboratory must calculate new retention time windows each time a new column is installed. The new windows must be generated before performing initial calibrations or analyzing samples.

9.4.4.6 Corrective Action for Retention Times – The retention times of all compounds in the continuing calibration that starts the sequence must be within the RT windows established during the initial calibration. If this condition is not met, a new initial calibration must be performed and a new RT window is established. Each subsequent continuing calibration must be within the retention time windows established by the CCV that started the 12-hour analytical sequence. If this condition is not met, all samples analyzed after the last compliant standard must be reanalyzed.

9.4.4.7 Daily Retention Time Windows – The center of the retention time windows is determined as in section 9.4.4.4. (See the method 8082A appendix for exceptions for multi-response components). The retention time windows must be updated by the CCV at the beginning of each analytical sequence, but not for any other calibration verification standards.

9.4.5 Percent Moisture – Analytical results may be reported as dry or wet weight, as required by the client. Percent moisture must be determined if results will be reported as dry weight. Determination procedures are outlined in the *Percent Solids and Percent Moisture* SOP [INM SOP ME0013F].

10.0 Data Analysis and Calculations

10.1 Qualitative Identification

10.1.1 Tentative identification occurs when a peak is found within the retention time window for an analyte, at a concentration above the reporting limit, or above the MDL if "J" flags are required. In some cases, GC/MS confirmation may be required. Client specific requirements may also define the need for second column confirmation and/or GC/MS confirmation. Refer to the appendices for test specific requirements for confirmation. Identification is confirmed if a peak is also present in the retention time window for that analyte on the confirmatory column, at a concentration greater than the reporting limit (MDL if "J" flags required).

10.1.2 Manual Integration

10.1.2.1 Manual changes to automated integration is called manual integration. Manual integration is sometimes necessary to correct inaccurate automated integrations but must never be used to meet QC criteria or to substitute for proper instrument maintenance and/or method set-up. To assure that all manual integrations are performed consistently and are ethically justified, all manual integrations must be performed, reviewed, and recorded in accordance with corporate SOP ENV-SOP-CORQ-0006, Manual Integration.

10.2 Quantitative Identification

10.2.1 Dual Column Quantitation – When sample results are confirmed using dual columns, the agreement between the quantitative results must be evaluated after the identification has been confirmed. Agreement is evaluated by calculating the relative percent difference (RPD) between the two concentrations using the equation below:

$$RPD = \frac{R_1 - R_2}{\frac{1}{2}(R_1 - R_2)} x100$$

Where:

R = Result

10.2.1.1 Large differences in the results may be indicative of positive interference with the higher of the results. If the RPD between the responses on the two columns is > 40%, or if in the opinion of an experienced analyst the complexity of the matrix is resulting in false positives, the confirmation is suspect and the results are qualified with a P flag.

10.2.1.2 For confirmed results, the lower of the two results is normally reported. The higher result is reported if any of the following occurs:

10.2.1.2.1 There is obvious chromatographic interference on the column with the lower result.

10.2.1.2.2 Bracketing calibration verification standards fail on the column with the lower result but are acceptable on the column with the higher result.

Note: If the higher result (reported) results in P flagged data, samples must be evaluated for re-analysis.

10.2.2 Multi-response Analytes – For multi-response analytes, the analyst should use the retention time window, but should rely primarily on pattern recognition. The pattern of peaks will normally serve as confirmation.

10.2.3 The experience of the analyst must weigh heavily in the interpretation of the chromatogram. For example, sample matrix or laboratory temperature fluctuation may result in variation of retention times.

10.3 Calibration Range – If concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions must target the most concentrated analyte in the upper half of the calibration range. It may be necessary to dilute samples due to matrix. In this case, an NCM is required.

10.4 Dilutions – Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hit or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

10.5 Guidance for Dilutions Due to Matrix – If the sample is initially run at a dilution and only minor matrix peaks are found, then the sample must be reanalyzed at a more concentrated dilution. Analyst judgment is required to determine the most concentrated dilution that will not result in instrument contamination.

10.6 Reporting Dilutions – The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

10.7 Interferences – If peak detection is prevented by interferences, further cleanup must be attempted. If no further cleanup is reasonable, then elevation of reporting levels and/or lack of positive identification must be addressed in the case narrative.

10.8 Calculations

See the *Laboratory Quality Assurance Manual* [QAMP ME0012K] for equations for common calculations.

10.8.1 Capabilities of individual data systems may require the use of different formulas than those presented here. When this is the case, the calculations used must be shown to be equivalent and must be documented in an appendix attached to this document.

10.8.2 External Standard Calculation for Aqueous Samples

$$\text{Concentration (µg/L)} = \frac{A_x \times V_t \times DF}{CF \times V_o}$$

Where: Ax = Response of the analyte in the sample
DF = Dilution factor
Vt = Volume of total extract, µL
Vo = Volume of sample extracted or purged, mL
CF = Calibration factor, area or height/µg/mL

10.8.3 External Standard Calculation for Aqueous Samples (Linear Regression)

$$X = \frac{(y - b)V_t DF}{aV_o}$$

Where: y = Instrument response
x = Concentration
a = Slope
b = Intercept

10.8.4 External Standard Calculation for Non-Aqueous and Solid Samples

$$\text{Concentration (mg/kg)} = \frac{A_x \times V_t \times DF}{CF \times W \times D}$$

Where: Ax = Response of the analyte in the sample
Vt = Volume of total extract, µL
DF = Dilution factor
W = Weight of sample extracted in grams
D = Dry weight correction
CF = Calibration factor

10.8.5 External Standard Calculation for Non-Aqueous and Solid Samples (Linear Regression)

$$x = \frac{(y - b)V_t DF}{aWD}$$

Where: y = Instrument response
x = Concentration
a = Slope
b = Intercept

10.8.6 Dry Weight Correction

$$D = \frac{100 - \%moisture}{100}$$

(D = 1 if wet weight is required)

10.8.7 Surrogate Recovery - Concentrations of surrogate compounds are calculated using the same equations as for the target compounds. The response factor from the initial calibration is used. Surrogate recovery is calculated using the following equation:

$$\%\ \text{Recovery} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) spiked}} \text{ x } 100$$

10.8.8 % Difference for External Standard Method

$$\%D = \frac{CF_c - \overline{CF}}{\overline{CF}} \text{ x } 100$$

Where:

CF_c = Calibration factor from the continuing calibration

$\overline{CF}$ = Average calibration factor from the initial calibration

10.8.9 % Drift - % Drift is used for comparing the continuing calibration to a linear curve. The criteria for % drift are the same as for % difference.

$$\%\ \text{Drift} = \frac{\text{Calc. Conc.} - \text{Theoretical Conc.}}{\text{Theoretical Conc.}} \text{ X } 100$$

10.8.10 Calibration Factor (CF)

$$CF = \frac{\text{Area of Peak}}{\text{Conc. of Std}}$$

Where: CF = Calibration Factor

10.8.11 Standard Deviation (SD)

$$SD = \sqrt{\frac{\sum_{i=1}^{n}\left(CF_i - \overline{CF}\right)^2}{n-1}}$$

10.8.12 Mean Calibration Factor (CF)

$$mean\ CF = \overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

Where:

$\overline{CF}$ = Average calibration factor

n = Number of calibration levels

$\sum_{i=1}^{n} CFi$ = Sum of response factors for each calibration level

10.8.13 Relative Standard Deviation (RSD)

$$RSD = \frac{SD}{\overline{CF}}\ x\ 100$$

10.8.14 Linear Regression - The response must increase with increasing concentration. The linear fit uses the following functions.

y = ax + b

or

$$x = \frac{(y - b)}{a}$$

Where:

y = Instrument response a = Slope

x = Concentration b = Intercept

10.8.15 % Error

$$\%\ \text{Error} = \frac{X_i - X'_i}{X_i}\ \text{x}\ 100$$

Where:
x'i = Measured amount of analyte at calibration level i, in mass or concentration units.
xi = True amount of analyte at calibration level i, in mass or concentration units.

10.8.16 Relative Standard Error (RSE) - RSE - The RSE acceptance limit criterion for the calibration model is the same as the RSD limit criterion (Section 9.2.6).

$$RSE = \sqrt{\sum_{i=1}^{n}\left[\frac{x_i' - x_i}{x_i}\right]^2 \Big/ (n-p)}$$

Where:
x'i = Measured amount of analyte at calibration level i, in mass or concentration units.
xi = True amount of analyte at calibration level i, in mass or concentration units.
p = Number of terms in fitting equation (average = 1, linear = 2)
n = Number of calibration points.

11.0 Quality Control and Method Performance

Note: Refer to appendices for state and/or program specific method performance criteria, which superseded and/or supplement the general method performance criteria prescribed in this SOP.

11.1 Quality Control

The following QC samples are prepared and analyzed with each batch of samples.

QC Item	Frequency
Method Blank (MB)	1 per batch
Laboratory Control Sample (LCS)	1 per batch
Laboratory Control Sample Duplicate (LCSD)	As needed
Matrix Spike (MS)	1 per 10 samples
Matrix Spike Duplicate (MSD)	1 per 10 samples

11.2 Instrument QC

The following Instrument QC checks are performed.

QC Item	Frequency
Initial Calibration	Every day samples are analyzed
Initial Calibration Verification	After calibration.
Continuing Calibration Verification	At the beginning of sequence, after every 20 injections and at end of sequence
LOQ Verification	Annually, or whenever significant changes are made to procedure

11.3 Acceptance Criteria and Required Corrective Action

11.3.1 Initial and Continuing Demonstrations of Capability (IDOC and CDOC) – To establish the ability of an analyst to generate acceptable accuracy, each analyst must make a satisfactory initial demonstration of capability prior to using any method and at any time there is a change in instrument type or method. Thereafter, a continuing demonstration of capability is required annually. Refer to the *Demonstration of Capability* SOP [QA SOP ME001F2] for additional information.

11.3.2 Control Limits – In-house historical control limits are determined for surrogates, matrix spikes, and laboratory control samples where allowed by regulatory agencies. These are reviewed regularly and updated as needed; refer to the Trend Analysis of Data Using Control Charts policy [QA Policy ME001IW] for additional detail.

11.3.2.1 Control limits may not be greater than ± 3 standard deviations of the mean LCS recovery.

11.3.2.2 Control limits specified in DoD/DOE QSM Appendix C tables shall be used for batch control unless project specific criteria exist. For analytes not listed in the tables, in-house control limits may be used.

11.3.2.3 All surrogate, LCS, and MS/MSD recoveries must be entered into PAS-SC LIMS. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

11.3.3 Method Blank (MB) / Laboratory Reagent Blank (LRB) – One method blank must be processed with each preparation and/or analytical batch. The method blank consists of a similar matrix to the batch of associated samples in which no target analytes or interferences are present at concentrations that impact the analytical results. The

method blank is to contain all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data.

11.3.3.1 Method blanks are generally considered acceptable if target analyte concentrations are less than ½ the LoQ, or less than the project specific requirements. If the method blank contains an analyte of interest above ½ the LoQ or above project specific requirements, then the method blank and associated samples must be reanalyzed. If the method blank contamination is confirmed, the entire batch must be re-prepared and reanalyzed. Reanalysis or re-extraction may not be required if the samples in the batch are unaffected. Any method blank that does not meet acceptance criteria, is flagged on the data report with a B flag. Where permitted by the program area or client, the following exceptions apply:

11.3.3.1.1 Method blank detection is not present in the samples. If detected, re-prep and reanalyze all samples that do not fall within this criterion.

11.3.3.1.2 The sample concentration is ≥ 10X the blank concentration. If not, re-prep and reanalyze all samples that do not fall within this criterion.

11.3.3.2 To clarify the compounds of interest that are associated with each sample the LIMS generated worksheet printout for each sample will be contained in the batch data file. This worksheet printout will list the required target compounds and the reporting limits.

11.3.3.3 The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, corrective action must be taken. The method blank must be reanalyzed if the analyst feels that the failure was attributed to instrument problems. If the failure is due to a poor extraction, the entire batch must be re-extracted.

11.3.4 Instrument Blank – Instruments must be evaluated for contamination during each 12-hour analytical run. This may be accomplished by analysis of a method blank. If a method blank is not available, an instrument blank must be analyzed. The solvent used for the instrument blank may vary with instrumentation. Surrogate standards may be added to the instrument blank for additional quality control. The instrument blank is evaluated in the same way as the method blank.

11.3.5 Laboratory Control Sample (LCS)/Laboratory Fortified Blank (LFB) – Analyze an LCS for each batch of samples. LCS contains all or a majority of the analytes of interest, and must contain the same analytes as the matrix spike(s). The LCS is of a different source than the ICAL. If any analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur. The initial corrective action

will be to check all calculations. If the calculations are correct, the ensuing corrective action will be the re-preparation and reanalysis of the batch.

11.3.5.1 Refer to individual test appendices for compounds and surrogate recovery criteria for the LCS.

11.3.5.2 When there are a large number of analytes in the LCS or matrix spike, there is a high statistical probability that a few analytes will recover outside of control limits. This may not indicate that the system is out of control, therefore corrective action may not be necessary. The number of allowable marginal exceedances (ME) is based on the number of analytes in the LCS. If more analytes exceed the LCS control limits than is allowed, corrective action is necessary. The number of allowable marginal exceedances is as follows:

Method Applicability	Number of Analytes in LCS	Number Allowed as ME	ME Limits
8081B	11-30	1	ME within 10% of LCS limits

11.3.5.3 Some clients require additional analytes for spiking in the LCS. The added compounds must be statistically evaluated to determine if the in-house recovery limits are achievable and realistic.

11.3.5.4 For DoD all target analytes, including multi-component pesticides (Toxaphene), must be spiked in the LCS, MS, and MSD (with the exception of PCB analysis which is spiked per the method). Target analytes are identified by the client on a project-specific basis. Tech Chlordane is evaluated through the spiking of Alpha and Gamma Chlordane.

11.3.6 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) / Laboratory Fortified Matrix (LFM) and Laboratory Fortified Matrix Duplicate (LFMD) – For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and concentrations are the same as for the LCS. Compare the percent recovery and relative percent difference (RPD) to those in the laboratory specific, historically generated limits.

11.3.6.1 If any individual recovery falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check all calculations. If the calculations are correct, check the recovery of that analyte in the laboratory control sample (LCS). If the recovery of the analyte in the LCS is within limits, then matrix interference has been demonstrated and the laboratory operation may proceed.

11.3.6.2 If the recovery for any component is outside QC limits for the matrix spike/matrix spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will include re-preparation and reanalysis of the batch. An NCM must be generated to document this occurrence.

11.3.6.3 Every effort is made to ensure that an MS/MSD pair is included in every batch. On the rare occasion that one is not possible (sample bottle was broken, etc.) then an LCSD is analyzed.

11.3.6.4 The matrix spike/matrix spike duplicate must be analyzed at the same dilution as the parent sample (the un-spiked sample).

11.3.7 Surrogates – Every sample, blank, and QC sample is spiked with at least one surrogate compound. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits. The compounds routinely included in the surrogate spiking solutions, along with recommended standard concentrations, are listed in the appendices for each analysis.

11.3.7.1 If any surrogate is outside the acceptance limits, an NCM must be written and the following corrective actions must take place (except for dilutions ≥ 5x).

11.3.7.2 Check all calculations for error.

11.3.7.3 Ensure that instrument performance is acceptable. If the system is demonstrated to be out of control, all steps taken to return the system to control must be fully documented as part of the corrective action.

11.3.7.4 Recalculate the data and/or reanalyze the extract if either of the above corrective actions reveals a problem.

11.3.7.5 If the above corrective actions have taken place and the result is a surrogate that is outside of the limits, re-extract and reanalyze the sample. If the re-extract is outside of limits, flag the data as “Estimated Concentration” due to demonstrated matrix effect.

11.3.7.6 It is only necessary to re-extract/reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

11.3.8 LOQ Verification – A verification of the LoQ is performed at least annually, and whenever significant changes are made to the preparation and/or analytical procedure. Refer to the Method Validation policy [QA Policy ME003BF] for additional detail.

11.3.8.1 When reporting concentrations below the LOQ, the results are qualified as estimated with a J flag on the analytical report.

Note: A sample analyzed at or below the LOQ is required on each day samples are analyzed for North Carolina. See appendix D for details.

11.3.8.2 LOQ verification is prepared as per appropriate instructions for a standard at or below the LOQ listed in the appendices.

11.3.9 Nonconformance and Corrective Action – Any deviations from QC procedures must be documented with a nonconformance memo (NCM), with applicable cause and corrective action(s) approved by the QA Officer.

11.3.10 Quality Assurance Summaries – Certain clients may require specific project or program QC that may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

11.4 Method Performance

11.4.1 Method Validation

11.4.1.1 Detection Limits

Detection limits (DL) and limits of quantitation (LOQ) are established at initial method setup and verified on an on-going basis thereafter. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 Method Validation and Instrument Verification and to the *Method Validation SOP* [QA Policy ME003BF] for these procedures.

11.5 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to the *Demonstration of Capability* SOP [QA SOP ME001F2] for more information.

12.0 Data Review and Corrective Action

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employee's complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to the *Data Review* SOP [QA SOP ME003LP] for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

13.0 Pollution Prevention

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

14.0 Modifications

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 Method Validation and Instrument Verification for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

For compliance samples, all procedures outlined in this SOP must be followed without exception. In the event that a deviation from this SOP cannot be avoided (i.e., demonstrated and uncorrectable matrix interference, non-compatible matrix, insufficient sample amount due to client or laboratory error) it is imperative that an NCM is completed that clearly documents the anomaly. This anomaly must be provided to the client such that corrective action activity including sample recollection can be performed. Based upon the type and severity of anomaly, the results may not be appropriate for compliance reporting. Discussions between the client and the appropriate regulatory authority will be necessary to resolve certain anomalies.

14.1 Chapter 1 of SW-846 states that the Method Blank should not contain any analyte of interest at or above the Method Detection Limit. This SOP states that the Method Blank must not contain any analyte of interest at or above the reporting limit.

15.0 Responsibilities

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 Attachments

16.1 Appendix A: Pesticides by Method 8081B

16.2 Appendix B: PCBs by Method 8082A

16.3 Appendix C: Modifications for Method 608.3

16.4 Appendix D: North Carolina Requirements

16.5 Appendix E: DoD/DOE Method Specific QC Requirements

17.0 References

Note: Where reference exclude a date or edition, the latest edition of the referenced document adopted/recognized by the laboratory's accreditation bodies applies. Refer to the *Laboratory Quality Manual* [QAMP ME0012K] for details.

17.1 *Consolidated Quality System Manual (QSM) for Environmental Laboratories*. Department of Defense (DoD) / Department of Energy (DoE).

17.2 *General Requirements for the Competence of Testing and Calibration Laboratories*. International Standard ISO/IEC 17025.

17.3 *Laboratory Accreditation Standards*. TNI Standard. The NELAC Institute.

17.4 Test Methods for evaluation of solid waste, Physical/Chemical Methods, SW-846 Update V, *Determinative Chromatographic Separations,* Revision 4, July 2014; Method 8000D.

17.5 Method 608.3 - *Organochlorine Pesticides and PCBs by GC/HSD*, Environmental Protection Agency, December 2014.

18.0 Revision History

Revision #	Revision Date	Section Modified	Modification	Reason for Modification
-10	**09/28/2021**	Signature Page	Updated QM to Kelly Nance and added Kristina Bouknight as EHSO	Personnel update
		All	Re-wrote and re-formatted entire document	Compliance with Pace policy
		Table C-2	Added chlordane	SC DHEC audit finding #6

Appendix A: Pesticides by Method 8081B

A1. SCOPE AND APPLICATION

A1.1. This appendix describes procedures to be used when SW-846 Method 8081B is applied to the analysis of organochlorine pesticides by GC/ECD. This appendix may also be applied when the discontinued SW-846 Method 8080A is requested, and is applicable to extracts derived from any matrices that are prepared according to the appropriate Shealy sample extraction SOPs.

A1.2. Table A-1 lists compounds that are routinely determined by this method and gives the Reporting Limits (RL) for each matrix. The RLs given are based on the low-level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

A2. SUMMARY OF METHOD

A2.1. This method presents conditions for the analysis of prepared extracts of organochlorine pesticides. The pesticides are injected onto the column, separated, and detected by electron capture detection. Quantitation may be achieved by external standard methods.

A3. DEFINITION

A3.1. Refer to the QAMP for definitions of terms used in this document. Definitions may also be found in the main body of this SOP.

A4. INTERFERENCES

A4.1. Refer to the main body of this SOP for information regarding chromatographic interferences.

A4.2. Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.

A4.3. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts, including Sulfuric Acid (3665A), Florisil cleanup (Method 3620), Gel Permeation Chromatography (Method 3640), and Sulfur cleanup (Method 3660). These cleanup procedures are included in the *Semi-Volatiles Clean-up Procedures* SOP [EXT SOP ME001DV]. Normally, the Sulfur and Florisil cleanup procedures are used.

A5. SAFETY

A5.1. Refer to Section 5 of the main body of this SOP for general safety requirements.

A5.2. Samples may contain aroclors. Aroclors have been classified as a potential carcinogen under OSHA. Concentrated solutions of aroclors must be handled with extreme care to avoid excess exposure. Contaminated gloves and clothing must be removed immediately. Contaminated skin surfaces must be washed thoroughly.

A6. EQUIPMENT AND SUPPLIES

A6.1. Refer to Section 7 of the main body of this SOP. Two ^{63}Ni electron capture detectors are required.

A6.2. Refer to Table A-2 for analytical columns.

A6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

A7. REAGENTS AND STANDARDS

A7.1. Refer to the main body of this SOP for general requirements for reagents and supplies.

A7.2. Refer to Table A-3 for details about the calibration standards.

A7.3. ***Surrogate Standards*** – Tetrachloro-m-xylene and decachlorobiphenyl are the surrogate standards. Refer to Tables A-5 and A-6 for details about surrogate standards.

A7.4. ***Column Degradation Evaluation Mix*** – A mid-level standard containing 4,4'-DDT and Endrin and not containing any of their breakdown products must be prepared for evaluation of degradation of these compounds by the GC column and injection port. This mix must be replaced after 6 months, or whenever corrective action involving columns fails to eliminate the breakdown of the compounds, whichever is shorter. Refer to Table A-4 for details of the column degradation evaluation mix.

A8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

A8.1. Refer to Section 6 of the main body of this SOP.

A9. QUALITY CONTROL

A9.1. Refer to Section 11 of the main body of this SOP for additional QC criteria other than surrogate information.

A9.2. ***Surrogates*** – Every sample, blank, and QC sample is spiked with two surrogate compounds. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits. See Table A-8 for limits.

A9.2.1. Current surrogate recovery limits are found in the LIMS.

A9.2.2. QC - Both surrogates must pass on a single column for the MB and the LCS. If these criteria are not met, all affected samples must be re-prepared and reanalyzed.

A9.2.3. *Samples* - Samples must pass both surrogates on the reporting column; however, due to matrix interferences if one surrogate is outside of criteria, the data must be flagged and qualified appropriately. LIMs flags surrogate failures in the client report.

A9.2.3.1. PAS-SC policy is to report the lower result between the two columns. If both surrogates failed for the reported column, the surrogates from both columns must be reported to show that at least one of the surrogates passed (documenting that the prep procedure was performed within performance limits).

A9.2.3.2. If the surrogates fail for both columns, the following corrective actions must take place (except for ≥5x dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze the extract if either of the above checks reveals a problem.

A9.2.3.3. If the results are confirmed, re-prepare and reanalyze the sample once. Re-preparation is not necessary if there is obvious chromatographic interference. In any case, an NCM must be filed to document the specifics.

A9.2.3.4. If the reanalysis shows failing surrogate recoveries, matrix interference has been demonstrated and an NCM must be written so that the data can be reported with a flag.

A9.2.3.5. If the reanalysis shows a passing surrogate recovery, the re-prep results are reported.

A9.2.4. If the surrogates are out of control for the sample in the matrix spike and the matrix spike duplicate, then matrix affect has been demonstrated for that sample and re-preparation is not necessary. If only one is out of control, then re-preparation or flagging of the data is required. Each scenario requires an NCM.

A9.3. Refer to Tables A-5 and A-6 for spiking concentrations.

A10. CALIBRATION AND STANDARDIZATION

A10.1. Refer to Section 9 of the main body of this SOP for general calibration requirements.

A10.2. Refer to Table A-2 for details on GC operation conditions. The conditions listed should result in resolution of all analytes listed in Table A-1 on both columns.

A10.3. ***Column degradation evaluation*** – A column evaluation mix must be injected before each initial or daily calibration and every 12hrs. The degradation of DDT and Endrin must be calculated (see equations in Section A12.6 below) and each shown to be <15% before calibration can proceed. If the breakdown of DDT and/or Endrin exceeds the limits given above, corrective action must be taken. This action may include:

- Replacement of the injection port liner or the glass wool.
- Cutting off a portion of the injection end of the capillary column.
- Replacing the GC column.

A10.4. ***Initial Calibration*** – Refer to Section 9 of the main body of this SOP for details on calibration procedures.

A10.4.1. Refer to Table A-7 for the initial calibration analytical sequence.

A10.4.2. The response for each single-peak analyte will be calculated by the procedures described in the general method 8000D for GC analysis.

A10.4.3. The surrogate calibration curve is calculated from the individual pesticides mix. Surrogates in the other calibration standards are used only as retention time markers.

A10.4.4. For multi-component pesticides – Single point or multi-point calibration is used for multi-component pesticides (typically toxaphene and technical chlordane). Two options are possible; the same quantitation option must be used for standards and samples. Refer to Section A12.3 below for guidance on which option to use. For any DoD samples with a detection for Toxaphene or Chlordane, a valid multi-point calibration must be generated and the samples re-analyzed against that calibration.

A10.4.5. For multi-component analytes, the mid-level standard must be analyzed as part of the initial calibration. This single point calibration is used to quantitate multi-component analytes.

A10.4.6. The analyst may include a full five-point calibration for any of the multi-component analytes with the initial calibration.

A10.5. Continuing Calibration Verification (CCV) – The pesticides calibration mix is analyzed as the continuing calibration standard. This is analyzed at the beginning of each analytical sequence, after every 20 injections, and at the end of the sequence. The continuing calibration verification standard need not include multi-component analytes.

A10.5.1. A mid-level calibration standard is used for the continuing calibration verification.

A10.5.2. The centers of the retention time windows are updated with the mid-point of the initial calibration or the CCV that starts the analytical sequence.

A11. PROCEDURE

A11.1. Refer to the main body of this SOP for general procedural requirements.

A11.2. ***Extraction*** – The extraction procedures are described in the *Continuous Liquid-Liquid Extraction* [EXT SOP ME00155]; *Waste Dilution Method* [EXT SOP ME00150]; *Ultrasonic Extraction* [EXT SOP ME00154]; or *Microwave Extraction* [EXT SOP ME00156] SOP.

A11.3. The cleanup procedures are described in the *Semi-Volatile Clean-up Procedures* SOP [EXT SOP ME001DV].

A11.4. The suggested gas chromatographic conditions are given in Table A-2.

A11.5. Allow the extracts to warm to ambient temperature before injection.

A11.6. The suggested analytical sequence is given in Table A-7.

A12. DATA ANALYSIS AND CALCULATIONS

A12.1. Refer to the main body of this SOP for identification and quantitation of single component analytes.

A12.2. ***Identification of Multi-Component Analytes*** – Retention time windows are also used for identification of multi-component analytes, but the “fingerprint” produced by major peaks of those compounds in the standard is used in tandem with the retention times to identify the compounds. The ratios of the areas of the major peaks are also taken into consideration. Identification of these compounds may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst’s judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

A12.3. ***Quantitation of Multi-Component Analytes*** – Use 5 major peaks for quantitation as described in Section 9.2, initial calibration of multi-component analytes.

A12.3.1. *Multiple peak option* – This option is particularly valuable if toxaphene is identified but interferences make quantitation based on total area difficult. Select 5 major peaks in the analyte pattern. Find the response of each of the 5 peaks per multi-peak pesticide, and use these responses independently, averaging the resultant concentrations found in samples for a final concentration result. When using this option, it is appropriate to remove peaks that appear to be co-eluting with contaminant peaks from the quantitation. (i.e.: peaks which are significantly larger than would be expected from the rest of the pattern.)

A12.4. Chlordane may be quantitated using the multiple peak option, or by quantitation of the major components, alpha-chlordane, gamma-chlordane and heptachlor.

A12.5. ***Second column confirmation*** - Multi-component analytes must be confirmed on a dissimilar second column.

A12.6. Surrogate recovery results are calculated and reported for decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCMX). If it is determined that sample interference has adversely affected the quantitation of DCB, then TCMX is considered in lieu of DCB, and may be used in samples in which DCB recovery is low. If only one surrogate meets acceptance criteria, the data must be qualified with a flag. Additional corrective action is only necessary if DCB and TCMX are both outside of acceptance limits.

A12.7. ***Calculation of Column Degradation / % Breakdown (%B)***

$$\text{DDT \% B} = \frac{A_{DDD} + A_{DDE}}{A_{DDD} + A_{DDE} + A_{DDT}} \times 100$$

Where: A_{DDD}, A_{DDE}, and A_{DDT} = the response of the peaks for 4,4'- DDD, 4,4'-DDE, and 4,4'-DDT in the column degradation evaluation mix.

$$\text{Endrin \% B} = \frac{A_{EK} + A_{AE}}{A_{EK} + A_{AE} + A_{E}} \times 100$$

Where: A_{EK}, A_{EA}, and A_{E} = the response of endrin ketone, endrin aldehyde, and endrin in the column degradation evaluation mix.

A13. METHOD PERFORMANCE

A13.1. Performance limits for the four replicate initial demonstration of capability samples required under Section 11 of the main body of this SOP are presented in Table A-8. The spiking level should be equivalent to a mid-level calibration.

A14. POLLUTION PREVENTION

A14.1. Refer to the main body of this SOP.

A15. WASTE MANAGEMENT

A15.1. Refer to the main body of this SOP.

A16. REFERENCES

A16.1. Test Methods for Evaluation of Solid Waste, Physical/Chemical Methods, SW-846 Update V, *Determinative Chromatographic Separations, Revision 4, July 2014 Method 8000D*.

A16.2. Method 608.3 - *Organochlorine Pesticides and PCBs by GC/HSD*, Environmental Protection Agency, December 2014.

A17. MISCELLANEOUS

Refer to Section 14 of the main body of this SOP.

TABLE A-1. Standard Analyte List and Reporting Limits, Method 8081B

Compound	CAS #	Reporting Limit	
		Water (ug/L)	Soil (ug/Kg)
alpha-BHC	319-84-6	0.04	1
beta-BHC	319-85-7	0.04	1
delta-BHC	319-86-8	0.04	1
gamma-BHC (Lindane)	58-89-9	0.04	1
cis-Chlordane	5103-71-9	0.04	1
trans-Chlordane	5103-74-2	0.04	1
4,4'-DDD	72-54-8	0.04	1
4,4'-DDE	72-55-9	0.04	1
4,4'-DDT	50-29-3	0.04	1
Aldrin	309-00-2	0.04	1
Chlordane (Technical)	57-74-9	0.4	2
Dieldrin	60-57-1	0.04	1
Endosulfan I	959-98-8	0.04	1
Endosulfan II	33213-65-9	0.04	1
Endosulfan Sulfate	1031-07-8	0.04	1
Endrin	72-20-8	0.04	1
Endrin Aldehyde	7421-93-4	0.04	1
Endrin Ketone	53494-70-50	0.04	1
Heptachlor	76-44-8	0.04	1
Heptachlor Epoxide	1024-57-3	0.04	1
Methoxychlor	72-43-5	0.16	4
Toxaphene	8001-35-2	0.4	10
APPENDIX IX ADD ONs			
Kepone*	143-50-0	0.4	17
Mirex	2385-85-5	0.04	1
Permithirin (cis + trans)	52645-53-1	0.04	1

*Kepone is sometimes requested for analysis by method 8081B; however, kepone may produce peaks with broad tails that elute later than the standard by up to a minute (presumably due to hemi-acetal formation). As a result, kepone analysis by 8081B is unreliable and not recommended. Analysis by method 8270E is a possible alternative.

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Volume	Final Volume
Ground water	250 mL	10 mL
Low-level soil	10 g	10 mL
Non-aqueous waste	1 g	10 mL
TCLP	100 mL	5mL

TABLE A-2. Recommended Instrumental Conditions, Method 8081B

Parameter	Recommended Conditions
Injection port temperature	200°C
Detector temperature	325°C
Temperature program	120°C for 1 min, 9°C/min to 300°C, 1 min hold
Column 1	DB-35MS 30 m x 0.32 mm id, 0.25 µm
Column 2	DB-XLB 30 m x 0.32 mm id, 0.25 µm
Injection	1.0 µL or 5.0 µL for Large Volume Injection (LVI)
Carrier gas	Hydrogen
Make up gas	Nitrogen
Instrument configuration	Single injection split with y-splitter, dual detector, dual column

TABLE A-3. Calibration Levels (ng/mL), Method 8081B

Compound	Level 1	Level 2	Level 3	Level 4	Level 5
Individual Mix (Accustandard M-8081-SC)					
alpha-BHC	5.0	10	20	50	100
beta-BHC	5.0	10	20	50	100
delta-BHC	5.0	10	20	50	100
gamma-BHC (Lindane)	5.0	10	20	50	100
cis-Chlordane [1]	5.0	10	20	50	100
trans-Chlordane [1]	5.0	10	20	50	100
4,4'-DDD	5.0	10	20	50	100
4,4'-DDE	5.0	10	20	50	100
4,4'-DDT	5.0	10	20	50	100
Aldrin	5.0	10	20	50	100
Dieldrin	5.0	10	20	50	100
Endosulfan I	5.0	10	20	50	100
Endosulfan II	5.0	10	20	50	100
Endosulfan Sulfate	5.0	10	20	50	100
Endrin	5.0	10	20	50	100
Endrin Aldehyde	5.0	10	20	50	100
Endrin Ketone	5.0	10	20	50	100
Heptachlor	5.0	10	20	50	100
Methoxychlor	20	40	80	200	400
Multi-Component Standards					
Chlordane (Technical) (Restek 32021)	10	50	100	200	500
Toxaphene (Accustandard P-093S-H-10X)	50	100	250	500	1000
Surrogates (Accustandard CLP-032-R)					
Decachlorobiphenyl	5	10	20	50	100
Tetrachloro-m-xylene	5	10	20	50	100
[1] Compounds may be used in lieu of running a daily technical Chlordane standard for samples that are non-detect for technical Chlordane.					

TABLE A-4. Column Degradation Evaluation Mix, Method 8081B

Vendor	Catalogue #	Components	Conc. µg/mL	Aliquot Amount (µL)	Final Volume (mL) hexane	Conc. ng/mL
Accustandard	M-8081-DS	4,4'DDT	200	100	100	200
		Endrin	200	100		200

TABLE A-5. LCS/Matrix Spike and Surrogate Spike Levels, Method 8081B

Component	Aqueous (µg/L)	Solid (µg/kg)
4,4'-DDD	0.80	20
4,4'-DDE	0.80	20
4,4'-DDT	0.80	20
Aldrin	0.80	20
alpha-BHC	0.80	20
beta-BHC	0.80	20
delta-BHC	0.80	20
gamma-BHC (Lindane)	0.80	20
Dieldrin	0.80	20
cis-Chlordane	0.80	20
trans-Chlordane	0.80	20
Endosulfan I	0.80	20
Endosulfan II	0.80	20
Endosulfan sulfate	0.80	20
Endrin	0.80	20
Endrin ketone	0.80	20
Endrin Aldehyde	0.80	20
Heptachlor	0.80	20
Heptachlor Epoxide	0.80	20
Methoxychlor	0.80	20
Decachlorobiphenyl (Surrogate)	0.80	20
Tetrachloro-m-xylene (Surrogate)	0.80	20

TABLE A-6. LCS/Matrix Spike and Surrogate Spike Levels for TCLP Pesticides

Component	Aqueous (µg/L)
Endrin	800
Heptachlor	800
Heptachlor Epoxide	800
Lindane	800
Methoxychlor	800
Chlordane (Technical)	800
Toxaphene	1600
Decachlorobiphenyl (Surrogate)	25
Tetrachloro-m-xylene (Surrogate)	25

TABLE A-7. Suggested Analytical Sequence for Method 8081B

Initial Calibration

Breakdown mix
Instrument blank (optional)
Individual ICAL Mix 1-5
Individual ICV
Toxaphene ICAL: single point (5 point for DOD)
Toxaphene ICV
Technical Chlordane ICAL: single point (5 point for DOD)
Technical Chlordane ICV
Instrument blank (required if no MB to be analyzed)
Up to 20 injections (10 field samples for DoD) (unless 12 hours come first)
Individual mix
Instrument blank (optional)
Samples
Individual mix
Any other single component analytes
Any multi-component analytes

Note: A five-point curve for any of the multi-component analytes may be included.

12-hour Calibration

At least every 12 hours, counting from the start of the initial calibration, or from the start of the last daily calibration, the retention time windows must be updated using the individual mix, and the breakdown mix must be run before the continuing calibration.

TABLE A-8. LCS and Surrogate Recovery Limits, Method 8081B

Compound	Aqueous	Solid	Non-Aqueous	RSD limits
4,4'-DDD	70-130	70-130	70-130	20
4,4'-DDE	70-130	70-130	70-130	20
4,4'-DDT	70-130	70-130	70-130	20
Aldrin	70-130	70-130	70-130	20
alpha-BHC	70-130	70-130	70-130	20
cis-Chlordane	70-130	70-130	70-130	20
beta-BHC	70-130	70-130	70-130	20
delta-BHC	70-130	70-130	70-130	20
Dieldrin	70-130	70-130	70-130	20
Endosulfan I	70-130	70-130	70-130	20
Endosulfan II	70-130	70-130	70-130	20
Endosulfan Sulfate	70-130	70-130	70-130	20
Endrin	70-130	70-130	70-130	20
Endrin Aldehyde	70-130	70-130	70-130	20
Endrin Ketone	70-130	70-130	70-130	20
gamma-BHC	70-130	70-130	70-130	20
trans-Chlordane	70-130	70-130	70-130	20
Heptachlor	70-130	70-130	70-130	20
Heptachlor Epoxide	70-130	70-130	70-130	20
Toxaphene	70-130	70-130	70-130	20
Methoxychlor	70-130	70-130	70-130	20

8081 Water	Lower Control Limit	Upper Control Limit
Decachlorobiphenyl	10	122
TCMX	46	119
8081 Solid	**Lower Control Limit**	**Upper Control Limit**
Decachlorobiphenyl	57	110
TCMX	37	91
8081 Non-Aqueous	**Lower Control Limit**	**Upper Control Limit**
Decachlorobiphenyl	65	158
TCMX	64	134

TABLE A-9. Pesticide Calibration Stock Standards and Pesticide ICAL Stock Standard Preparation

Vendor	Catalogue #	Analyte(s)	Concentration (µg/mL)	Aliquot Amount (µL)	Final Volume (mL hexane)
AccuStandard	M-8081-SC	Pesticide Mix	1000	50	50
Supelco	861428-U	Mirex	1000	50	
AccuStandard	P-064S-10X	Methoxychlor	1000	150	
Accustandard	CLP-032-R	DCB & TCMX	200	250	

TABLE A-10. Pesticide Calibration Standard Preparation*

Calibration Level	µL Pesticide of ICAL Stock	Final Volume (mL hexane)
ICAL-1	50	10
ICAL-2	100	10
ICAL-3	1000	50
ICAL-4	500	10
ICAL-5	2000	10

*Standards are diluted by a factor of 5 for Large Volume Injection (LVI)

TABLE A-11. Technical Chlordane Calibration Stock Standard and ICAL Stock Standard Preparation

Vendor	Catalogue #	Analyte	Concentration (µg/mL)	Aliquot Amount (µL)	Final Volume (mL hexane)	Final Concentration (µg/mL)
Restek	32021	Technical Chlordane	1000	250	25	10

TABLE A-12. Technical Chlordane Calibration Standard Preparation*

Calibration Level	µL Technical Chlordane Stock	Final Volume (mL hexane)
ICAL-1	10	10
ICAL-2	50	10
ICAL-3	500	50
ICAL-4	200	10
ICAL-5	1000	10

*Standards are diluted by a factor of 5 for Large Volume Injection (LVI)

TABLE A-13. Toxaphene Calibration Stock Standard and ICAL Stock Standard Preparation

Vendor	Catalogue #	Analyte	Concentration (µg/mL)	Aliquot Amount (µL)	Final Volume (mL hexane)	Final Concentration (µg/mL)
AccuStandard	P-093S-H-10X	Toxaphene	1000	500	50	10

TABLE A-14. Toxaphene Calibration Standard Preparation*

Calibration Level	µL Technical Chlordane Stock	Final Volume (mL hexane)
ICAL-1	10	10
ICAL-2	20	10
ICAL-3	250	50
ICAL-4	100	10
ICAL-5	200	10

*Standards are diluted by a factor of 5 for Large Volume Injection (LVI)

APPENDIX B: PCBS BY METHOD 8082A

B1. SCOPE AND APPLICATION

B1.1 This appendix describes procedures to be used when SW-846 Method 8082A is applied to the analysis of polychlorinated biphenyls (PCBs) by GC/ECD. SW-846 Method 8082A is applicable to extracts, derived from any matrix, which are prepared according to the appropriate Shealy sample extraction SOP. The PCBs are determined and quantitated as aroclor mixes.

B1.2 Table B-1 lists compounds that are routinely determined by this method and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low-level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

Note: SW-846 method 8082A provides incomplete guidance for determination of individual PCB congeners. This SOP does not include directions for congener specific analysis.

B2. SUMMARY OF METHOD

B2.1 This method presents conditions for the analysis of prepared extracts of PCBs. The PCBs are injected onto the column, separated, and detected by electron capture detection. The external standard method is used to quantitate the PCBs contained in the extracts.

B3. DEFINITIONS

B3.1 Refer to the main body of this SOP.

B4. INTERFERENCES

B4.1 Refer to the main body of this SOP for information regarding chromatographic interferences.

B4.2 Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.

B4.3 Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts, including Sulfuric Acid (3665A), Florisil cleanup (Method 3620), Gel Permeation Chromatography (Method 3640), and Sulfur cleanup (Method 3660). These cleanup procedures are included in the *Semi-Volatiles Clean-up Procedures* SOP [EXT SOP ME001DV]. Normally, the sulfuric acid cleanup procedure is used for extracts containing aroclors.

B5. SAFETY

B5.1 Refer to the main body of this SOP for general safety requirements.

B5.2 Aroclors have been classified as a potential carcinogen under OSHA. Concentrated solutions of aroclors must be handled with extreme care to avoid excess exposure. Contaminated gloves and clothing must be removed immediately. Contaminated skin surfaces must be washed thoroughly.

B6. EQUIPMENTS AND SUPPLIES

B6.1 Refer to Section 7 of the main body of this SOP. Two ^{63}Ni electron capture detectors are required.

B6.2 Refer to Table B-2 for analytical columns.

B6.3 Microsyringes, various sizes, for standards preparation, sample injection and extract dilution.

B7. REAGENTS AND STANDARDS

B7.1 Refer to the main body of this SOP for general requirements for reagents and supplies. All standards for this method must be replaced.

B7.2 Refer to Table B-3 for details about the calibration standards.

B7.3 Surrogate Standards – Tetrachloro-m-xylene and decachlorobiphenyl are the surrogate standards. Other surrogates may be used at the client's request. Refer to Table B-3 for details about the surrogate standards.

B8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

B8.1 Refer to Section 6 of the main body of this SOP.

B9. QUALITY CONTROL

B9.1 Refer to the main body of this SOP.

B9.2 Surrogates – Every sample, blank, and QC sample is spiked with two surrogate compounds. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits in Table B-5. Current surrogate recovery limits are also located in LIMs.

B9.2.1 QC - Both surrogates must pass for a single column for the MB and the LCS. If these criteria are not met, all affected samples must be re-prepared and reanalyzed.

B9.2.2 Samples - Samples must pass both surrogates on the reporting column; however, due to matrix interference, if one surrogate is outside of criteria, the data must be flagged and qualified appropriately. LIMs flags surrogate failures on the client report.

B9.2.2.1 Shealy policy is to report the lower result between the two columns. If both surrogates failed for the reported column, the surrogates from both columns must be reported to show that at least one of the surrogates passed (documenting that the prep procedure was performed within performance limits).

B9.2.2.2 If the surrogates fail for both columns, the following corrective actions must take place (except for ≥5x dilutions):

- Check all calculations for errors.
- Ensure that the instrument performance was acceptable.
- Recalculate the data and/or reanalyze the extract if either of the above checks reveals a problem.

B9.2.2.3 If the results are confirmed, re-prepare and reanalyze the sample once. Re-preparation is not necessary if there is obvious chromatographic interference. In any case, an NCM must be filed to document the specifics.

B9.2.2.4 If the reanalysis shows failing surrogate recoveries, matrix interference has been demonstrated and an NCM must be written so that the data can be reported with a flag.

B9.2.2.5 If the reanalysis shows a passing surrogate recovery, the re-prep results are reported.

B9.2.2.6 If the surrogates are out of control for the sample, matrix spike, and a matrix spike duplicate, then matrix affect has been demonstrated for that sample and re-preparation is not necessary. If only one is out of control, then re-preparation or flagging of the data is required. Each scenario requires an NCM.

B9.2.3 Refer to Table B-4 for spiking concentrations.

B10. CALIBRATION AND STANDARDIZATION

B10.1 Refer to the main body of this SOP for general calibration requirements.

B10.2 ***Initial Calibration***

B10.2.1 Refer to Table B-5 for the initial calibration analytical sequence.

B10.2.2 The response for each aroclor will be calculated by the procedures described in the general method 8000D for GC analysis, with the following modifications.

B10.2.3 A five-point calibration of the Aroclor 1016/1260 mix is generated with at least mid-level single points for the other five aroclor mixes analyzed initially for identification purposes. If another aroclor is detected in the sample, the extract is re-analyzed with a single point, an ICV, and a closing for the single point. It is recommended that a valid five-point calibration will be run for any aroclor that is detected, however this is required for the DoD.

B10.2.4 The surrogate calibration curve is calculated from the PCB mix containing 1016/1260. Surrogates in the other calibration standards are used only as retention time markers.

B10.2.5 ***Quantitating Aroclors***- The same quantitation technique must be used for standards and samples. The same selection of peaks must be consistent throughout the analytical sequence. The only exception is described below.

B10.2.5.1 Select 3 to 5 major peaks in the analyte pattern. Find the response of each of the 3 to 5 peaks per aroclor, and use these responses independently, averaging the resultant concentrations found in samples for a final concentration result. It is appropriate to remove peaks that appear to be co-eluting with contaminant peaks from the quantitation (i.e., peaks that are significantly larger than would be expected from the rest of the pattern).

B10.2.5.2 To assess possible interference caused by DDT, DDD, and DDE eluting at the same retention time as major Aroclor peaks used for quantitation, a standard containing the DDT analogs is analyzed with the ICAL. In the case of co-elution, a different Aroclor peak is chosen.

B10.3 ***Continuing Calibration Verification (CCV)*** – The Aroclor 1016/1260 calibration mix is analyzed as the CCV. This is analyzed at the beginning of each analytical sequence, after every 20 injections, (10 field samples for DoD) and at the end of the sequence.

B10.3.1 A mid-level standard is used for the calibration verification.

B10.3.2 At a minimum, the calibration verification includes analysis of the Aroclor 1016/1260 mix.

B10.3.2.1 It is adequate to verify calibration with a mixture of Aroclors 1016 and 1260. If a specific aroclor is expected, it must be included in the daily calibration check.

B10.3.3 The centers of retention time windows for target analytes are updated with the mid-point of the initial calibration or the CCV that starts the analytical

sequence. The centers of retention time windows must be updated every 12 hours. The widths of the windows will remain the same until new windows are generated following the installation of a new column.

B10.3.4 Refer to Section 9 of the main body of this SOP for CCV criteria.

B11. PROCEDURE

B11.1 Refer to the main body of this SOP for general procedural requirements.

B11.2 ***Extraction*** – The extraction procedures are described in the *Continuous Liquid-Liquid Extraction* [EXT SOP ME00155]; *Waste Dilution Method* [EXT SOP ME00150]; *Ultrasonic Extraction* [EXT SOP ME00154]; or *Microwave Extraction* [EXT SOP ME00156] SOP.

B11.3 ***Cleanup*** –The method blank and LCS must be cleaned up with the sample. If the sample requires both 8081B and 8082A analysis, only a portion of the extract is acid cleaned. The acid cleaned extract can only be analyzed for PCBs.

B11.4 The suggested gas chromatographic conditions are given in Table B-2.

B11.5 Allow the extracts to warm to ambient temperature before injection.

B11.6 The suggested analytical sequence is given in Table B-6.

B12. DATA ANALYSIS AND CALCULATIONS

B12.1 ***Identification of Aroclors*** – Retention time windows are used for identification of aroclors, but the “fingerprint” produced by major peaks of those analytes in the standard is used in tandem with the retention times for identification. The ratios of the areas of the major peaks are also taken into consideration. Identification may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst’s judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

B12.2 A clearly identifiable aroclor pattern serves as confirmation of single column GC analysis. However, if the pattern is not clear, or if no historical data for the site is available, then second column confirmation must be performed.

B12.3 ***Quantitation of Aroclors*** – Use 5 major peaks for quantitation.

B12.3.1 If the analyst believes that a combination of Aroclor 1254 and 1260 or a combination of 1242, 1248, and 1232 is present, then only the predominant aroclor is quantitated and reported, but the suspicion of multiple aroclors is discussed in the narrative. An NCM is written by the analyst. If well-separated aroclor patterns are present, then both aroclors are quantitated and reported.

B12.4 Surrogate recovery results are calculated and reported for decachlorobiphenyl (DCB) unless it is determined that sample interference has adversely affected the quantitation of

that surrogate. Tetrachloro-m-xylene (TCMX) recovery is reported in lieu of DCB in samples having the interference, and may be used for samples in which DCB recovery is low. Corrective action is only necessary if DCB and TCMX are both outside of acceptance limits; however, the results will be flagged accordingly. Refer to section 11 of the main body of this SOP for corrective action in such an instance.

B13. METHOD PERFORMANCE

B13.1 Performance limits for the four replicate initial demonstration of capability samples are recovery of 70 – 130%. The spiking level should be equivalent to a mid-level calibration.

B13.2 Method detection limits (MDL) are determined for each Aroclor individually and for the surrogates.

B14. POLLUTION PREVENTION

B14.1 Refer to Section 13 of the main body of this SOP.

B15. WASTE MANAGEMENT

B15.1 Waste generated in this procedure must be segregated and disposed according to the *PAS-SC Hazardous and Non-hazardous Laboratory Waste Management Plan* [ME0012A]. The Hazardous Waste Manger should be contacted if additional information is required.

B16. REFERENCES

B16.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, *Polychlorinated Biphenols (PCBs) by Gas Chromatography*, Revision 1, February 2007; Method 8082A.

B16.2 Method 608.3 *Organochlorine Pesticides and PCBs by GC*, Environmental Protection Agency, December 2014.

B16.3 Test Methods for Evaluation of Solid Waste, Physical/Chemical Methods, SW-846 Update V, *Determinative Chromatographic Separations,* Revision 4, July 2014; Method 8000D.

B17. MISCELLANEOUS

B17.1 ***Modifications from Reference Method*** – Method 8082A includes limited direction for congener specific quantitation. This is outside the scope of this SOP.

TABLE B-1. Standard Analyte List and Reporting Limits, Method 8082A

Compound	Reporting Limit		
	Water (µg/L)	Soil (µg/Kg)	Waste (mg/Kg)
Aroclor-1016	0.4	10	1.0
Aroclor-1221	0.4	10	1.0
Aroclor-1232	0.4	10	1.0
Aroclor-1242	0.4	10	1.0
Aroclor-1248	0.4	10	1.0
Aroclor-1254	0.4	10	1.0
Aroclor-1260	0.4	10	1.0
Aroclor 1262*	0.4	10	1.0
Aroclor 1268*	0.4	10	1.0

*Project Specific Analyte

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol	Final Vol
Ground water	250 mL	10 mL
Low-level soil	10 g	10 mL
Non-aqueous waste	1 g	10 mL

TABLE B-2. Recommended Instrumental Conditions, Method 8082A

Parameter	Recommended Conditions
Injection port temperature	200°C
Detector temperature	325°C
Temperature program	120°C for 1 min, 9°C/min to 300°C, 1 min hold
Column 1	DB-35MS 30 m x 0.32 mm id, 0.25 µm
Column 2	DB-XLB 30 m x 0.32 mm id, 0.25 µm
Injection	0.5 uL, 1.0 µL or 5.0 µL for Large Volume Injection (LVI)
Carrier gas	Hydrogen
Make up gas	Nitrogen
Instrument configuration	Single injection with y-split, , dual detector, dual column

TABLE B-3. Aroclor Calibration Standard Preparation

Aroclor Stock Standards

Vendor	Catalog #	Analyte	Concentration (µg/mL)
AccuStandard	C-216S-H-10X	Aroclor 1016	1000
AccuStandard	C-260S-H-10X	Aroclor 1260	1000
AccuStandard	C-221S-H-10X	Aroclor 1221	1000
AccuStandard	C-232S-H-10X	Aroclor 1232	1000
AccuStandard	C-242S-H-10X	Aroclor 1242	1000
AccuStandard	C-248S-H-10X	Aroclor 1248	1000
AccuStandard	C-254S-H-10X	Aroclor 1254	1000
AccuStandard	C-262-H-10X	Aroclor 1262	1000
AccuStandard	C-268S-H-10X	Aroclor 1268	1000
AccuStandard	CLP-032-R	TCMX & DCB	200

Aroclor ICAL Stock Standard Preparation in Hexane*

Analyte	Aliquot Amount (mL)	Final Volume (mL)	Concentration (µg/mL)
Aroclor 1016	100	10	10
Aroclor 1260	100		10
TCMX & DCB(surrogates)	50		1

*****Other ICAL stock combinations with surrogates: Aroclors 1221 & 1254, 1232 & 1262, 1242 & 1268, and 1248

Aroclor Calibration Standard Preparation*

Calibration Level	µL of Aroclor ICAL Stock	Final Volume (mL hexane)
ICAL-1	50	10
ICAL-2	200	10
ICAL-3	2500	50
ICAL-4	1000	10
ICAL-5	2000	10

*Standards are diluted by a factor of 5 for Large Volume Injection (LVI)

Calibration Levels (ng/mL), Method 8082A

Compound	Level 1	Level 2	Level 3	Level 4	Level 5
Aroclor 1016/1260	50	200	500	1000	2000
Aroclor 1221 + 1254[1]	50	200	500	1000	2000
Aroclor 1242 + 1268[1]	50	200	500	1000	2000
Aroclor 1248[1]	50	200	500	1000	2000
Aroclor 1232 + 1262[1]	50	200	500	1000	2000
Decachlorobiphenyl	5	20	50	100	200
Tetrachloro-m-xylene	5	20	50	100	200

[1] Level 3 will be used for single point calibration
*Project Specific Analyte

TABLE B-4. LCS/Matrix Spike and Surrogate Spike Levels, Method 8082A

Compound	Aqueous (µg/L)	Soil (µg/Kg)	Waste (mg/Kg)
Aroclor 1016/1260	4	100	2.5
Aroclor 1221	4	100	5.0
Aroclor 1232	4	100	2.5
Aroclor 1242	4	100	2.5
Aroclor 1248	4	100	2.5
Aroclor 1254	4	100	2.5
Aroclor 1262*	4	100	2.5
Aroclor 1268*	4	100	2.5
Decachlorobiphenyl (Surrogate)	4	100	2.5
Tetrachloro-m-xylene (Surrogate)	4	100	2.5

Note: Each Aroclor must be used within a two-year period to meet NELAC and DOD requirements.
*Project Specific Analyte

TABLE B-5. LCS and Surrogate Recovery Limits, Method 8082A

Compound	Recovery Limits	RSD limits
Aroclor 1016/1260	70-130	20

8082A Water	Lower Control Limit	Upper Control Limit
Decachlorobiphenyl	10	133
TCMX	31	122
8082A Solid	**Lower Control Limit**	**Upper Control Limit**
Decachlorobiphenyl	41	132
TCMX	35	106
8082A Non-Aqueous (Waste Dilution)	**Lower Control Limit**	**Upper Control Limit**
Decachlorobiphenyl	45	131
TCMX	38	121
8082A Wipes	**Lower Control Limit**	**Upper Control Limit**
Decachlorobiphenyl	46	132
TCMX	35	121

TABLE B-6. Suggested Analytical Sequence for Method 8082A

Initial Calibration

Injection #		
1	Instrument blank (optional)	
2	Aroclor 1232/1262	Level 3
3	Aroclor 1242/1268	Level 3
4	Aroclor 1248	Level 3
5	Aroclor 1221/1254	Level 3
6	Aroclor 1016/1260	Level 1
7	Aroclor 1016/1260	Level 2
8	Aroclor 1016/1260	Level 3
9	Aroclor 1016/1260	Level 4
10	Aroclor 1016/1260	Level 5
11	Instrument blank (optional)	
12	ICV (second source)	
13	MB	
14	LCS (second source)	
15-31	Up to 20 injections (10 field samples for DOD)	
32	Aroclor 1016/1260 (CCV)	Mid-level CCV (usually Level 3) etc.

12-hour Calibration

At least every 12 hours, counting from the start of the initial calibration, or from the start of the last daily calibration, the retention time windows must be updated using the Aroclor 1016/1260 mix. Mid-level standards of any other aroclors expected to be present in the samples are also injected.

APPENDIX C: MODIFICATIONS FOR METHOD 608.3

C1. This section describes the different QC requirements for EPA method 608.3.

C2. The equipment and supplies as well as the instrument conditions for 608.3 analyses are the same as those for method 8081B and 8082A.

C3. The stock standards are the same as those used in 8081B. The standards used and compound lists are found in Tables A-3 and A-4.

C4. Method 608.3 is required for demonstration of compliance with NPDES wastewater discharge permits. The standard analyte list and reporting limits are found in Table C-1.

C5. This method can be applied only to aqueous matrices.

C6. Initial calibration requirements:

C6.1 The initial calibration curve for this method requires at least five points and the low point must be ≤ LOQ.

C6.1.1 A minimum of 5 standards containing both Aroclors 1016 and 1260 must be used for analyzing aroclors. Single midpoint standards of all other Aroclors must be used.

C6.1.2 A minimum of 3 standards must be used for analysis of toxaphene.

C6.2 Target compounds must have RSD < 20%.

C6.3 If this requirement cannot be met, a regression curve must be constructed for the non-compliant compounds. Refer to the main body of this SOP for evaluation of linear regression curve.

C7. *Initial calibration verification requirements* - The concentrations must be within 20% difference of the true value.

C8. *Continuing calibration verification requirements* - Calibration must be verified at the beginning and end of each 24-hour shift. All target compounds must have percent difference (%D) of ≤ 25%.

C9. *Matrix Spike and LCS requirements* - A full analyte spike is required for method 608.3. The spiking levels are given in Table C-2. For every batch of 20 samples, one LCS and one MS/MSD pair are required. The acceptance criteria are listed in Table C-2.

C9.1 At least 80% of the analytes tested in the MS/MSD must have in-house QC acceptance criteria that are tighter than those in Table C-2.

C9.2 A minimum of 80% of the analytes tested for in the LCS must have QC acceptance criteria tighter than those in Table C-2.

C10. *Method Blank* - If any analyte of interest is found in the blank at a concentration greater than the MDL, at a concentration greater than one-third the regulatory compliance limit, or at a concentration greater than one-tenth the concentration in a sample in the batch, whichever is greatest, analysis of samples must be halted and samples in the batch must be re-extracted and the extracts reanalyzed.

C11. *GC resolution*—Resolution is acceptable if the valley height between two peaks (as measured from the baseline) is less than 40% of the shorter of the two peaks.

C11.1 DB-608 column —DDT and endrin aldehyde

C11.2 DB-1701 column—alpha and gamma chlordane

TABLE C-1. Standard Analyte List and Reporting Limits, Method 608.3

Compound	CAS #	Reporting Limits (µg/L)
alpha-BHC	319-84-6	0.04
beta-BHC	319-85-7	0.04
delta-BHC	319-86-8	0.04
gamma-BHC (Lindane)	58-89-9	0.04
4,4'-DDD	72-54-8	0.04
4,4'-DDE	72-55-9	0.04
4,4'-DDT	50-29-3	0.04
Aldrin	309-00-2	0.04
Aroclor 1016	12674-11-2	0.4
Aroclor 1221	1104-28-2	0.4
Aroclor 1232	11141-16-5	0.4
Aroclor 1242	53469-21-9	0.4
Aroclor 1248	12672-29-6	0.4
Aroclor 1254	11097-69-1	0.4
Aroclor 1260	11096-82-5	0.4
Chlordane (Technical)	57-74-9	0.4
Dieldrin	60-57-1	0.04
Endosulfan I	959-98-8	0.04
Endosulfan II	33213-65-9	0.04
Endosulfan Sulfate	1031-07-8	0.04
Endrin	72-20-8	0.04
Endrin Aldehyde	7421-93-4	0.04
Heptachlor	76-44-8	0.04
Heptachlor Epoxide	1024-57-3	0.04
Methoxychlor	72-43-5	0.16
Toxaphene	8001-35-2	0.4

Table C-2. Surrogate Spike Level and QC Acceptance Criteria, Method 608.3

Compounds (Restek 32291)	Surrogate Concentration (µg/L)	CCV % Recovery	DOC % SD	LCS & MS/MSD % Recovery	MS/MSD Max % RPD
alpha-BHC	0.8	69-125	28	37-140	36
beta-BHC	0.8	75-125	38	17-147	44
delta-BHC	0.8	75-125	43	19-140	52
gamma-BHC (Lindane)	0.8	75-125	29	32-140	39
4,4'-DDD	0.8	75-125	32	31-141	39
4,4'-DDE	0.8	75-125	30	30-145	35
4,4'-DDT	0.8	75-125	39	25-160	42
Aldrin	0.8	75-125	25	42-140	35
Chlordane	0.8	75-125	24	45-140	35
Dieldrin	0.8	48-125	42	36-146	49
Endosulfan I	0.8	75-125	25	45-153	28
Endosulfan II	0.8	75-125	63	D-202	53
Endosulfan Sulfate	0.8	70-125	32	26-144	38
Endrin	0.8	5-125	42	30-147	48
Endrin Aldehyde	0.8	75-125	30	60-140	30
Heptachlor	0.8	75-125	28	34-140	43
Heptachlor Epoxide	0.8	75-125	22	37-142	26
Surrogates (Accustandard CLP-032-H-5X)					
2,4,5,6-Tetrachloro-m-xylene(surrogate)	0.8			60-140	
Decachlorobiphenyl (surrogate)	0.8			60-140	

APPENDIX D – NORTH CAROLINA REQUIREMENTS

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Method Blank	One per sample preparation batch of up to 20 samples	The result must be < 1/2 LoQ	Reanalyze samples
LOQ check standard	One per each day that North Carolina samples are analyzed	30-150%	Reanalyze samples. If passes, continue analysis, however all samples associated with failing LOQ check must be reanalyzed. If fails again, re-prep and reanalyze.

APPENDIX E DoD/DOE Method Specific Quality Control Requirements

Sections found in this appendix supersede and/or supplement the existing sections of this SOP. In addition to the general method performance criteria, these requirements must be met when analyzing samples for the Department of Defense (DoD) and the Department of Energy (DOE) as stipulated in the DoD/DOE Quality System Manual.

Table B-1. Organic Analysis by Gas Chromatography (GC)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Breakdown check (Endrin/DDT Method 8081 only)	Before sample analysis and at the beginning of each 12-hour shift.	Degradation of DDT and Endrin must each be ≤ 15%.	Correct problem, then repeat breakdown checks.	Flagging is not appropriate.	No samples shall be run until degradation of DDT and Endrin is each ≤ 15%.
Initial Calibration (ICAL) for all analytes (including surrogates)	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	ICAL must meet one of the three options below: Option 1: RSD for each analyte ≤ 20%; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. Quantitation for multicomponent analytes such as chlordane, toxaphene, and Aroclors must be performed using a 5-point calibration. Results may not be quantitated using a single point. No samples shall be analyzed until ICAL has passed.
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Calculated for each analyte and surrogate.

Table B-1. Organic Analysis by Gas Chromatography (GC)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention Time (RT) window width	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from the 72-hour study or 0.03 minutes, whichever is greater.	NA.	NA.	Calculated for each analyte and surrogate. Only applicable if internal standard calibration is not used.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within established RT windows. All reported analytes within ± 20% of true value.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence with the exception of CCVs for Pesticide multi-component analytes (i.e., Toxaphene, Chlordane and Aroclors other than 1016 and 1260), which are only required before sample analysis.	All reported analytes and surrogates within established RT windows. All reported analytes and surrogates within ± 20% of true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without valid CCVs. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-1. Organic Analysis by Gas Chromatography (GC)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal Standards (IS)	If employed, every field sample, standard, and QC sample.	Retention time within ± 0.06 RRT UNITS from retention time of the midpoint standard in the ICAL; Internal standard signal (area or height) within -50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV can be used.	Inspect GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the Case Narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	NA.
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid Method Blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-1. Organic Analysis by Gas Chromatography (GC)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error).
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. RPD ≤ 30% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.

Table B-1. Organic Analysis by Gas Chromatography (GC)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project if available; otherwise use DoD/DOE QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch if sufficient sample material is available. If obvious chromatographic interference is present, reanalysis may not be necessary, but the client must be notified prior to reporting data, and the failures must be discussed in the Case Narrative.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the Case Narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Confirmation of positive results (second column)	All results > the DL must be confirmed (except for single column methods such as TPH by Method 8015 where confirmation is not a requirement).	Calibration and QC criteria for second column are the same as for initial or primary column analysis. Results between primary and secondary column RPD ≤ 40%.	NA.	Apply J-flag if RPD > 40%. Discuss in the Case Narrative.	Use project-specific reporting requirements if available; otherwise, use method requirements if available; otherwise report the result from the primary column.

TABLE E-1. Aqueous LCS/MS Control Limits for Target Organochlorine Pesticides for DoD

Analyte	Aqueous Control Limits	
	Lower	Upper
4,4'-DDD	56	143
4,4'-DDE	57	135
4,4'-DDT	51	143
Aldrin	45	134
alpha-BHC	54	138
cis-Chlordane	60	129
beta-BHC	56	136
delta-BHC	52	142
Dieldrin	60	136
Endosulfan I	62	126
Endosulfan II	52	135
Endosulfan Sulfate	62	133
Endrin	60	138
Endrin Aldehyde	51	132
Endrin Ketone	58	134
gamma-BHC	59	134
trans-Chlordane	56	136
Heptachlor	54	130
Heptachlor Epoxide	61	133
Methoxychlor	54	145
Toxaphene	33	134
Chlordane	62	140

NOTE: Marginal exceedances are not allowed for those analytes determined by a project to be target analytes without project specific approval.

TABLE E-2. Solid LCS/MS Control Limits for Target Organochlorine Pesticides for DoD

Analyte	Aqueous Control Limits	
	Lower	Upper
4,4'-DDD	56	139
4,4'-DDE	56	134
4,4'-DDT	50	141
Aldrin	45	136
alpha-BHC	45	137
cis-Chlordane	54	133
beta-BHC	50	136
delta-BHC	47	139
Dieldrin	56	136
Endosulfan I	53	132
Endosulfan II	53	134
Endosulfan Sulfate	55	136
Endrin	57	140
Endrin Aldehyde	35	137
Endrin Ketone	55	136
gamma-BHC	49	135
trans-Chlordane	53	135
Heptachlor	47	136
Heptachlor Epoxide	52	136
Methoxychlor	52	143
Toxaphene	33	141
Chlordane	43	149

Note: Marginal exceedances are not allowed for those analytes determined by a project to be target analytes without project specific approval.

TABLE E-3. Surrogate Control Limits for Organochlorine Pesticides for DoD

8081 Water	Lower Control Limit	Upper Control Limit
Decachlorobiphenyl	10	122
TCMX	44	124
8081 Solid	**Lower Control Limit**	**Upper Control Limit**
Decachlorobiphenyl	57	110
TCMX	42	129

TABLE E-4. Polychlorinated Biphenyls Target List for DoD

PCB Compound	CAS #	Comments
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	Analyzed per client request
Aroclor 1268	11100-14-4	Analyzed per client request
Decachlorobiphenyl	2051-24-3	surrogate
Tetrachloro-m-xylene	877-09-8	surrogate

TABLE E-5. Aqueous LCS/MS Control Limits for Polychlorinated Biphenyls for DoD

Analyte	Aqueous Control Limits	
	Lower	Upper
Aroclor 1016	46	129
Aroclor 1260	45	134

TABLE E-6. Solid LCS/MS Control Limits for Polychlorinated Biphenyls for DoD

Analyte	Solid Control Limits	
	Lower	Upper
Aroclor 1016	47	134
Aroclor 1260	53	140

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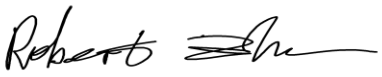
Daniel J. Wright
General Manager 1

5/14/2021 8:55:06 AM
Kelly M. Nance
Quality Manager



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Robert Zhu
Technical Specialist

5/14/2021 8:57:47 AM
Bradley E. Belding
Operations Manager

5/14/2021 8:37:25 AM
Kristina P. Bouknight
Environmental Health and Radiation Safety Officer

5/14/2021 9:44:03 AM
David A. Lucas
Supervisor

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Toxicity Characteristic Leaching Procedure
METHOD: EPA Method 1311 / ISM02.4 / SOM02.4 / SFAM01.1
ISSUER: Pace ENV - Local Quality - WCOL

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TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Toxicity Characteristic Leaching Procedure
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ISSUER: Pace ENV - Local Quality - WCOL

1. SCOPE AND APPLICATION

1.1. This SOP describes the application of the Toxicity Characteristic Leaching Procedure (TCLP), SW-846 Method 1311. The Toxicity Characteristic (TC) of a waste material is established by determining the levels of 8 metals and 31 organic chemicals in the aqueous leachate of a waste. The TC is one of four criteria in 40 CFR Part 261 to determine whether a solid waste is classified as a hazardous waste. The TC Rule utilizes the TCLP method to generate the leachate under controlled conditions that were designed to simulate leaching through a landfill. EPA's "worst case" waste disposal model assumes mismanaged wastes will be exposed to leaching by the acidic fluids generated in municipal landfills. The EPA's model also assumes the acid/base characteristics of the waste will be dominated by landfill fluids. The TCLP procedure directs the testing laboratory to use a more acidic leaching fluid if the sample is an alkaline waste, again in keeping with the model's assumption that the acid fluids will dominate leaching chemistry over time.

1.2. The specific list of TC analytes and regulatory limits may be found in Appendix A.

Note: The list in Appendix A does not include the December 1994 EPA rule for Universal Treatment Standards for Land Disposal Restrictions. Those requirements include 216 specific metallic and organic compounds and, in some cases, lower detection limit requirements (see 40 CFR 268.40). TCLP leachates are part of the new Universal Treatment Standards, but the conventional analytical methods will not necessarily meet the new regulatory limits. Consult with the client before establishing the instrumental methods for these regulations.

1.3. The TCLP is a method-defined parameter and therefore, it must be performed as written, which includes meeting all specifications for holding and tumbling times. The effect of failing to perform the method as written is that the results are not valid for the purposes of determining whether the waste is hazardous based on the toxicity characteristic.

1.4. These procedures are applicable to liquid, solid, and multiphase wastes.

1.5. The results obtained are highly dependent on the pH of the extraction solution, the length of time that the sample is exposed to the extraction solution, the temperature during extraction, and the particle size/surface area of the sample. These parameters must be carefully controlled.

1.6. The reporting limits are based on the individual samples as well as the individual analysis techniques. However, the sample is determined to be hazardous if it contains any analyte at levels greater than or equal to the regulatory limits.

1.7. If a total analysis of the waste demonstrates that individual analytes are not present in the waste or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the procedure need not be run. If the total analysis results indicate that TCLP is not required, the decision to cease TCLP analysis should be remanded to the client.

1.8. If an analysis of any of the liquid fractions of the procedure leachate indicates that a regulated compound is present at such a high concentration (after accounting for dilution from the other fractions of the leachate) that the concentration would be equal to or above the regulatory level for that compound, then the waste is hazardous and it may not be necessary to analyze the remaining fractions of the leachate. However, the remaining analyses should not be terminated without the approval of the client.

1.9. Volatile organic analysis of the leachate obtained using a bottle extraction, normally used for extractable organics and metals can be used to demonstrate that a waste is hazardous, but only the Zero Headspace Extractor (ZHE) option can be used to demonstrate that the concentration of volatile organic compounds is below regulatory limits.

1.10. For the extraction, digestion or analysis of the TCLP leachates, see appropriate sample preparation or analysis SOPs.

2. SUMMARY OF METHOD

2.1. This method has been modified within the flexibility allowable under 40 CFR 136.6. Method modification details are outlined in Section 17 of this document.

2.2. For liquid wastes that contain less than 0.5% dry solid material, the waste, after filtration through 0.6 to 0.8 µm glass fiber filter, is defined as the TCLP leachate.

2.3. For wastes containing greater or equal to 0.5% solids, the liquid, if any, is separated from the solids, and stored for later analysis. The particle size of the remaining solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. Two leachates may be generated: a) one for analysis on non-volatile constituents (semi-volatile organics, pesticides, herbicides and metals and/or b) one from a Zero Headspace Extractor (ZHE) for analysis of volatile organic constituents. Following extraction, the liquid leachate is separated from the solid phase by filtration through a 0.6 to 0.8 µm glass fiber filter.

2.4. If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste, defined as filtrate, is added to the liquid leachate and these are prepared and analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3. DEFINITIONS

Note: Refer to the *Quality Assurance Management Plan* [QAMP ME0012K] to reference additional terms used in this procedure.

3.1. ***Non-conformance Memo (NCM)*** - A form used to document a non-conforming event. An analyst must document a non-conformance memo when a non-conforming event occurs. A non-conforming event may include the reporting of analytical data outside of method or SOP criteria, or when there is a deviation from a written policy or procedure. Information in an NCM may be used by project managers to flag data in the report narrative, or by the quality department to track trends and initiate corrective actions, where applicable. Additional information on the NCM policy and procedure is located in the *Complaints and Nonconformances* SOP [QA SOP ME001BO].

3.2. ***Leachate*** refers to the TCLP solution generated from this procedure.

3.3. ***Percent Wet Solids*** is that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure.

4. INTERFERENCES

4.1. Oily wastes may present unusual filtration and drying problems. As recommended by EPA (see US Environmental Protection Agency Memorandum #35, pages 1 and 10), oily wastes will be assumed to be 100% liquid and analysis for total concentrations of contaminants will be performed. This applies specifically to samples containing viscous non-aqueous liquids that would be difficult to filter.

4.2. Wastes containing free organic liquids (i.e., those with separable non-aqueous liquid phases) will be assumed to be 100% liquid and totals analysis will be performed to determine if the oil exceeds TCLP limits.

4.3. Solvents, reagents, glassware, and other sample processing 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 as described in the individual determinative SOPs.

4.4. Glassware and equipment contamination may result in analyte degradation. Soap residue on glassware and equipment may contribute to this. All glassware and equipment should be rinsed very carefully to avoid this problem.

4.5. Phthalates may be eliminated by proper glassware cleanup and by avoiding plastics. Only glass, Teflon, or Type 316 stainless steel tumblers may be used for leachates to be analyzed for organics. Plastic tumblers may be used for leachates to be analyzed for metals.

4.6. Overexposure of the sample to the environment will result in the loss of volatile components.

4.7. Potential interferences that may be encountered during analysis and are discussed in the individual analytical methods.

5. SAFETY

5.1. Procedures must be carried out in a manner protective of the health and safety of all PAS-SC personnel. All work must be stopped in the event of a known or potential compromise to the health and safety of a PAS-SC employee. The situation must be reported immediately to the Environmental Health and Safety Officer (EHSO).

5.2. As stated in the *PAS-SC Comprehensive Chemical Hygiene, Safety, and Hazard Communication Plan* [HS SOP ME0012D], eye protection that satisfies ANSI Z87.1, a laboratory coat, and at least latex or nitrile gloves must be worn while samples, standards, and reagents are being handled. Contaminated gloves are to be removed and discarded.

5.3. Exposure to chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred, and prepared in a fume hood.

5.4. Analysts must ensure that the proper type of glassware is selected for each application.

5.5. Discard chipped or broken glassware to prevent injury. All glassware must be discarded into a broken glass container. Chipped or broken non-volumetric glassware may be sent off-site for repair as an alternative to disposal.

5.6. To ensure safe operation, analysts must adhere to safety notices and labels located on the equipment utilized during the processes outlined in this procedure.

5.7. The health and safety hazards of many of the chemicals used in these procedures have not been fully defined. Additional health and safety information can be obtained from safety data sheets (SDS) maintained electronically in the public directory. Physical and health hazards specific to this procedure:

- The following material(s) are classified as **carcinogen**: Methylene chloride.
- The following material(s) are classified as **flammable**: Methanol.
- The following material(s) are classified as **corrosive**: Hydrochloric acid, Nitric acid, Sulfuric acid, Acetic acid, and Sodium hydroxide.
- The following material(s) are classified as **oxidizer**: Nitric acid

5.8. Gas pressurized equipment is employed in this procedure. Be sure all valves and gauges are operating properly and that none of the equipment, especially tubing, is over-pressurized.

Note: Do not open equipment that has been pressurized until it has returned to ambient pressure.

5.9. Rotary agitation apparatus is used in this procedure. Certain samples may break the glass jars used in the procedure. For these samples, extra caution, including plastic or polyethylene over wraps of the glass jar, may be necessary.

5.10. Secure tumbler and extraction apparatus before starting rotary agitation apparatus. Stay clear of the apparatus while it is in motion.

5.11. During sample rotation, pressure may build up inside the bottle. Periodic venting of the bottle will relieve pressure.

5.12. Any chemical spill above 50 ml, injuries, and/or accidents must be reported to the lab supervisor, team leader, and the safety officer.

6. EQUIPMENT AND SUPPLIES

Note: Refer to *the Major Operational Equipment List* [QA Control Log ME001PM] for specific details regarding the equipment utilized during this procedure.

6.1. ***Extraction vessels***

6.1.1. *For volatile analytes* – Zero-headspace extraction (ZHE) vessel, gas-pressure actuated, Millipore ZHE1000 or equivalent, internal volume of 560 mL, and is equipped to accommodate a 90-mm filter.

6.1.2. *For metals and non-volatiles organics* – disposable one time use vessels

6.1.3. *For non-volatile organics* – Borosilicate glass jars (2200-mL) or Teflon bottles may be used.

6.2. ***Vacuum filtration apparatus (47-mm diameter filter) and stainless-steel pressure filtration apparatus*** (142-mm diameter filter), capable of 0 – 50 psi.

6.3. ***Borosilicate glass fiber filters (acid washed TCLP filters)***, 0.6 – 0.8 µm (Environmental Express – 47, 90, and 142-mm diameter or equivalent).

Note: Glass fiber filters are fragile and must be handled with care.

6.4. ***Rotary agitation apparatus, multiple-vessels*** (Associate Design # 3740-12 BRE) – Capable of rotating the extraction vessels in an end-over-end fashion at 28-32 rpm.

6.5. ***ZHE Extract Collection Devices*** (Associate Design # 3790-GTS/SS) – Stainless steel gas-tight syringes, used to collect the initial liquid phase and the final extract of the waste when from the ZHE device.

6.6. ***Laboratory Balance*** – Any laboratory balance accurate to within ± 0.01 grams may be used (all mass measurements are to be within ± 0.1 grams).

6.7. ***Analytical Balance*** – Laboratory analytical balance capable of accurately weighing to the nearest 0.005 g (±5% at 5 mg, and ±0.5% at 1000 mg), calibrated daily before use.

6.8. ***pH meter and probe*** (Orion 920 or equivalent).

6.9. ***Magnetic stirrer/hotplate and stirring bars***

6.10. ***VOA vials***, 40-mL, with caps and septa.

6.11. ***500 mL, non-preserved plastic containers***, for TCLP metals.

6.12. ***Miscellaneous laboratory glassware and equipment***.

6.13. ***Min/Max thermometer***

7. REAGENTS AND STANDARDS

Note: Other volumes of standards and reagents may be prepared to account for expected usage. As long as all ratios are kept constant, this is not considered a deviation from the approved procedure.

Note: All stored reagents and standards must be labeled as required by the *Preparation and Documentation of Laboratory Standards and Reagents* SOP [QA SOP ME001HG], the Pace Safety Manual [MasterControl], and the *Quality Assurance Management Plan* [QAMP ME0012K].

7.1. ***Reagent water*** – PAS-SC employs a series of in-house deionized (DI) tanks to purify the incoming water. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks. See the *Deionized Water System* SOP [QA SOP ME0012S] for further information.

7.2. ***Hydrochloric acid (HCl), 1 N*** – Carefully add 83 mL of concentrated HCl to 800 mL reagent water. Allow the solution to cool to room temperature. Transfer to a 1 L volumetric flask, dilute to volume with reagent water, and mix well. Follow manufacturer expiration date of stock material.

7.3. ***Nitric acid (HNO3), 1 N*** –Carefully add 64 mL of concentrated HNO3 to 800 mL reagent water. Allow the solution to cool to room temperature. Transfer to a 1 L volumetric flask, dilute to volume with reagent water, and mix well. Follow manufacturer expiration date of stock material.

7.4. ***Sodium hydroxide (NaOH), 1 N*** – Carefully add 40 g NaOH to 800 mL reagent water and dissolve. Allow the solution to cool to room temperature. Transfer to a 1 L volumetric flask, dilute to volume with reagent water, and mix well. Follow manufacturer expiration date of stock material.

Note: Heat is generated during this process.

7.5. ***Acetic acid, glacial (HOAc or CH3COOH)*** – Concentrated, reagent grade liquid.

7.6. ***pH buffers*** – pH 4, 7, and 10. Commercially available.

7.7. ***TCLP Leaching Fluids***:

7.7.1. *General comments*:

7.7.1.1. The pH of both solutions listed below must be monitored daily. pH probes are to be calibrated prior to use.

7.7.1.2. The leaching fluids must be prepared correctly. If the desired pH range is not achieved and maintained, the TCLP may yield erroneous results due to improper leaching. If the pH is not within the specifications, the fluid must be discarded and fresh extraction fluid prepared.

7.7.2. TCLP Fluid #1 – Carefully, add 22.8 mL glacial acetic acid and 257.2 mL of 1 N NaOH to 500 mL reagent water. Allow the solution to cool to room temperature. Transfer to a 1-liter volumetric flask and dilute to volume. Dilute to a final volume of 4 L with reagent water, cap, and shake to mix well. When correctly prepared, the pH of this solution is 4.93 ± 0.05.

7.7.3. TCLP Fluid #2 – Carefully, add 22.8 mL glacial acetic acid to 500 mL reagent water. Allow to the solution to cool to room temperature. Transfer to a 1 L volumetric flask and dilute to volume. Dilute to a final volume of 4 L with reagent water, cap and shake to mix well. When correctly prepared, the pH of this solution is 2.88 ± 0.05.

7.8. ***Nitric acid (HNO3), 50% solution*** – Carefully add 500 mL concentrated HNO3 to 500 mL reagent water. Allow the solution to cool to room temperature. Follow manufacturer expiration date of stock material.

7.9. ***Methanol and methylene chloride*** – Used to aid in cleaning oil contaminated equipment.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

Note: Refer to the *Sample Receiving* SOP [AD SOP ME0013H] for details regarding sample shipment and receipt.

Note: Refer to the *EQI Analytical Methods* list [AD Form ME002BS] for details regarding sample holding time, collection, preservation, and storage requirements.

8.1. Samples that are to be analyzed for non-volatile organic compounds should be collected and stored in glass containers with Teflon lid liners. Chemical preservatives must not be added until after leachate generation.

8.2. Samples that are to be analyzed for metals only can be collected in either glass or polyethylene containers.

8.3. When the sample is to be evaluated for volatile analytes, care must be taken to minimize the loss of volatiles. Samples must be collected in Teflon lined septum capped vials with minimal headspace. Samples must be opened only immediately prior to leaching.

8.4. All samples should be stored at 4 ± 2°C unless refrigeration results in irreversible physical changes to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.

8.5. The physical state or states of the waste and the analytes of concern determine the minimum TCLP sample collection size. The amount of waste required varies with the percent solids. The lower the percent solids, the more waste will be required for preliminary and final testing. For aqueous samples containing between 0.5 and 10% solids, several kilograms of sample are required to complete the analyses. The general minimal requirements when the samples are 100% solids include one 32-oz jar for semi-volatile organic and metals analyses, and one 4-oz jar for volatile organic analysis. Low-density sample materials, such as rags or vegetation, will require larger volumes of sample. For liquid samples (less than 50% solids), minimum requirements are two 32-oz jars for semi-volatile organic and metals analyses, and two 8-oz jars for volatile organic analysis. If volatile organic analysis is the only requested parameter, two separate jars are required. If matrix spike or duplicate control samples are requested for analysis, additional sample volume is required. If sufficient sample volumes were not received, analyses cannot be started and the client should be notified as soon as possible.

Note: A Nonconformance Memos (NCM) should be filed and forwarded to the project manager, QA Officer, and the Operations Director. The project manager should then notify the client as soon as possible. Upon client request, the TCLP will be followed using the available sample volume or amount; however, any results attained from the procedure may not be valid for the purposes of determining whether the waste is hazardous based on the toxicity characteristics.

8.6. TCLP leachates should be prepared for analysis and analyzed as soon as possible following extraction. Leachates or portions of leachates for metallic analyte determinations must be acidified with nitric acid to a pH less than 2, unless precipitation occurs. If precipitation occurs upon addition of nitric acid to a small aliquot of the leachate, then the remaining portion of the leachate must not be acidified and the leachate shall be analyzed as soon as possible. All other leachates should be stored at 4 ± 2°C until analyzed. ZHE leachates must be stored in VOA vials filled to eliminate all headspace.

8.7. Samples are subjected to appropriate treatment within the following time periods:

Parameter	From Collection to Start of TCLP Leach (days)	From End of TCLP Tumble to Complete Filtration (days)	From Extraction Prep to Analysis (days)	Total Elapsed Time (days)
Volatiles	14	NA	14	28
Semi-volatiles	14	7	40	61
Mercury	28	NA	28	56
Other Metals	180	NA	180	360

8.8. The ISM02.4 & SOM02.4 contract holding time requirements are as follows:

8.8.1. *ISM02.4*: The holding time for metals is 180 days from Validated Time of Sample Receipt (VTSR) to analysis. The holding time for the analysis of TCLP or SPLP leachates is 180 days from the date of extraction.

8.8.2. *SOM02.4*: The holding time for ZHE extraction of volatile soil samples or waste samples containing > 0.5% solids is 10 days from Validated Time of Sample Receipt (VTSR). The holding time for TCLP or SPLP extraction of non-volatile soil samples or waste samples containing > 0.5% solids is 10 days from VTSR. The holding time for TCLP or SPLP filtration of aqueous samples is 5 days from VTSR.

9. QUALITY CONTROL

Note: Further details of QC and corrective action guidelines are presented in the *Quality Assurance Management Plan* [QAMP ME0012K].

Note: Refer to appendices for state and/or program specific method performance criteria, which supersede and/or supplement the general method performance criteria prescribed in this SOP.

9.1. ***Quality Control for Leaching Procedure***

9.1.1. *TCLP Leaching Blanks* – A minimum of one blank (using the same fluid(s) as used for the samples) must be prepared and analyzed for every batch of samples. Depending on the type of analysis required, different types of extraction vessels may be used. One blank must be prepared for each extraction fluid per vessel type (i.e., borosilicate glass, Teflon, plastic). The blanks are generated in the same way as the samples, such as; blanks will be tumbled and filtered with the samples. Extraction vessels will be uniquely numbered. Per section 8.1 of EPA method 1311, a blank will be processed through each extraction vessel. Analysts will use a TCLP Vessel Blank Tracking Log [EXT Forms ME00195] to track the status of each vessel with regard to its use for a method blank. Blank acceptance criteria are listed in individual analysis SOPs.

9.2. ***Quality Control for Generalized Filtrate/ Leachate Batching*** - Refer to determinative SOPs for various analyses.

9.2.1. *Filtrate/ Leachate Prep Batch* – A group of up to 20 samples that are leached with the same extraction fluid and are processed together within 24 hours using the same procedures and reagents. Each leachate batch must contain a TCLP extraction blank (method blank).

9.2.2. *Sample Count (Leachate Batch)* – Laboratory generated QC samples (MB, LCS, MS, and MSD) are not counted towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

9.2.3. *TCLP Extraction Blank (Method Blank)* – One method blank must be processed with each filtrate/ leachate prep batch. The method blank consists of the same type of extraction fluid used for the associated samples. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated concentration of compounds or false data. Acceptance criteria of method blanks and corrective actions are contained within the individual analytical method SOP.

9.2.4. *Laboratory Control Sample (LCS)* – A LCS is required with each batch of 20 or fewer samples. The LCS shall be generated after a batch of TCLP leachates have been generated (i.e., at the time of the preparative digestion or extraction) by spiking an aliquot of the appropriate extraction fluid used for that batch (an aliquot of the processed TCLP blank. Consult the individual analysis SOPs for additional LCS guidance (i.e., spike amounts, spike levels, recovery criteria, etc.).

9.2.5. *Matrix Spike (MS/MSD)* – Matrix spikes are used to monitor the performance of the analytical methods on the matrix and to assess the presence of interferences. A MS (and MSD, if applicable) is required with each filtrate / leachate prep batch of 20 or fewer samples.

9.2.5.1. Matrix Spikes are to be added after filtration of the TCLP leachate. Spikes are not to be added prior to the TCLP leaching.

9.2.5.2. The use of internal calibration or alternate methods may be needed when the recovery of the matrix spike is below the expected performance.

9.2.5.3. Consult the individual analysis SOPs for additional guidance on spike compounds and levels.

9.3. ***Corrective Actions***

9.3.1. Consult the individual analysis SOPs for corrective action for blanks and LCS.

9.3.2. Method of Standard Additions (MSA) shall be used for metals if all of the following conditions are met:

9.3.3. Recovery of the analyte in matrix spike is not at least 50%.

9.3.4. The concentration of the analyte does not exceed the regulatory level.

9.3.5. The concentration of the analyte measured in the sample is within 20% of the appropriate regulatory level.

9.3.6. If the MS recovery is 5% or less due to dilution or matrix interference, contact the project manager and client for guidance. The client should also be contacted prior to initiation of any MSA steps. Refer to the individual analysis SOPs for details on how to perform MSA analysis.

10. CALIBRATION AND STANDARDIZATION

10.1. Incubators, water baths, refrigerator units, bottle top dispensers, pipettes, thermometers, and ovens are maintained and verified as required by the *Equipment and Instrumentation* SOP [QA SOP ME002JT].

10.2. The balance is verified at the beginning of each analytical day using a certified weight set. Refer to the *Equipment and Instrumentation* SOP [QA SOP ME002JT] for balance verification procedures and acceptance criteria.

10.3. Three-point calibration of pH meter is required prior to use. A QC buffer check after calibration is also required. Refer to the *pH* SOP [Wet Chem SOP ME0014S] regarding pH calibration procedures.

10.4. Refer to appropriate analysis SOPs.

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Toxicity Characteristic Leaching Procedure
METHOD: EPA Method 1311 / ISM02.4 / SOM02.4 / SFAM01.1
ISSUER: Pace ENV - Local Quality - WCOL

11. PROCEDURE

11.1. ***Preliminary Sample Evaluations***

11.1.1. *Full Scan TCLP Samples* - Analysts must proceed with extreme caution when using methylene chloride and acetone on a full TCLP sample. Only full scan TCLP (FTCLP) will be analyzing for these solvents. This will be noted in the description. Due to the potential for solvent contamination with TCLP Volatile samples (acetone and methylene chloride), the following procedure will be followed to reduce possible contamination. If a sample is in for FTCLP or any TCLP Volatiles, water used for all aspects of the TCLP leaching process (MB and samples) should come from the INM Di water system. The extraction fluid needs to be made outside the extractions laboratory to reduce solvent exposure (i.e.: the extraction fluid can be made in the inorganic non-metals laboratory). Processing of samples need to occur outside of the extractions area or under an extractions hood that is free from these solvents. Removal of the samples from the ZHEs needs to occur outside of the extractions lab environment or in an extractions hood that is free from these solvents. The ZHE must be properly cleaned. The ZHE should be cleaned in the GPC room sink to reduce solvent exposure. Volatile (VOA) vials need to come from a solvent free area (not stored in/near extractions). A graduated cylinder(s) needs to be designated and covered with parafilm to minimize solvent contamination. (Optional: ZHE metal screens should be baked at 100 degrees C to minimize contamination). These precautions must be taken to prevent solvent contamination of the sample.

11.1.2. *Preliminary TCLP evaluations* (percent solids, particle size, selection of extraction fluid, and fluid/leachate compatibility) are required to be done using a minimum of a 100-gram aliquot of waste. This aliquot may also undergo the actual TCLP extraction for non-volatiles ONLY IF it has NOT been oven dried. If the solid portion is oven dried, a separate aliquot must be used for the actual leaching procedure.

11.1.3. Consult the holding times for the appropriate tests and prioritize extractions such that holding times are not exceeded.

11.1.4. Determine the total volume of TCLP leachate (solid phase leachate + liquid filtrate) that needs to be generated for analysis using the following table as a guideline:

Analysis	Approximate Required Volume (mL)
Volatiles	2 x 40
Semi-volatiles	1000 (SOM); 100 (regular)
Pesticides	1000 (SOM); 100 (regular)
Herbicides	100 (SOM); 100 (regular)
Metals	500

11.1.5. *Sample Description*

11.1.5.1. Solid – Record the visible presence of a solid material heavier than water. If the sample contains more than one solid phase (e.g., wood and sediment mixed with water), make note in TCLP logbook.

11.1.5.2. Liquid – Record the number of liquid phases observed in the sample according to apparent density. It may be impossible to distinguish apparent density if only one liquid phase is observed and there is no indication on the COC form. If this is the case, record it as aqueous material and let the subsequent analytical record show if the liquid is organic.

11.1.6. *Percent Solid Phase*

11.1.6.1. Percent Solids and ZHE Extractions – The ZHE filtration apparatus cannot accurately determine percent solids less than 5%. If an extraction is to be performed solely for volatile organic compounds and the percent solids concentration is apparently greater than 5%, proceed to Section 11.3 (Procedure: ZHE Extraction Procedure, Volatile Constituents). Otherwise, continue with the steps in this section. The aliquot of sample used here cannot be used again for the ZHE extraction.

11.1.6.2. Determine Type of Filtration Apparatus Needed

11.1.6.2.1. If the waste will obviously yield no free liquid when subjected to pressure filtration (i.e., it is 100% solid), then proceed to Section 11.1.7 (Particle-size Reduction for Fluid Selection).

11.1.6.2.2. If the sample is mostly a non-viscous liquid (water or non-viscous organic liquid) of low solids content (<10%) or a highly granular, liquid containing waste, vacuum or pressure filtration may be used to separate filtrate from solid phase.

11.1.6.2.3. If the sample is viscous (sludge or has high solids content), use pressure filtration to separate filtrate from solid phase.

11.1.6.3. Weight of filter – Measure and record this value before loading the filter into the filter holder. The filter will collect the solid phase of the waste. The mass of the solid phase will then be used in the determination of percent wet solids (Section 11.1.6.6) and/or percent dry solids (Section 11.1.6.7).

11.1.6.4. *Weight of sub-sample and filtrate for percent solids measurement*

11.1.6.4.1. Assemble the filtration apparatus (use blunt forceps to handle the 0.6 to 0.8 µm filter membrane).

11.1.6.4.2. Homogenize the waste; transfer a minimum of a 100 g of sub-sample to a pre-weighed vessel. Measure and record the mass of the sub-sample.

11.1.6.4.3. Obtain an additional vessel to collect the filtrate, or liquid phase of the waste. Measure and record the weight of the filtration vessel.

11.1.6.4.4. Transfer the sub-sample to the filtration device, attempting to spread the waste sample evenly over the surface of the filter. Measure and record the mass of the empty weighing vessel and any residual sample. Subtract the mass of any residual sample from the mass of the sub-sample. This result, the initial mass of waste, will be used in the calculation of percent wet solids (Section 12.1.2). Pressure filtration will be needed to separate the filtrate, or liquid phase, from the

solid phase of the waste. Refer to Section 11.1.6.5 (Pressure Filtration Technique).

11.1.6.4.5. Calculate and record the mass of the solid phase that remained on the filter. This mass will also be used in the percent wet solids calculation.

11.1.6.5. *Pressure Filtration Technique*

11.1.6.5.1. Slowly apply gentle pressure or vacuum of 10 psi to the filtration apparatus. Allow the sample to filter until no significant additional liquid has passed through the filter during a 2-minute period.

11.1.6.5.2. Repeat previous step by increasing the pressure in 10-psi increments until a maximum of 50 psi is reached. Stop the filtration when no addition filtrate is generated within a 2-minute period.

Note: Some samples will contain liquid material that does not filter (e.g., oil). Do not attempt to filter the sample again by exchanging filters. Viscous oils or any waste, which does not pass through the filter, is classified as 100% liquid and analyzed as a total (Section 4.1).

11.1.6.5.3. Remove the filtrate collection vessel, weigh, and record the mass of the filtrate.

11.1.6.5.4. Pour the filtrate into a graduated cylinder. Measure and record the volume of the aqueous phase. Measure and record the volume of any organic phase. If more than one organic phase is observed, provide a description at the bottom of worksheet.

11.1.6.5.5. Retain the filtrate for use in Section 11.1.9 (Determination of Filtrate/Extraction Fluid Compatibility) and for possible recombination (Section 12.1.6.1) with the filtrate obtained in Section 11.1.6.4.4.

11.1.6.6. *Percent of wet solids*

11.1.6.6.1. Calculate and record the mass of filtrate. This result will be used in the calculation of the total mass of wet solids using equation in Section 12.1.1.

11.1.6.6.2. Calculate the percent of wet solids using equation in Section 12.1.2.

11.1.6.6.3. If the percent wet solids result is ≥ 0.5% and < 5.0% and it is noticed that a small amount of the aqueous filtrate is entrained in the wetting of the filter, proceed to Section 11.1.6.7 to complete the percent solids measurement on a dry-weight basis.

NOTE: If obviously oily (non-aqueous) material is observed on the filter, do not dry the filter; proceed to Section 11.1.7 (Particle-size Reduction for Fluid Selection).

11.1.6.6.4. If the percent wet solids result is greater than 5.0%, proceed to Section 11.1.7 (Particle-size Reduction for Fluid Selection) and make a note in the TCLP logbook.

11.1.6.6.5. If percent wet solids result is < 0.5%, discard the solid phase. No leaching will be necessary; the filtrate is equivalent to the final leachate.

11.1.6.7. W*eight percent of dry solids (skip this step for oily samples)*

Note: These steps are required only if it is noticed that a small amount of the filtrate is entrained in wetting of the filter and the percent wet solids content is ≥.5% and < 5.0%. This step is not to be used for the determination of percent solids for ZHEs.

11.1.6.7.1. Remove the filter with the wet solids from the filtration apparatus.

11.1.6.7.2. Dry the filter and solid phase at 100 ± 20°C.

11.1.6.7.3. Remove the filter from the oven and allow cooling in a desiccator.

11.1.6.7.4. Weigh and record the dry mass.

11.1.6.7.5. Repeat the drying step. Weigh and record the second dry mass. If the two weightings do not agree within 1%, perform additional drying and weighing until successive weighing agree within 1%.

11.1.6.7.6. Calculate the percent of dry solids using equation in Section 12.1.5.

11.1.6.7.7. If the dry solids result is ≥ 0.5% and the sample will be extracted for non-volatile constituents, proceed to Section 11.2.7 (Particle-size Reduction) using a fresh wet portion of waste.

11.1.6.7.8. If the percent solids result is < 0.5%, discard the solid phase. No leaching will be necessary; the filtrate is the TCLP leachate. Proceed to Section 11.1.9 (Determination of Filtrate/Extraction fluid Compatibility) to determine whether the material is a non-aqueous, immiscible liquid.

11.1.7. *Particle-size Reduction for Fluid Selection*

11.1.7.1. The sub-sample used for fluid selection must consist of particles < 1 mm in diameter (versus the < 9.5 mm requirement for the material used for the actual extraction). The method requires a smaller particle size to partially compensate for the shorter duration of contact time with the leachate solution as compared to the full extraction. Inappropriate use of coarser materials could result in the selection of the wrong fluid type.

11.1.7.2. Surface area exclusion – Size reduction is not required if the sample surface area is ≥ 3.1 cm^2/g.

11.1.7.3. If the sample contains particles > 1 mm in diameter, crush, cut, or grind the solids to the required size. Note in logbook that sample required particle-size reduction for fluid selection.

11.1.7.4. Consult with group leader or supervisor when dealing with unusual sample matrices (e.g., wood, cloth, metal, brick). Scissors or shears may be used to cut cloth, plastic, or sheet metal. Saws may be used for wood or solid metal. Brick, rocks, or other solids amenable to grinding may be subcontracted out for particle size reduction. Note that size reduction to fine powder is not appropriate, and could invalidate results.

11.1.8. *Determination of Appropriate Extraction Fluid*

11.1.8.1. If the solid content is ≥ 0.5%, and if the sample is being analyzed for metals or non-volatile organic compounds, the type of leaching solution must be determined.

11.1.8.2. Follow times, temperature, and particle size specified in this section as closely as possible. If reaction time between the acid solution and solid waste is too short or too long, the procedure may produce false pH readings.

11.1.8.3. The TCLP leaching fluid for all volatiles is Fluid #1.

11.1.8.4. For TCLP leaching fluid determination for non-volatile analytes, continue with the following steps.

11.1.8.5. Calibrate the pH meter with fresh buffer solution. Weigh out a 5.0 ±0.1 g sub-sample (< 1 mm particle size) of the solid phase into a 150-mL beaker. If different weight used, enter the actual weight in TCLP logbook.

11.1.8.6. Add 96.5 mL of reagent water, cover with a watch glass, and stir for 5 minutes on a stirrer. If a different volume used, enter the actual volume in TCLP logbook.

11.1.8.7. Measure and record the sample pH.

11.1.8.8. If the pH is ≤ 5.0, use Fluid #1 and proceed to Section 11.1.9 (Determination of Filtrate/Extraction Fluid Compatibility).

11.1.8.9. If the fluid pH is > 5.0, add 3.5 mL 1 N HCl solution and cover with a watch glass. Slurry the sample briefly then heat at 50°C for 10 minutes.

Note: The heating cycle is a critical step. If the solid waste does not remain in contact with the acidic solution under specified time and temperature conditions, an erroneous pH may be measured.

11.1.8.10. Cool to room temperature.

11.1.8.11. Measure and record the pH immediately after the sample has reached the room temperature.

11.1.8.11.1. If the pH is ≤ 5.0, use Fluid #1.

11.1.8.11.2. If the pH is > 5.0, use Fluid #2.

11.1.9. *Determination of Filtrate/Extraction Fluid Compatibility*

11.1.9.1. Place 5 mL of the appropriate leaching fluid (determined in the previous step) into a 12-mL vial with PTFE lined screw cap.

Note: Use fluid type #1 if simply testing the filtrate for a sample with < 0.5% solids.

11.1.9.2. Add 5 mL of the initial filtrate cap and shake.

11.1.9.3. If the phases are miscible, the initial filtrate and solid phase leachate will be physically recombined upon completion of the leachate generation.

11.1.9.4. If the phases are NOT miscible, note in TCLP logbook. The initial filtrate and the solid phase leachate will be prepared and analyzed separately and the results mathematically combined (see Section 12.1.6.1).

11.1.10. For samples requiring analysis for semi-volatile organics, pesticides, herbicides or metals, proceed to Section 11.2.

11.1.11. For samples requiring analysis for volatile organics (ZHE), proceed to Section 11.3.

11.2. ***Bottle Extraction Procedure*** - Non-volatile Constituents (Semi-volatiles, Pesticides, Herbicides, Metals)

11.2.1. All masses should be recorded to the nearest 0.1 g.

11.2.2. The aliquot used in the Preliminary Evaluation MAY be use for this procedure ONLY IF it was not oven dried. If the sample is 100% solid or if the preliminary aliquot was not oven dried, proceed directly to Section 11.2.7 (Particle-size Reduction). If the preliminary evaluation aliquot was oven dried then, using a fresh aliquot of sample, continue a described in Sections 11.2.3 through 11.2.6.

11.2.3. Examine the sample and determine the type of filtration to employ per Section 11.1.6.2.

11.2.4. Repeat the steps outlined in Section 11.1.6.4.2 through 11.1.6.7.4 using a sufficient aliquot to obtain 100 g of the solid phase for agitation or enough solid to support the analyses to be performed on the TCLP leachate.

11.2.5. Determine and record the volume (mass) of the initial filtrate. Transfer filtrate from the collection vessel into a 1-L amber jar that is labeled appropriately (leach date and sample ID) and store at 4 ± 2°C until needed.

11.2.6. Determine and record the “solid” phase mass by subtracting the mass of the liquid filtrate from the mass of the sub-sample.

11.2.7. Evaluate the solid portion of the waste for particle size. If it contains particles > 9.5 mm in size, prepare the solid portion of the waste for leaching by crushing, cutting, or grinding such that all particles are < 9.5 mm in size (i.e., capable of passing through a 9.5 mm, 0.375-inch, standard sieve). Size reduction is not required if the sample surface area is ≥ 3.1 cm2/g. If particle size reduction was required, note in TCLP logbook.

Note: Consult the group leader when dealing with unusual sample matrices (e.g., wood, cloth, metal, brick). Scissors or shears may be used to cut cloth, plastic, or sheet metal. Saws may be used for wood or solid metal. Brick, rocks, or other solids amenable to grinding may be subcontracted out for particle size reduction. Note that size reduction to fine powder is not appropriate, and could invalidate results.

11.2.8. Weigh a maximum of 100 g of solid phase into an appropriate bottle (plastic for metals only, glass, or Teflon for all others) and slowly add 20 times its mass (2000 mL if 100 g is used) of appropriate leaching fluid as determined under Section 11.1.8. Record the weight of the sample aliquoted for the extraction and the amount of extraction fluid added in *TCLP* logbook [EXT forms ME0014A]

11.2.9. Ensure any effervescence has stopped before capping the bottle tightly. Secure in a rotary agitator and rotate end-over-end at 28-32 rpm for 16-20 hours. The temperature of the room must be 23 ± 2°C. The room temperature and time must be checked at both the start and end of the extraction and recorded in *TCLP* logbook [EXT forms ME0014A]. The minimum and maximum temperature over the entire time period must be recorded in the logbook as well. The minimum and maximum temperature can be monitored via the use of a min/max thermometer. If the room temperature is outside of this criterion, an NCM must be submitted to QA and the project manager.

Note: As agitation continues, pressure may build up within the bottle for some types of wastes. To relieve excessive pressure, the bottle may be removed and opened periodically in a properly vented hood (e.g. at 15, 30, and 60 minutes.)

11.2.10. Remove the bottle and filter the sample using vacuum or pressure filtration by filtering through a new glass fiber filter as discussed in Section 11.1.6.4.1. For final filtration of the TCLP leachate, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filters must be acid washed if metals are to be determined (see Section 6.3). Sufficient volume should be generated to support the required analyses and/or calculations (Section 11.1.4).

11.2.11. If the waste did yield an initial filtrate, consult the TCLP logbook for initial filtrate/leachate compatibility. If they are compatible, they are to be combined in the correct proportions (see Section 12.1.6) and mixed well. This combined solution is defined as the TCLP leachate.

11.2.12. If the individual phases are NOT compatible, they are to be prepared and analyzed separately and the results combined mathematically. See Section 12.1.6.1.

11.2.13. Measure and record the pH of the TCLP leachate in appropriate logbook. (Do not attempt to measure the pH of oily samples, as the probe may be rendered inoperable.)

11.2.14. Immediately preserve the leachates as follows:

11.2.14.1. Metals aliquots must be acidified with nitric acid to pH < 2. If precipitation is observed upon addition of nitric acid to a small aliquot of the extract, then the remaining portion of the extract for metals analyses shall not be acidified and the extract shall be analyzed as soon as possible.

11.2.14.2. All other aliquots must be stored under refrigeration (4 ± 2°C) until analyzed. The TCLP extract shall be prepared and analyzed according to appropriate analytical methods.

Note: Refer to Section 8.6 if precipitation occurs upon preservation.

11.2.15. Label each sample with the appropriate information (leach date and sample ID) and submit to the appropriate analytical groups for prep and analysis with copies of the TCLP preparation worksheet.

11.3. ***ZHE Extraction Procedure*** - Volatile Constituents

11.3.1. Use the ZHE device to obtain a TCLP leachate for analysis of volatile compounds only. Leachate resulting from the use of the ZHE must NOT be used to evaluate the mobility of non-volatile analytes (e.g., metals, pesticides, etc.).

11.3.2. Due to some shortcomings of the method, losses of volatile compounds may occur. Extra care should be observed during the ZHE procedure to ensure that such losses are minimized. Charge the ZHE with sample only once and do not open the device until the final extract has been collected. Do not allow the waste, the initial liquid phase, or the extract to be exposed to the atmosphere any longer than necessary.

11.3.3. If the TCLP extraction is for volatile components only, refer to Section 11.1.1 before proceeding.

11.3.4. All masses should be recorded to the nearest 0.1 g.

11.3.5. Consult the TCLP logbook and examine the sample. If the sample appears to be different from the preliminary information found in the TCLP logbook, consult your group leader or supervisor.

11.3.6. If the preliminary evaluations indicated the need for particle size reduction, homogenize the waste, weigh out a sufficient size sample and prepare for leaching by crushing, cutting, or grinding such that all particles are < 9.5 mm in size as measured with a ruler (Do NOT sieve the sample). Size reduction is not required if the sample surface area is ≥ 3.1 cm2/g. If particle size reduction was required, record this in TCLP logbook.

Note: To minimize loss of volatiles, samples for volatiles that require particle size reduction should be kept in sample storage (at 4 ± 2°C) until immediately before size reduction. Aggressive reduction, which would generate heat, should be avoided and exposure of the waste to the atmosphere should be avoided to the extent possible. Size reduction to a fine powder is not appropriate.

Note: Consult your supervisor or manager when dealing with unusual sample matrices (e.g., wood, cloth, metal, brick). Scissors or shears may be used to cut cloth, plastic, or sheet metal. Saws may be used for wood or solid metal. Brick, rocks, or other solids amenable to grinding may be subcontracted out for particle size reduction.

11.3.7. Determine the appropriate size sub-sample to weigh using the percent solids information from Section 11.1.6 and record the weight used in TCLP logbook.

11.3.7.1. For wastes that are 100% solids, a maximum of 25g of sample is used.

11.3.7.2. For wastes containing < 0.5% solids, the liquid portion of the waste is defined as the TCLP leachate.

11.3.7.3. For wastes containing ≥ 0.5% and < 5.0% solids, a 500 g sub-sample of waste is recommended for leaching.

11.3.7.4. If the sample has ≥ 5.0% solids, the appropriate sample size should be determined using the equation in Section 12.1.3.

Note: For wastes containing > 0.5% wet or dry solids (Section 11.1.6), the "solids" value from the ZHE filtration process may be used to determine the volume of fluid to load into the ZHE. This approach is recommended since the solids value from Section 11.1.6 may differ from the filtration solids due to sample variability or differences in the filtration apparatus.

11.3.8. Homogenize and transfer an appropriate size sub-sample of the waste into the ZHE and record the mass in TCLP logbook.

11.3.9. Carefully place the glass fiber filter between the support screens and secure to the ZHE. Tighten all the fittings.

11.3.10. Place the ZHE in a vertical position; open both the gas and liquid inlet/outlet valves. Attach a gas line to the gas inlet/outlet valve.

11.3.11. If the waste is 100% solid, slowly increase the pressure to a maximum of 50 psi to force out as much headspace as possible and proceed to Section 11.3.20.

11.3.12. If the waste is < 100% solid, carefully apply gentle pressure of 10 psi (or more, if necessary) to force all headspace slowly out the ZHE. At the FIRST appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue gas pressure.

11.3.13. Assemble a syringe and place the plunger in all the way. Adjust the tension on the plunger to provide slight drag. Attach the syringe to the liquid inlet/outlet valve and open the valve. Measure and record the tare weight of a 40 mL VOA vial in the TCLP logbook.

11.3.14. Carefully apply gas pressure to no more than 10 psi to force out the liquid phase. Allow the sample to filter until no significant additional filtrate has passed in a 2-minute period.

Note: If the capacity of the syringe is reached, close the liquid inlet/outlet valve, discontinue gas pressure, and remove the syringe. Discharge the filtrate from syringe into the 40 mL VOA vials so that there is no headspace when the cap is placed on the vial. Return to Section 11.3.13 to collect any additional filtrate that may be left in the ZHE.

11.3.15. Repeat previous step increasing the pressure in 10-psi increments until 50 psi is reached and no significant liquid has passed in a 2-minute period. Remove the collection device and transfer the filtrate to the pre-weighed 40 mL VOA vial. Close the valve and discontinue gas pressure. A 5 mL portion of the filtrate will need to be collected separately to check Filtrate/Extraction Fluid Compatibility (Section 11.1.9).

11.3.16. Record the total weight of the VOA vial with filtrate on worksheet. Label the vial appropriately (sample ID, method, prep batch number). Calculate the weight of filtrate collected and record in TCLP logbook.

Note: If the original waste contained < 0.5 % solids, this filtrate is defined as the TCLP leachate and you may proceed to Section 11.3.25. Otherwise, save the vials by storing at 4 ± 2°C under minimal headspace conditions, for recombination as in Section 11.3.24.

The material remaining in the ZHE is defined to be the “solid” phase. Calculate the weight of the solid phase using equation in Section 12.1.1.

11.3.17. Based on the information from Section 11.3.7 and using the formula in 12.1.4, determine the mass of fluid to load into the ZHE on the “solid” phase. The ZHE device has approximately a 500-mL capacity. Based on the need to add an amount of extraction fluid equal to 20 times the mass of the “solid” phase, the ZHE can therefore accommodate a maximum of 25 grams of “solid”.

Note: The TCLP ZHE prep uses only TCLP fluid #1; the SPLP ZHE prep uses only SPLP fluid #3.

11.3.18. Using a Class A graduated cylinder, add the proper amount of extraction fluid to the vessel. Place a glass fiber filter between the support screens and secure to the ZHE. Tighten all fittings.

11.3.19. Attach a gas line to the gas inlet/outlet valve. Carefully apply a gentle pressure 10 psi (or more, if necessary) to force all headspace slowly out of the ZHE. Close the liquid inlet/outlet valve.

11.3.20. Pressurize the ZHE to 5-10 psi (record as initial psi in TCLP logbook) and place in the rotary agitator. Rotate at 28-32 rpm for 16-20 hours. Room temperature must be 23 ± 2°C. The room temperature and time must be checked at both the start and end of the extraction and recorded in the *TCLP* logbook [EXT Forms ME0014A]. The minimum and maximum temperature over the entire time period must be recorded in the logbook as well. The minimum and maximum temperature can be monitored via the use of a min/max thermometer.

11.3.21. Confirm that the pressure of 5-10 psi (record as final psi in TCLP logbook) was maintained throughout the leaching. If it was NOT maintained, return to Section 11.3.17 and repeat the leachate with a new aliquot of sample.

11.3.22. Assemble a syringe and place the plunger in all the way. Adjust the tension on the plunger to provide slight drag. Attach the syringe to the liquid inlet/outlet valve and open the valve.

11.3.23. Carefully apply gas pressure to no more than 10 psi to force out the liquid phase. Allow the sample to filter until no significant additional filtrate has passed in a 2-minute period.

Note: If the capacity of the syringe is reached, close the liquid inlet/outlet valve, discontinue gas pressure, and remove the syringe. Discharge the filtrate from syringe into the 40 mL VOA vials so that there is no headspace when the cap is placed on the vial.

11.3.24. Repeat previous step increasing the pressure in 10-psi increments until 50 psi is reached and no significant liquid has passed in a 2-minute period. Remove the collection device and transfer the filtrate to a 40 mL VOA vial. Close the valve and discontinue gas pressure.

11.3.25. If the waste contained an initial filtrate that is miscible with the solid phase leachate, the solid phase leachate and the initial filtrate are directly recombined in the correct proportions (see Section 12.1.6). If the individual phases are NOT compatible, they are to be collected, prepped, and analyzed separately.

Note: Chill the filtrate and receiving vessels before recombining.

11.3.26. Following collection, store the TCLP leachate in two 40-mL VOA vials with minimal headspace at 4 ± 2°C and prepare for analysis

11.3.27. If the individual phases are analyzed separately, combine the results mathematically by using the recombination calculation in Section 12.1.6.1.

11.4. ***Procedural Variations*** – For compliance samples, all procedures outlined in this SOP must be followed without exception. In the event that a deviation from this SOP cannot be avoided (i.e., demonstrated and uncorrectable matrix interference, non-compatible matrix, insufficient sample amount due to client or laboratory error) it is imperative that a NCM is completed that clearly documents the anomaly. This anomaly must be communicated to the client such that corrective action activity including sample recollection can be performed. Based upon the type and severity of anomaly, the results may not be appropriate for compliance reporting. Discussions between the client, the appropriate regulatory authority, and Shealy will be necessary to resolve certain anomalies. In any case, it is essential that any anomaly be documented on an NCM.

12. DATA ANALYSIS AND CALCULATIONS

12.1. ***Calculations***

12.1.1. *Mass of Wet Solids*

Mass of Wet Solids = Total mass of sub – sample – mass of filtrate

12.1.2. *Calculation of Percent Wet Solids*

$$\text{Percent Wet Solid} = \frac{\text{Mass "Solid" Phase} \times 100}{\text{Mass (initial sub sample)}}$$

12.1.3. *Calculation of mass of waste to charge to ZHE*

$$\text{Mass of waste to charge to ZHE} = \frac{25}{\%\text{ wet solids}} \times 100$$

12.1.4. *Calculation of mass of extraction fluid to use*

$$\text{Mass of extraction fluid} = \frac{20 \times \%\text{ wet solid} \times \text{weight of waste to be extracted}}{100}$$

12.1.5. *Calculation of Percent Dry Solids*

Note: Percent dry solids calculations are not to be used in the TCLP ZHE leaching procedures.

Mass of dry waste and filter – mass of filter = Percent Dry Solids

12.1.6. *Calculation of volume of initial filtrate phase to recombine with solid phase leachate*

12.1.6.1. Mathematically recombination of analytical results:

$$\text{Final analyte concentration} = \frac{(V1 \times C1) + (V2 \times C2)}{(V1 + V2)}$$

Where,

V1 = total volume of the initial filtrate phase (L)
C1 = analyte concentration in initial filtrate phase (mg/L)
V2 = volume of the theoretical solid phase leachate (L)
C2 = analyte concentration in solid phase leachate (mg/L)

12.2. ***Reporting requirements***

12.2.1. *Follow these reporting conventions for multi-phase samples*:

12.2.1.1. If both phases have positive results, use the values from each phase to calculate the recombined result. Use the reporting limit for each phase to calculate the recombined reporting limit.

12.2.1.2. If both phases are “ND”, not detected, the recombined result is “ND”, and the reporting limit is calculated from the reporting limit for each phase.

12.2.1.3. If one phase is “ND” and the other phase has a positive result, use the reporting limit for the “ND” phase and the positive value for the other phase to calculate the combined result. The combined reporting limit is based on the reporting limit for both phases. If the combined result is less than the combined reporting limit, then supply a footnote to indicate, “A positive result was detected below the calculated detection limit.”

12.2.2. *Units* – Regardless of the nature of the sample, all TCLP results are reported in units of mg/L.

12.2.3. For limits and significant figures, refer to appropriate analytical methods.

12.2.4. *Anomalies* – All anomalies observed during the leach procedure must be noted in the TCLP logbook. Some examples of such anomalies are:

12.2.4.1. Sample was monolithic – Sub-sample was obtained by crushing, cutting, grinding, sawing, etc.

12.2.4.2. Insufficient sample – Less than the required 100 g minimum was available.

12.2.4.3. Multiple phases – “X” phases were present.

12.2.4.4. Sample was oil – Single phase.

12.2.4.5. Sample contained liquid that did not filter under test conditions.

12.3. ***Review requirements***

12.3.1. Review all applicable holding times. If a holding time was exceeded, confirm that a Nonconformance Memo was properly documented and routed.

12.3.2. If Total analysis results are available, those results may be compared with the TCLP analysis results according to the following:

Total ≥ 20 × TCLP (Assumes the sample is 100% solid)

12.3.3. Total constituent analysis results can be used to demonstrate the TCLP protocol is unnecessary. In performing a TCLP analysis, there is a 20:1 dilution of the original sample with the leaching solution. Thus, if the "total constituent" result is less than 20 times the TC level, it is impossible for the leachate to "fail" and the TCLP does not need to be performed. For example, the TC level for lead is 5.0 mg/L (ppm). Therefore, if a sample of lead-contaminated soil contains less than 100 ppm total lead, a TCLP test need not be run for lead.

13. METHOD PERFORMANCE

13.1. Prior to institution of any method for which data will be used for compliance reporting, the method must be validated; routine quality control procedures are utilized to monitor the validity of the method. Refer to the *Quality Assurance Management Plan* [QAMP; ME0012K] for information regarding method performance and data quality objectives.

13.2. Refer to the individual analysis SOPs.

13.3. ***Training Qualification*** – The group leader has the responsibility to ensure that an analyst who has been properly trained in its use and has the required experience performs this procedure.

14. POLLUTION PREVENTION

14.1. Pollution prevention encompasses any technique used to reduce the volume or toxicity of a waste at the point of generation. Reagents and standards should be purchased and/or prepared in volumes consistent with laboratory use to minimize the production of hazardous waste from unused and/or expired surplus chemicals.

15. WASTE MANAGEMENT

15.1. Wastes generated in this procedure must be segregated and disposed according to the *PAS-SC Hazardous and Non-Hazardous Laboratory Waste Management Plan* [HS SOP ME0012A]. The Waste Manager must be contacted if additional information is required.

16. REFERENCES

Note: Where references exclude a date or edition, the latest edition of the referenced document adopted/recognized by the laboratory's accreditation bodies applies. Refer to the *Quality Assurance Management Plan* [QAMP ME0012K] for details.

16.1. SW-846, Test Method for Evaluating Solid Waste, Third Edition – *Toxicity Characteristic Leaching Procedure, Method 1311*, Revision 0, July 1992.

16.2. *USEPA Contract Laboratory Program SOW for Inorganic Analysis*.

16.3. *USEPA Contract Laboratory Program SOW for Organic Analysis*.

16.4. *General Requirements for the Competence of Testing and Calibration Laboratories.* International Standard ISO/IEC 17025.

16.5. *Laboratory Accreditation Standards.* TNI Standard. The NELAC Institute.

17. MISCELLANEOUS

17.1. Sufficient records must be maintained to allow for historical reconstruction of testing procedures. Refer to the *Logbook and Data Recording* SOP [QA SOP ME0012T] for details regarding documentation requirements.

17.2. ***Modifications/Interpretations from Reference Methods***

17.2.1. *Preliminary Evaluations* - Section 7.1 of the source method states that the sample aliquot used for the preliminary evaluation "… may not actually undergo TCLP extraction." Section 7.1.5 of the source method indicates that the portion used for the preliminary evaluation may be used for either the ZHE or non-volatile extraction if the sample was 100% solid. Section 7.1.5 further indicates that if the sample was subjected to filtration (i.e., < 100% solid) that this aliquot may be used for the non-volatile extraction procedure only as long as sufficient sample is available (minimum 100 g). Samples that have been subjected to the oven-drying step may not be used for TCLP extraction because solid phase degradation may result upon heating.

17.2.2. *Percent Solids Determination* - Section 7.1.2 of the source method indicates that "if the percent wet solids are ≥ 0.5% and it is noticed that a small amount of the filtrate is entrained in wetting of the filter: that the filter should be oven dried to determine percent dry solids. Drying of oil or organic matrices can both be hazardous and inappropriate. Additionally, it may be impossible to achieve a constant weight when performing this step. Due to safety concerns, if obviously oily or heavy organic matrices are observed on the filter, the filter is not oven dried.

17.2.3. *Preliminary Determination of Filtrate/Extraction Fluid Compatibility* -Section 7.2.13 of the source method provides no guidance as to how to make this determination. As a result, the procedure herein was developed and incorporated into the Preliminary Determination section.

17.2.4. *Determination of Appropriate Extraction Fluid* - Method 1311 does not address the appropriate approach to take if the pH equals 5.0. This SOP requires that Fluid #1 must be used if the pH is ≤ 5.0.

17.2.5. Section 8.2.2 of the source method states, "In most cases, matrix spikes should be added at a concentration equivalent to the corresponding regulatory level." The method also states "If the analyte concentration is less than one half the regulatory level, the spike concentration may be as low as one half of the analyte concentration but may not be less than five times the method detection limit". For several analytes, spiking at the regulatory level is inappropriate to the range of analysis afforded by the determinative methods. Due to the wide range in these levels, Shealy spikes at the levels specified in the determinative SOPs.

17.3. ***Cleaning of the ZHE apparatus*** – The Zero Headspace Extractor (ZHE) can be difficult to clean and can pose a high risk for contaminating future TCLP samples if the following cleaning procedures are not used.

17.3.1. If the ZHE has been previously used, then make sure all solids from the unit have been properly disposed.

17.3.2. Disassemble the ZHE with care so that the individual parts are not nicked or otherwise damaged.

17.3.3. Wipe all of the ZHE parts with paper towels to remove excess waste and dispose of these towels with the sample waste. Use detergent to clean all of the ZHE parts followed by methanol and water rinses. Repeat this step until there is no visible contamination when the surface and crevices of the ZHE are wiped with a clean, white towel.

17.3.3.1. In cases where the ZHEs are contaminated with oily waste it may be necessary to first wipe or rinse all parts with methylene chloride, followed by methanol and water. Do not soak the o-rings or other polymer/plastic parts in the methylene chloride since this may result in damage. Following the methanol rinse, clean each part again thoroughly with detergent. Repeat these steps until there is nothing physically stuck or coated on the ZHEs and o-rings.

17.3.4. Soak the small stainless steel and o-rings in a beaker with methanol.

17.3.5. Reassemble the ZHEs.

17.3.6. After a ZHE has been cleaned, allow it to air dry. This will eliminate the possibility of corrosion or mold growth. The components must be covered with aluminum foil.

APPENDIX A: TABLES

Table A1. Toxicity Characteristic Analytes and Regulatory Levels

Analyte	Regulatory Level (mg/L)
Arsenic	5.0
Barium	100.0
Benzene	0.5
Cadmium	1.0
Carbon tetrachloride	0.5
Chlordane	0.03
Chlorobenzene	100.0
Chloroform	6.0
Chromium	5.0
o-Cresols	200.0
m-Cresols	200.0
p-Cresols	200.0
Total Cresols (used if isomers not resolved)	200.0
2,4-D	10.0
2,4-Dichlorobenzene	7.5
1,2-Dichloroethane	0.5
2,4-Dinitrotoluene	0.13
1,1-Dichloroethylene	0.7
Endrin	0.02
Heptachlor (& Epoxide)	0.008
Hexachlorobenzene	0.13
Hexachlorobutadiene	0.5
Hexachloroethane	3.0
Lead	5.0
Lindane	0.4
Mercury	0.2
Methoxychlor	10.0
Methyl ethyl ketone	200.0
Nitrobenzene	2.0
Pentachlorophenol	100.0
Pyridine	5.0
Selenium	1.0
Silver	5.0
Tetrachloroethylene	0.7
Toxaphene	0.5
Trichloroethylene	0.5
2,4,5-Trichlorophenol	400.0
2,4,6-Trichlorophenol	2.0
2,4,5-TP (Silvex)	1.0
Vinyl chloride	0.2

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Toxicity Characteristic Leaching Procedure
METHOD: EPA Method 1311 / ISM02.4 / SOM02.4 / SFAM01.1
ISSUER: Pace ENV - Local Quality - WCOL

REVISION HISTORY

Revision #	Revision Date	Section Modified	Modification	Reason for Modification
-07	**7/17/2020**	All	Updated some occurrences of 'should' to 'must'	As per NC SOP audit
		11.1.8.6	Updated amount of reagent water added from 95 mL to 96.5 mL in response to finding and in concurrence with method.	In response to NC DEQ on-site audit 01/2020 - Finding III (CP-0GG)
-08	**05/14/2021**	All	Added reference to SFAM	New CLP SOW

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Daniel J. Wright
General Manager 1

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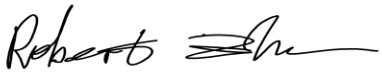
Kelly M. Nance
Quality Manager



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Robert Zhu
Technical Specialist

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Bradley E. Belding
Operations Manager

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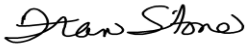
Kristina P. Bouknight
Environmental Health and Radiation Safety Officer



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Fran Stone
Supervisor

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Inductively Coupled Plasma - Atomic Emission Spectroscopy
METHOD: Method 6010D
ISSUER: Pace ENV - Local Quality - WCOL

Table of Contents

1.0 Scope and Application

This standard operating procedure (SOP) describes the laboratory procedure for the analysis of trace elements including metals in solution by Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES) using SW-846 Method 6010D. Table I lists the elements appropriate for analysis by Methods 6010D. Additional elements may be analyzed under Method 6010D provided that the method performance criteria presented in Section 13 are met. Appendix H lists the requirements and elements for analyzing DoD samples by method 6010D.

ICP analysis provides for the determination of metal concentrations over several orders of magnitude. Detection limits, sensitivity and optimum concentration ranges of the metals will vary with the matrices and instrumentation used.

1.1 Target Analyte List and Limits of Quantitation (LOQ)

The target analytes and the normal LOQ that can be achieved with this procedure are provided in Table I, Appendix A.

LOQ are established in accordance with Pace policy and SOPs for method validation and for the determination of detection limits (DL) and quantitation limits (LOQ). DL and LOQ are routinely verified and updated when needed. The current LOQ for each target analyte that can be determined by this SOP as of the effective date of this SOP is provided in Table 1, Appendix A.

The reporting limit (RL) is the value to which analytes are reported as detected or not detected in the final report. When the RL is less than the lower limit of quantitation (LLOQ), all detects and non-detects at the RL are qualitative. The LLOQ is the lowest point of the calibration curve used for each target analyte.

DL, LOQ, and RL are always adjusted to account for actual amounts used and for dilution.

1.2 Applicable Matrices

Method 6010D is applicable to the determination of dissolved, suspended, total recoverable and total elements in ground water, aqueous samples, soils, sludges, wastes, sediments, TCLP, and other leachates/extracts. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples. Although digestion is not specifically required by the method, some clients and regulators may require digestion of dissolved samples and this must be clarified and documented before project initiation. Silver concentrations upon analysis must be below 2.0 mg/L in aqueous samples and 100 mg/kg in solid matrix samples. If the concentration exceeds this level, samples must be re-digested at a dilution until the analysis solution contains < 2.0 mg/ L or 100 mg/kg of silver. Precipitation may occur in samples where silver concentrations exceed these levels and lead to the generation of erroneous data

2.0 Summary of Method

This method describes a technique for multi-elemental determinations in solution using sequential or simultaneous optical systems and axial radial viewing of the plasma. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer. The intensities of the emission lines are monitored. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interferences and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences should also be recognized, and appropriate actions taken. Alternatively, multivariate calibration methods may be chosen for which point selection for background correction is superfluous since whole spectral regions are processed.

Refer to appropriate SOPs for details on sample preparation methods.

3.0 Interferences

3.1 Spectral, physical, and chemical interference effects may contribute to inaccuracies in the determination of trace elements by ICP. Spectral interferences are caused by:

- Overlap of a spectral line from another element.
- Unresolved overlap of molecular band spectra.
- Background contribution from continuous or recombination phenomena.
- Stray light from the line emission of high concentration elements.

3.2 A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background correction is not required in cases where a background corrective measurement would actually degrade the analytical result.

3.3 Inter-element correction factor (IEC's) is necessary to compensate for spectral overlap. Inter-element interferences occur when elements in the sample emit radiation at wavelengths so close to that of the analyte that they contribute significant intensity to the analyte channel. If such conditions exist, the intensity contributed by the matrix elements will cause an excessively high (or sometimes low) concentration to be reported for the analyte. Inter-elements corrections must be applied to the analyte to remove the effects of these unwanted emissions.

3.4 Physical interferences are generally considered to be effects associated with sample transport, nebulization, and conversion within the plasma. These interferences may result in differences between instrument responses for the sample and calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects), at the point of aerosol formation and transport to the plasma (e.g., surface tension) or during excitation and ionization processes within the plasma itself. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, dilution of the sample, use of a peristaltic pump, mass flow controller, use of an internal standard, and/or use of a high solids nebulizer can reduce the effect.

3.5 Chemical interferences are characterized by molecular compound formation, ionization effects, and solute vaporization effects. Normally these effects are not significant with the ICP technique but if observed can be minimized by buffering the sample, matrix matching, or standard dilution procedures.

4.0 Definitions

Refer to the *Laboratory Quality Manual* [QAMP ME0012K] for a glossary of common lab terms and definitions.

4.1 Dissolved Metals – Those elements that pass through a 0.45 □m membrane. (Sample is acidified after filtration.)

4.2 Suspended Metals – Those elements that are retained by a 0.45 □m membrane.

4.3 Total Metals – The concentration determined on an unfiltered sample following vigorous digestion.

4.4 Total Recoverable Metals – The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

4.5 Preparation Batch – Composed of 1 to 20 environmental samples of the same matrix

4.6 Non-conformance Memo (NCM) - A form used to document a non-conforming event. An analyst must document a non-conformance memo when a non-conforming event occurs. A non-conforming event may include the reporting of analytical data outside of method or SOP criteria, or when there is a deviation from a written policy or procedure. Information in an NCM may be used by project managers to flag data in the report narrative, or by the quality department to track trends and initiate corrective actions, where applicable. Additional information on the NCM policy and procedure is located in the *Complaints and Non-conformances* SOP [QA SOP ME001BO].

5.0 Health and Safety

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

To ensure safe operation, analysts must adhere to safety notices and labels located on the equipment utilized during the procedures outlined in this procedure.

UV radiation is released when the spray changer is disassembled. UV radiation may lead to severe eye injury or blindness. Do not disassemble the spray chamber when the plasma is still on. Do not operate the system when the plasma door window is damaged. Never look directly at the plasma.

The RF generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

6.0 Sample Collection, Preservation, Holding Time, and Storage

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory performs samples collection for samples to be analyzed by this SOP in accordance with the *Field Services* SOP [FS SOP ME001BS]. Refer to this SOP for these instructions.

The laboratory will provide containers for the collection of samples upon client request for analytical services. Bottle kits are prepared in accordance with the *Sample Container Shipping* SOP [AD SOP ME001DS].

Requirements for container type, preservation, and field quality control (QC) for the common list of test methods offered by Pace are listed in the *Pace-WCOL Analytical Methods List* [ME002BS].

General Requirements

Matrix	Routine Container	Min. Sample Amount[1]	Preservation	Holding Time
Aqueous	250 mL	100 mL	Thermal: NA Chemical: HNO3	Collection to Analysis: 180 days
Non-Aqueous (Waste)	2 oz glass teflon-lined lid	10 g	Thermal: NA Chemical: None	Collection to Analysis: 180 days
Solid	2 oz glass teflon-lined lid	10 g	Thermal: NA Chemical: None	Collection to Analysis: 180 days

[1]Minimum amount needed for each discrete analysis.

Aqueous samples are preserved with nitric acid to a pH of < 2 and may be stored in either plastic or glass. If boron or silica is to be determined, plastic containers are preferred. Refrigeration is not required.

Dissolved samples must be filtered through a 0.45 µm pore diameter membrane filter at the time of collection or as soon thereafter as possible. Acidify the filtrate with (1:1) nitric acid immediately following filtration to pH <2.

For total recoverable elements, preservation may be added at time of collection, or, upon receipt at the laboratory. The time allowed for traveling to the laboratory should not to exceed two weeks from the sample collection date. Following acidification at the laboratory, the sample is mixed, held for 24 hours, and then verified to be pH <2 just before analysis or prep (if prep required). If pH is not <2, add more acid and hold for another 24 hours until verified to be pH <2.

Soil samples do not require chemical preservation but must be stored at 4 ± 2°C until the time of preparation.

Additional volume is required for MS/MSD and field duplicate, if requested. A full routine container is sufficient volume to analyze field and matrix QC.

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with the *Sample Receiving* SOP [AD SOP ME0013H]. Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are stored at room temperature until sample preparation. Prepared samples (extracts, digestates, distillates, other) are stored at room temperature until sample analysis.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 28 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

7.0 Equipment and Supplies

NOTE: Refer to the *Major Operational Equipment List* [QA Control Log ME001PM] for specific details regarding the equipment utilized during this procedure.

7.1 Equipment

7.1.1 Inductively Coupled Plasma Atomic Emission Spectrometer equipped with autosampler, chiller and background correction.

7.1.2 ICPs currently in use (or equivalent)

Instrument	Manufacturer	Model
Inductively Coupled Plasma (ICP4)	Thermo Scientific	iCAP6500
Inductively Coupled Plasma (ICP5)	Themo Scientific	iCAP7600

7.1.2.1 ICP software information can be found in the *Major Operational Equipment List* [QA Control Lot ME001PM].

7.1.3 Autosampler - Teledyne Cetac Technologies ASX-520 or equivalent

7.1.4 Chiller – Thermofisher Scientific Thermoflex 2500 or equivalent

7.1.5 Radio frequency generator.

7.1.6 Argon gas supply, welding grade or equivalent.

7.1.7 Coolflow or appropriate water-cooling device.

7.1.8 Peristaltic pump.

7.1.9 Calibrated automatic pipettes with appropriate pipette tips.

7.2 Supplies

7.2.1 Filter paper – Whatman No. 41 or Environmental Express push FilterMate or equivalent.

7.2.2 Class A volumetric flasks.

7.2.3 Autosampler tubes.

8.0 Reagents and Standards

NOTE: Other volumes of standards and reagents may be prepared to account for expected usage. As long as all ratios are kept constant, this is not considered a deviation from the approved procedure.

NOTE: All stored reagents and standards must be labeled as required by the Preparation and *Documentation of Laboratory Standards and Reagents* SOP [QA SOP ME001HG], the *Contingency and Emergency Preparedness Plan* [HS SOP ME0012D], the *Safety Manual* [Corp Manual COR-MAN-HSE], and the *Laboratory Quality Manual* [QAMP ME0012K].

NOTE: All standards and reagents are prepared using reagent water unless otherwise noted.

8.1 Reagents

8.1.1 Concentrated nitric acid (HNO_3), trace metal grade or better.

8.1.2 Concentrated hydrochloric acid (HCl), trace metal grade or better.

8.1.3 Reagent water – A series of in-house deionized (DI) tanks is employed to purify the incoming water. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks. See the *Deionized Water System* SOP [QA SOP ME0012S] for further information.

8.2 Standards

8.2.1 Stock standards are purchased as custom multi-element mixes or as single-element solutions. All standards must be stored in FEP fluorocarbon or unused polyethylene or polypropylene bottles. Standard solutions must be replaced prior to the expiration date provided by the manufacturer. If the expiration date is not provided by the manufacturer or vendor, then the expiration date defaults to 5-years after the material receipt date unless verification from an independent source indicates a problem (see the *Procurement* SOP ME0015U for more information). Expiration dates can be extended provided that the acceptance criteria described in laboratory-specific SOPs are met.

8.2.2 Working standard solutions (refer to Tables II-V) may be used for up to 3 months and must be replaced sooner if verification from an independent source indicates a problem. Standards are to be prepared in a matrix of 5% hydrochloric acid and 5% nitric acid.

8.2.3 Internal standard solution: Scandium (Sc) at 10 mg/L. The concentration may differ between instruments and may be adjusted depending upon the intensity of the internal standard lines. Lithium (2%) may be added to the internal standard solution to act as an ionization buffer

9.0 Procedure

9.1 Equipment Preparation

9.1.1 Instrument

9.1.1.1 Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required). While the instrument is warming up, both the internal standard probe and autosampler probe are stored in a rinse solution. If the torch has been removed, cleaned, or replaced, then a torch alignment must be performed prior to analysis. A 2 ppm solution of zinc is used for alignment of the torch.

9.2 Initial Calibration

9.2.1 Optimize and calibrate the instrument according to the instrument manufacturer's recommended procedures. Flush the system with the calibration blank between each standard or as the manufacturer recommends. Refer to the ICP instrument manual for detailed set up and operation protocols.

9.2.2 Calibration Design

9.2.2.1 PAS-WCOL uses a linear, multi-point calibration curve (refer to Table III for calibration standard concentrations). A minimum of five standards are required, and the correlation coefficient must be > 0.995 (or the coefficient of determination (r^2) must be > 0.990). Results for any analyte that do not meet this criterion on any given analytical run may not be reported.

9.2.3 Calibration Sequence

9.2.3.1 Calibration standards are generally analyzed in sequence from lowest to highest concentration to minimize the chance of carryover from a higher concentration standard.

9.2.4 ICAL Evaluation

9.2.4.1 The concentration of the lowest calibration standard must be at or below the LOQ concentration and must quantitate (%D) to within 80-120% of the true value. If the low-level readback (%D) fails criteria, the cause needs to be determined and the instrument recalibrated.

9.2.4.2 The mid- level calibration standard should quantitate to within 90-110% of the true value. If the mid-level standard fails criteria, re-calibration may be deemed necessary based on analyst judgement.

9.2.4.3 Instrument calibration must be performed daily, and the curve verified with an ICV, ICB, low-level calibration standard readback (%D), and the mid-level readback (%D). Samples may be analyzed continuously for periods exceeding 24 hours as long as all calibration verification (ICV, ICB), continuing calibration verification

(CCV, CCB), and interference check (ICSA) QC criteria are met. The instrument standardization date and time must be included in the raw data.

9.2.4.4 Refer to Section 11 for calibration verification procedures, acceptance criteria, and corresponding corrective actions.

9.2.4.5 Relative Standard Error (%RSE) is evaluated for acceptability of the curve and recovery must be ≤20%. If this criterion is not met, re-prepare the standards and recalibrate.

9.3 Analysis

9.3.1 Sludges, soils, and sediments are weighed to 1.0g and are digested according to ME001J7.

9.3.2 All dissolved samples (groundwater, and wastewater) must be digested according to ME001H8 or ME001IB.

9.3.3 All other samples (groundwater and wastewater) are digested according to ME001H8 or ME001IB.

9.3.4 Three exposures (replicates) are performed for each standard, field sample, and QC sample. The average of the exposures is reported. If the % RSD between the three replicates is > 30 % for sample results > 2 times the LOQ, the sample must be reanalyzed once. Report the result with the lowest % RSD or use analyst judgment to select the most appropriate result.

9.3.5 Prior to calibration and between each sample/standard the system is rinsed with the calibration blank solution. The minimum rinse time between analytical samples is 60 seconds unless by following a protocol outlined in Section 11.3.6, it can be demonstrated that a shorter rinse time may be used. Triton-X can be added to the rinse solution to facilitate the rinse process.

9.3.6 The use of an autosampler for all runs is strongly recommended.

9.3.7 The use of automated QC checks by the instrument software is highly recommended for all calibration verification samples (ICV, CCV), blanks (MB, ICB, CCB), interference check (ICSA), and field samples (linear range) in order to improve or facilitate the data review process.

9.3.8 To facilitate the early identification of QC failures and samples requiring reanalysis, it is strongly recommended that sample data be reviewed periodically during the analytical run.

9.3.9 To facilitate the data review and reporting processes it is strongly recommended that all necessary dilutions be performed on the same analytical run, if feasible.

9.3.10 The use of an internal standard is required on the ICP. The following procedural guidelines must be followed when using an internal standard:

9.3.10.1 Typically used internal standards are yttrium, indium, and/or scandium (Note: Any element may be used that is not typically found in environmental samples at a high rate of occurrence.)

9.3.10.2 The internal standard (IS) must be added to every sample and standard at the same concentration. It is recommended that the IS be added to each analytical sample automatically through use of a third pump channel and mixing coil. Internal standards should be added to blanks, samples, and standards in a like manner, so that dilution effects resulting from the addition may be disregarded.

9.3.10.3 The concentration of the internal standard should be sufficiently high to obtain good precision in the measurement of the IS analyte used for data correction and to minimize the possibility of correction errors if the IS analyte is naturally present in the sample.

9.3.10.4 The internal standard raw intensity counts must be printed on the raw data.

9.3.10.5 The analyst must monitor the response of the internal standard throughout the analytical run. This information is used to detect potential problems and identify possible background contributions from the sample (i.e., natural occurrence of IS analyte).

9.3.10.5.1 Internal standard percent recoveries are considered to be acceptable if the internal standard counts fall within ± 50 % of the counts observed in the calibration blank.

If the internal standard counts in the field samples exceed the acceptance criteria of ± 50%, the field samples must then be diluted and reanalyzed unless instrument drift is suspected.

ICP Internal Standard Associations

Internal Standard Scandium_(Sc227.318), Axial

As, B, Cd, Co, Mo, Ni, Pb, Sb, Se, Si (ICP5 only), Sn, Tl, W, Zn

Internal Standard Scandium (Sc361.384),Radial

Ba, Ca, Fe, K, Mg, Na, Sr

Internal Standard Scandium (Sc361.384),Axial

Ag, Al, Be, Cr, Cu, Mn, Ti, U, V, Si (ICP4 only)

9.3.11 Example Analytical Sequence

- Instrument Calibration

- ICV
- ICB
- ICSA
- CCV
- CCB
- Single element LDR Standard (for IEC adjustments)
- Up to 10 samples (refer to section 9.12 for a definition of sample)
- CCV
- CCB
- Up to 10 samples (see 9.12)
- CCV
- CCB
- Repeat sequence of up to 10 samples (see 9.12) between CCV/CCB pairs as required to complete the sequence

Note: Refer to Quality Control Section 11 for Method 6010D quality control criteria.

9.3.12 Full method required QC must be available for each wavelength used in determining reported analyte results.

9.3.13 Guidelines are provided in the appendices on procedures used to minimize contamination of samples and standards, preventive maintenance and troubleshooting.

9.3.14 All measurements must fall within the defined linear range (i.e. the concentration of the highest calibration standard) where spectral interference correction factors are valid. Dilute and reanalyze all samples for required analytes that exceed the concentration of the highest calibration standard. If an inter-element correction exists for an analyte which exceeds the linear range, the IEC may be inaccurately applied. Therefore, even if an over-range analyte may not be required to be reported for a sample, if that analyte is an interfering element for any requested analyte in that sample, the sample must be diluted. Acid strength must be maintained in the dilution of samples.

9.3.14.1 Dilution of samples (due to the nature of the sample matrix) which results in the reporting of any analytes as non-detect (with an elevated LOQ) require the completion of an NCM. For diluted samples that have any analyte(s) reported as estimated (“J” flagged), and in the absence of results that are non-detect as well, the project manager must be contacted to determine if an NCM is required.

9.3.15 For TCLP samples, full four-point MSA analysis will be required if all of the following conditions are met:

1) recovery of the analyte in the matrix spike is not at least 50%,

2) the concentration of the analyte does not exceed the regulatory level

3) the concentration of the analyte is within 20% of the regulatory level.

9.3.16 The reporting and regulatory limits for TCLP analyses as well as matrix spike levels are detailed in Table V (Appendix A). Appendix G provides guidance on performing MSA analyses

10.0 Data Analysis and Calculations

10.1 Sample results should be reported with up to three significant figures.

10.2 Appropriate factors must be applied to sample values if dilutions are performed

10.3 Calculations

See the *Laboratory Quality Assurance Manual* [QAMP ME0012K] for equations for common calculations.

10.3.1 Relative Standard Error (%RSE):

$$RSE = \sqrt{\sum_{i=1}^{n}\left[\frac{x_i' - x_i}{x_i}\right]^2 \Big/ (n-p)}$$

Where:

x'i = Measured amount of analyte at calibration level i, in mass or concentration units.
xi = True amount of analyte at calibration level i, in mass or concentration units.
p = Number of terms in fitting equation (average = 1, linear = 2)
n= Number of calibration points.

10.3.2 ICV or CCV Percent Recovery:

$$\%\ \text{Recovery} = \frac{X1}{X2} \text{x}\ 100$$

Where:

X1 = observed ICV (or CCV) concentration
X2 = true ICV (or CCV) concentration

10.3.3 LCS Percent Recovery:

$$\%\ \text{Recovery} = \frac{X}{t} \text{x}\ 100$$

Where:

X = observed concentration
t = spike concentration

10.3.4 MS or MSD Percent Recovery:

$$\% \text{ Recovery} = \frac{Xs - X}{t} \text{x } 100$$

Where:

X = observed concentration of unspiked sample
XS = observed concentration of spike sample
t = concentration of added spike

10.3.5 Relative Percent Difference between MS and MSD:

$$\text{RPD} = \frac{|X1 - X2|}{\frac{(X1 + X2)}{2}} \text{x } 100$$

Where:

X1 = the first detected concentration
X2 = the second detected concentration

10.3.6 The final concentration for a digested aqueous sample is calculated as follows:

$$\text{mg} = \frac{C \text{ x } V_1 \text{ x } D}{V_2}$$

Where:

C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V1 = Final volume in liters after sample preparation
V2 = Initial volume of sample digested in liters

10.3.7 The final concentration determined in digested solid samples when reported on a dry weight basis is calculated as follows:

$$\text{mg/kg, dry weight} = \frac{C \text{ x } V \text{ x } D}{W \text{ x } S}$$

Where:

C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V = Final volume in liters after sample preparation
W = Weight in Kg of wet sample digested

S = Percent solids in decimal form

Note: A percent solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. Refer to appropriate SOP for the determination of percent solid. If the results are to be reported on wet weight basis the "S" factor is omitted from the above equation.

10.3.8 The dilution test percent difference for each component is calculated as follows:

% Difference = (S x 5) – I / I x 100

Where:
I = Sample result
S = Dilution test result

10.3.9 Silica calculation from silicon:

Silica = Si + O2

$$mg/L\ SiO_2 = \frac{(mg/L\ Silicon\ from\ raw\ data)(FW\ of\ SiO_2)}{Weight\ of\ Silicon}$$

Where:
FW of SiO2 = 60.0843
Weight of Silicon = 28.0855

11.0 Quality Control and Method Performance

11.1 Quality Control

The following QC samples are prepared and analyzed with each batch of samples. Refer to Appendix B for acceptance criteria and required corrective action.

QC Item	Frequency
Method Blank (MB)	1 per batch
Laboratory Control Sample (LCS)	1 per batch
Matrix Spike (MS)	1 per batch
Matrix Spike Duplicate (MSD)	1 per batch

Dilution Test	1 per batch
Post-digestion Matrix Spike (PDS)	1 per batch

11.2 Instrument QC

The following Instrument QC checks are performed.

QC Item	Frequency
Instrument Detection Limit (IDL)	After initial instrument setup and prior to sample analysis; minimally on an annual basis, after major instrument maintenance, and/or at a frequency designated by the project
Linear Dynamic Range (LDR)	Perform study every 6 months in conjunction with the IEC study
Background Correction Points	Every 6 months in conjunction with the LDR study
Inter-element Corrections (IECs)	Prior to analysis of samples and every 6 months thereafter
Initial Calibration	Daily
Initial Calibration Blank	Daily
Initial Calibration Verification	Daily
Continuing Calibration Blank	After every 10 samples
Continuing Calibration Verification	After every 10 samples
Interference Check Solution Analysis (ICSA)	Beginning of each analytical run
Lower Limit of Quantitation Check (LLOQ)	Quarterly

11.3 Acceptance Criteria and Required Corrective Action

11.3.1 Initial and Continuing Demonstrations of Capability (IDOC and CDOC) – To establish the ability of an analyst to generate acceptable accuracy, each analyst must make a satisfactory initial demonstration of capability prior to using any method and at any time there is a change in instrument type or method. Thereafter, a continuing demonstration of capability is required annually. Refer to the Demonstration of Capability SOP [QA SOP ME001F2] for additional information.

11.3.2 Instrument detection limit (IDL) studies are instrument specific and must be performed before the analysis of samples may begin. IDLs can be estimated as the mean of the blank results plus three times the standard deviation of 10 replicate analyses of the reagent blank solution (use zero for the mean if the mean is negative). Each measurement is performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). IDLs must be established at minimum on an annual basis as well as determined at least once when using new equipment, after major instrument maintenance, and/or at a frequency designated by the project. Each IDL study is recorded in an electronic logbook (ME002HF) and stored in the following location: Metals drive > QA folder > IDL folder > select the appropriate instrument folder > individual IDL study spreadsheets.

11.3.3 Linear Dynamic Range (LDR): The linear range is the concentration above which results cannot be reported without dilution of the sample.

11.3.3.1 For the initial determination of the (LDR), determine the signal responses by analyzing a minimum of three to five different concentration standards (single element standards) across the estimated range for each wavelength that will be utilized on each instrument. One standard for each wavelength should be near the upper limit of the estimated range. The percent recovery of the concentration measured at the LDR must fall within 90-110% from the true value, and this is the concentration at which the IEC factors are based. The LDR study data must be documented and kept on file.

11.3.3.2 Once the LDR is established for each wavelength, an LDR study will be performed every six months in conjunction with the IEC study using single element standards. IEC factors are then based on the concentration of the single element standard for each analyte. The concentration of the single element LDR standard is also the concentration of the highest calibration standard.

11.3.3.3 If the instrument is adjusted in any way that may affect the linear ranges, the linear ranges must be verified and re-established if necessary. The LDR study data must be documented and kept on file.

11.3.4 Background Correction Points – To determine the appropriate location for off-line background correction when establishing methods, the user must scan the area on

either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line inter-element spectral interference or a computer routine must be used for automatic correction on all determinations. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference.

11.3.5 Inter-element Corrections (IECs) – ICP inter-element correction factors must be determined prior to the analysis of samples and every six months thereafter. If the instrument is adjusted in any way that may affect the IEC's, the IEC's must be re-determined. When initially determining IEC's for an instrument, wavelength scans must be performed to ensure that solutions in use are free from contaminants. If an IEC varies significantly from the previously determined IEC then the possibility of contamination should be investigated. The purity of the IEC check solution(s) can be verified by using a standard from a second source or an alternate method (i.e., ICPMS). Published wavelength tables can also be consulted to evaluate the validity of the IEC's. An IEC must be established to compensate for any inter-element interference which results in a false analyte signal greater than +/-½ LOQ. To determine IEC's, analyze a single-element standard at the established linear range. To calculate an IEC, divide the observed concentration of the analyte by the observed concentration of the "interfering element." The IEC calculation can also be determined by the instrument software.

11.3.5.1 The raw data will reflect the effective date (i.e. the date the correction factors are finalized) of the IECs that are determined for each six-month study (for each instrument). For Qtegra software, the effective date will show in the "Template" field.

NOTE: ICP IEC's are more sensitive to small changes in the plasma and instrument setup conditions. Adjustments in the IEC's will be required on a more frequent basis as reflected by the ICSA response. The IEC report from the instrument is generated daily along with the raw data.

11.3.6 Rinse Time Determination – To determine the appropriate rinse time for a particular ICP system, the LDR standard (see Section 11.3.3) should be aspirated as a regular sample followed by the analysis of a series of rinse blanks. The length of time required to reduce the analyte signals to <1/2 LOQ will define the rinse time for a particular ICP system. For some analytes it may be impractical to set the rinse time based on the linear range standard result (i.e., analytes not typically detected in environmental samples at that level and an excessive rinse time would be required at the linear range level). Until the required rinse time is established, the method recommends a rinse period of at least 60 seconds between samples and standards. If a memory effect is suspected, the sample must be reanalyzed after a rinse period of sufficient length. Rinse time studies can be conducted at additional concentration levels. These additional studies must be documented and kept on file, if a concentration other than the linear range level is used

to set the rinse time. The concentration levels used to establish the rinse time must be taken into consideration when reviewing the data.

11.3.7 Method Blank (MB) – One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. For a MB to be acceptable for use with the accompanying samples, the concentration in the blank must not exceed ½ the LOQ.

11.3.7.1 If the MB concentration exceeds ½ the LOQ for an analyte, re-analyze the MB. If still unacceptable, re-preparation and reanalysis of all samples associated with a contaminated method blank is required.

11.3.7.2 If the concentration of method blank exceeds ½ the LOQ for an analyte, but the sample results are non-detect (ND) then results may be reported with the completion of an NCM. For estimated results (J flagged), the results may be NCM'd with approval of the project manager.

11.3.7.3 If the concentration of the MB is less than 10% of the regulatory limit for an analyte or less than 10% of the sample concentration (for an analyte), then sample results may be reported with the completion of an NCM.

11.3.7.4 If the client requests that results be reported with estimated ("J") values and there is a "J" value for an analyte in the MB, then all sample analyses with a positive result for that analyte must be flagged.

11.3.7.5 If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. Such action must result in the completion of a NCM. Refer to Section 14 for more detail.

11.3.8 Laboratory Control Sample (LCS) – One aqueous LCS must be processed with each preparation batch. A Laboratory Control Sample Duplicate (LCSD) must be processed along with the LCS in the absence of an MS/MSD. The LCS must contain all analytes of interest and must be carried through the entire procedure, including preparation and analysis. LCS spike levels are provided in Table II (Appendix A). The control limits are 80-120%. The %RPD acceptance criteria is ≤20% for an LCS/LCSD pair. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable precision guidelines.

11.3.8.1 If any analyte falls outside the control limits, re-analyze the LCS. If results are still unacceptable, then the system can be considered out of control. Re-preparation and reanalysis of all samples associated with the LCS is required unless the following condition is applicable.

11.3.8.2 In the instance where the LCS recovery is greater than the upper control limit and the sample results are non-detect (ND), the data may be reported with the completion of an NCM. . For estimated results (J flagged), the results may be NCM'd with approval of the project manager.

11.3.9 Matrix Spike/Matrix Spike Duplicate (MS/MSD) – One MS/MSD pair must be processed in each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. The MS/MSD should be prepared at the same dilution as the parent sample. Some client specific data quality objectives (DQO's) may require the use of an unspiked sample duplicate (DUP) in place of or in addition to the MS/MSD. The MS/MSD (and/or MS/DUP) results are used to determine the effect of a matrix on the accuracy and precision of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD (or MS/DUP) analysis. Spiking levels are provided in Tables II (Appendix A). The MS/MSD control limits are 75% - 125% for spiked analytes. The RPD for MS/MSD or DUP is20%. MS/MSD recoveries that fall outside the control limits may be addressed in the report narrative.

11.3.9.1 If the percent recovery falls outside the acceptance range for any analyte, interference tests are performed. These tests include a dilution test and, if applicable, a post- digestion spike (PDS) as described in the following sub-sections. However, a dilution test must still be performed on at least one sample in a prep batch if the prep batch does not contain an MS/MSD or MS/DUP.

11.3.9.1.1 Dilution Test – A dilution test must be performed on at least one sample in every prep batch to determine whether significant physical or chemical interferences exist due to the sample matrix. The test is performed by analyzing a sample (typically the same sample as was used for the MS/MSD or MS/DUP, but another sample must be used in the absence of an MS/MSD or MS/DUP) at a 5x dilution. Samples identified as field blanks cannot be used for dilution tests. The results of the diluted samples, after correction for dilution, should agree within 20% of the original sample determination when the original sample concentration is greater than 25x the LOQ. If the results are not within 20%, the possibility of chemical or physical interference exists. Elements that fail the dilution test are reported as estimated values.

11.3.9.1.2 Post Digestion Spike (PDS) – The test only needs to be performed for the specific elements that fell outside MS and/or MSD acceptance limits, and only if the spike concentration added was greater than the concentration determined in the un-spiked sample. An aliquot, or dilution thereof, should

be obtained from the un-spiked (i.e. parent) sample, and spiked with a known quantity of target elements. Typically the parent sample is spiked with an aliquot of standard that is identical to the spike concentration of the LCS/MS/MSD. The recovery of the post-digestion spike should fall within 75-125% recovery. If the post-digestion spike recovery fails to meet the acceptance criteria, the sample results must be reported as estimated values. Another sample from the same preparation batch can be used as an alternative if the parent sample of the MS/MSD cannot be used. If both the MS/MSD and the post digestion spike fail, then matrix interference can be suspected.

11.3.9.2 The concentration of an analyte in the parent sample must fall within the linear range (LDR standard or highest calibration standard), but the MS/MSD is not required to be within range as long as the parent sample and the MS/MSD were prepared at the same dilution.

11.3.10 Initial Calibration Verification (ICV) –Calibration accuracy is verified by analyzing a mid-level second source standard. The ICV must fall within 10% of the true value for that solution with a relative standard deviation (%RSD) < 5% (minimum of three replicates). On a daily basis (i.e. each time the instrument is calibrated), the ICV stock standard is used to prepare the working ICV standard, which expires in 24 hours. If the ICV fails to meet criteria, proceed as follows:

11.3.10.1 In the instance where the ICV recovery is greater than the upper control limit and the sample results are non-detect (ND),then the data may be reported with the completion of an NCM. For estimated results (J flagged), the results may be NCM'd with approval of the project manager.

11.3.10.2 If the ICV fails criteria, it may be immediately reanalyzed one time. If the ICV is reanalyzed, then only analytes which pass criteria in the reanalyzed ICV may be reported (i.e. analytes in the original ICV analysis may not be reported even if it meets criteria for some analytes that may have failed in the reanalyzed ICV). If the ICV is reanalyzed for a single analyte only and passes criteria, then the initial ICV may be used for all passing analytes, and the reanalyzed single analyte ICV may be used for the analyte of interest if it passes criteria.

11.3.10.3 When the ICV does not meet acceptance criteria, then the cause must be determined, the instrument must be recalibrated, and the calibration must be re-verified.

11.3.11 Initial Calibration Blank (ICB) - An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must not exceed ½ the LOQ (see appendices for state or program-specific criteria). If the ICB fails to meet criteria for an analyte that is required to be reported on the analytical run, the analysis should be terminated, the problem corrected, the instrument re-calibrated, and the calibration verified. If the ICB fails to meet acceptance criteria, proceed as follows:

11.3.11.1 If the concentration of the ICB is less than 10% of the regulatory limit for an analyte or less than 10% of the sample concentration (for an analyte), then sample results may be reported with the completion of an NCM.

11.3.12 Continuing Calibration Verification/Continuing Calibration Blank (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard and instrument blank after every 10 (or fewer) samples. In this context, the term “samples” includes MBs, LCSs, field samples, field QC samples, MS/MSDs, sample duplicates, dilutions of samples, and samples for reanalysis. The CCV is a mid-range standard. The CCV must fall within 10% of the true value for that solution with relative standard deviation < 10% (minimum of three replicates). A CCB is analyzed immediately following the CCV. The CCB result must not exceed the LOQ (however, see appendices for state or program-specific requirements). Sample results may only be reported when bracketed by valid CCV/CCB pairs. If a CCV and/or CCB are outside acceptance criteria, the samples associated with the CCV/CCB cannot be reported and must be reanalyzed unless the following conditions apply:

11.3.12.1 In the instance where the CCV recoveries are greater than the upper control limit and the sample results are non-detect (ND), then the data may be reported with the completion of an NCM. For estimated results (J flagged), the results may be NCM’d with approval of the project manager.

11.3.12.2 In the instance where the CCB concentration for an analyte is greater than the LOQ and the sample results are non-detect (ND), then the data may be reported with the completion of an NCM. For estimated results (J flagged), the results may be NCM’d with approval of the project manager.

11.3.12.3 If the concentration of the CCB is less than 10% of the regulatory limit for an analyte or less than 10% of the sample concentration (for an analyte), then sample results may be reported with the completion of an NCM.

11.3.13 Interference Check Solution Analysis (ICSA) – The validity of the inter-element correction factors (IECs) is demonstrated through the successful analysis of an interference check solution (ICSA). The ICSA contains interfering (i.e. spiked) elements (Al, Ca, Fe, and Mg) and non-interfering elements (non-spiked). The composition of the ICSA can be found in Table V (Appendix A). Custom multi-elemental stock standards may be used to prepare the working ICSA solution. If the ICP displays over-correction as a negative number, then the non-interfering elements can be controlled from the ICSA (i.e. a negative number will be detected as a negative number, not falsely as zero).

11.3.13.1 The ICSA solution must be analyzed at the beginning of the analytical run (prior to analysis of samples). Acceptance criteria for the interfering elements (Al, Ca, Fe, and Mg) must fall within +/- 20% of the true value. Results may not be reported for any interfering (i.e. spiked) element that does not meet this criteria,

as well as for any non-interfering element(s) for which there is spectral interference caused by the spiked element.

11.3.13.2 ICSA results for the non-interfering elements must fall within +/- LOQ (however, see appendices for state or program-specific criteria). Correction factors that are applied to non-interfering elements which are affected by Al, Ca, Fe, or Mg (i.e. the interfering spiked elements) may be adjusted in order to meet acceptance criteria. This can be accomplished by analyzing a single-element standard of the interfering element and calculating the correction factor as described in section 11.3.5. This value is then added to the existing correction factor to produce a new (adjusted) IEC value. Under no circumstances can correction factors be created on a routine analytical run in order to make a non-interfering element pass criteria (i.e. only those elements that are affected by Al, Ca, Fe, and Mg may be adjusted, and only if the single-element standard(s) was/were analyzed on the same analytical run). If the ICSA results for the non-interfering elements do not meet +/- LOQ (or see appendices for state or program-specific criteria), then sample data must be evaluated as follows:

11.3.13.2.1 If the non-interfering element concentration in the ICSA is the result of contamination versus a spectral interference, and this reason is documented, the field sample data can be accepted.

11.3.13.2.2 If the affected element was not required to be reported then the sample data can be accepted

11.3.14 Lower Limit of Quantitation Check Sample (LLOQ) - The laboratory must establish the LLOQ as the lowest point of quantitation which, in most cases, is the lowest concentration in the calibration curve. The LLOQ is initially verified by the analysis of at least 7 replicate samples, spiked at the LLOQ and processed through all preparation and analysis steps of the method. The mean recovery and relative standard deviation of these samples provide an initial statement of precision and accuracy at the LLOQ. In most cases the mean recovery must be +/- 35% of the true value and RSD must be < 20%. In-house limits may be calculated when sufficient data points exist. Monitoring recovery of LLOQ over time is useful for assessing precision and bias. Ongoing LLOQ verification, at a minimum, is on a quarterly basis to validate quantitation capability at low analyte concentration levels.

11.3.15 Method of Standard Addition (MSA) – This technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample interference that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences, which cause a baseline shift. Refer to Section 9.3.15 for additional information on when MSA is required as well as Appendix G for specific MSA requirements.

11.4 Method Performance

11.4.1 Method Validation

11.4.1.1 Detection Limits

Detection limits (DL) and limits of quantitation (LOQ) are established at initial method setup and verified on an on-going basis thereafter. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 Method Validation and Instrument Verification and to the Method Validation SOP [QA Policy ME003BF] for these procedures.

11.5 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to the *Demonstration of Capability* SOP [QA SOP ME001F2] for more information.

12.0 Data Review and Corrective Action

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employee's complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to the *Data Review* SOP [QA SOP ME003LP] for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

13.0 Pollution Prevention

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

14.0 Modifications

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 Method Validation and Instrument Verification for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

For compliance samples, all procedures outlined in this SOP must be followed without exception. In the event that a deviation from this SOP cannot be avoided (i.e., demonstrated and uncorrectable matrix interference, non-compatible matrix, insufficient sample amount due to client or laboratory error) it is imperative that an NCM is completed that clearly documents the anomaly. This anomaly must be communicated to the client such that corrective action activity including sample recollection can be performed. Based upon the type and severity of the anomaly, the results may not be appropriate for compliance reporting. Discussions between the client, the appropriate regulatory authority, and the laboratory will be necessary to resolve certain anomalies. In any case, it is essential that any anomaly be documented with an NCM.

15.0 Responsibilities

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 Attachments

16.1 Appendix A: Tables

16.1.1 Table I: Method 6010D ICP Target Analyte List

16.1.2 Table II: Laboratory Control Sample and Matrix Spike Levels

16.1.3 Table III: Trace ICP Calibration Standards

16.1.4 Table IV: ICP Calibration Verification Standards

16.1.5 Table V: Interference Check Sample Concentrations

16.1.6 Table VI: Linear Range (LDR) Standard Concentrations

16.1.7 Table VII: TCLP LOQs, Regulatory Limits, and Matrix Spike Levels

16.2 Appendix B: ICP Data Review Checklist

16.3 Appendix C: Troubleshooting Guide

16.4 Appendix D: Contamination Control Guidelines

16.5 Appendix E: Preventative Maintenance

16.6 Appendix F: Cross-Reference of Terms Used in Methods 6010D and PAS-WCOL

16.7 Appendix G: Method of Standard Addition (MSA) Guidance

16.8 Appendix H: DoD QSM Requirements

16.8.1 Table I-DoD: DoD Method 6010D ICP Target Analyte List

16.8.2 Table II-DoD: DoD LCS/MS Control Limits for ICP

16.9 Appendix I: North Carolina QC Requirements

17.0 References

NOTE: Where references exclude a date or edition, the latest edition of the referenced document adopted/recognized by the laboratory's accreditation bodies applies. Refer to the *Laboratory Quality Manual* [QAMP ME0012K] for details.

17.1 *Consolidated Quality System Manual (QSM) for Environmental Laboratories*. Department of Defense (DoD) / Department of Energy (DoE).

17.2 *General Requirements for the Competence of Testing and Calibration Laboratories*. International Standard ISO/IEC 17025.

17.3 *Laboratory Accreditation Standards*. TNI Standard. The NELAC Institute.

17.4 40 CFR Part 136, Appendix B, *Definition and Procedure for the Determination of the Method Detection Limit Revision 2*.

17.5 SW-846 Update V, Test Method for Evaluating Solid Waste, Third Edition – *Inductively Coupled Plasma-Atomic Emission Spectrometry*, Method 6010D, Revision 4, July 2014

17.6 EPA/600/R-94/111 – *Methods for the Determination of Metals in Environmental Samples* – Supplement I, May 1994.

17.7 DoD/DOE Quality Systems Manual (QSM) for Environmental Laboratories.

17.8 Laboratory Quality Manual(QAMP) - ME0012K.

18.0 Revision History

Revision #	Revision Date	Section Modified	Modification	Reason for Modification
-08	**10/14/2021**	All	Re-wrote and re-formatted entire document	Compliance with Pace policy
		9.2.2	Changed from three to five standards	Match current practice
		9.3.2	Removed reference to ME001IF	Obsolete document
		9.3.10.5.3	Updated IS associations	Match current practice
		11.3.3	Updated LDR study information	Match current practice
		11.3.6	Changed < LOQ to < ½ LOQ	Incorrect in previous version
		11.3.7.2	Clarified ND and J flag detections	Clarification
		11.3.8	Added LCSD when no MS/MSD and clarified ND and J flag detections	Clarification
		11.3.9	Added MS/MSD or MS/DUP information	Clarification
		11.3.11.1 & 11.3.12.3	Added results may be reported with NCM if detection in blank is less than 10% of the detection.	Clarification
		Table II	Updated TCLP Spike for Strontium	Match current practice
		Table III	Replaced table	Match current practice
		Table VI	Replaced table	Match current practice

Appendix A: Tables

Table I: Method 6010D ICP Target Analyte List

ELEMENT	Symbol	CAS #	LOQ (mg/L) Aqueous	LOQ (mg/kg) Soil
Aluminum	Al	7429-90-5	0.400	20
Antimony	Sb	7440-36-0	0.020	1.0
Arsenic	As	7440-38-2	0.015	0.75
Barium	Ba	7440-39-3	0.025	1.3
Beryllium	Be	7440-41-7	0.005	0.25
Boron	B	7440-42-8	0.050	2.5
Cadmium	Cd	7440-43-9	0.005	0.25
Calcium	Ca	7440-70-2	5.0	250
Chromium	Cr	7440-47-3	0.010	0.5
Cobalt	Co	7440-48-4	0.025	1.3
Copper	Cu	7440-50-8	0.010	0.5
Iron	Fe	7439-89-6	0.100	5.0
Lead	Pb	7439-92-1	0.010	0.50
Magnesium	Mg	7439-95-4	5	250
Manganese	Mn	7439-96-5	0.015	0.75
Molybdenum	Mo	7439-98-7	0.040	2.0
Nickel	Ni	7440-02-0	0.040	2.0
Potassium	K	7440-09-7	5.0	250
Selenium	Se	7782-49-2	0.020	1.0
Silicon	Si	7440-21-3	0.5	25
Silver	Ag	7440-22-4	0.010	0.5
Sodium	Na	7440-23-5	5.0	250
Strontium	Sr	7440-24-6	0.010	1.0
Thallium	Tl	7440-28-0	0.050	2.5
Tin	Sn	7440-31-5	0.050	5.0
Titanium	Ti	7440-32-6	0.050	2.5
Tungsten	W	7440-33-7	0.500	25
Uranium	U	7440-06-11	0.500	25
Vanadium	V	7440-62-2	0.050	2.5
Zinc	Zn	7440-66-6	0.020	2.5

Table II: Laboratory Control Sample and Matrix Spiking Levels

ELEMENT	AQUEOUS	SOLID	TCLP
	LCS/Matrix Spike (mg/L)	LCS/Matrix Spike (mg/kg)	LCS/Matrix Spike (mg/L)
Aluminum	20	1000	20
Antimony	0.40	50	1.0
Arsenic	0.40	250	5.0
Barium	2.0	500	10.0
Beryllium	2.0	100	2.0
Boron	0.40	50	1.0
Cadmium	0.40	50	1.0
Calcium	40	2000	40
Chromium	2.0	250	5.0
Cobalt	2.0	100	2.0
Copper	2.0	100	2.0
Iron	20	1000	20
Lead	0.40	250	5.0
Magnesium	40	2000	40
Manganese	2.0	100	2.0
Molybdenum	2.0	100	2.0
Nickel	2.0	100	2.0
Potassium	40	2000	40
Selenium	0.40	50	1.0
Silicon	2.0	100	2.0
Silver	0.40	50	1.0
Sodium	40	2000	40
Strontium	1.0	50	1.0
Thallium	0.80	40	0.8
Tin	0.40	50	1.0
Titanium	0.40	50	1.0
Uranium	2.0	100	1.0
Vanadium	2.0	100	2.0
Tungsten	2.0	100	2.0
Zinc	2.0	100	2.0

Note: The spike concentrations are subject to change.

Table III: Trace ICP Calibration Standard Concentrations

Note: The concentrations in this table are subject to change.

	S01	S02	S03	S04	S05	S06	S07	S08	S09
Ag 328.068 {103} (Axial)	0.0100 ppm	0.0500 ppm	0.0900 ppm	0.1800 ppm	0.3000 ppm	0.4500 ppm	0.6000 ppm	N/A	N/A
Al 308.215 {109} (Axial)	0.4000 ppm	2.0000 ppm	45.0000 ppm	90.0000 ppm	150.0000 ppm	225.0000 ppm	300.0000 ppm	5.0000 ppm	20.0000 ppm
As 189.042 {478} (Axial)	0.0150 ppm	0.0750 ppm	0.7500 ppm	1.5000 ppm	2.5000 ppm	3.7500 ppm	5.0000 ppm	N/A	N/A
B 208.959 {461} (Axial)	0.0500 ppm	0.2500 ppm	0.3000 ppm	0.6000 ppm	1.0000 ppm	1.5000 ppm	2.0000 ppm	N/A	N/A
Ba 455.403 {74} (Radial)	0.0250 ppm	0.1250 ppm	1.5000 ppm	3.0000 ppm	5.0000 ppm	7.5000 ppm	10.0000 ppm	N/A	N/A
Be 313.042 {108} (Axial)	0.0050 ppm	0.0250 ppm	0.3000 ppm	0.6000 ppm	1.0000 ppm	1.5000 ppm	2.0000 ppm	N/A	N/A
Ca 317.933 {106} (Radial)	5.0000 ppm	25.0000 ppm	45.0000 ppm	90.0000 ppm	150.0000 ppm	225.0000 ppm	300.0000 ppm	N/A	N/A
Cd 228.802 {447} (Axial)	0.0050 ppm	0.0250 ppm	0.3000 ppm	0.6000 ppm	1.0000 ppm	1.5000 ppm	2.0000 ppm	N/A	N/A
Cr 267.716 {126} (Axial)	0.0100 ppm	0.0500 ppm	0.7500 ppm	1.5000 ppm	2.5000 ppm	3.7500 ppm	5.0000 ppm	N/A	N/A
Cu 327.396 {103} (Axial)	0.0100 ppm	0.0500 ppm	0.7000 ppm	1.5000 ppm	2.5000 ppm	3.7500 ppm	5.0000 ppm	N/A	N/A
Fe 261.187 {129} (Radial)	0.1000 ppm	0.5000 ppm	45.0000 ppm	90.0000 ppm	150.0000 ppm	225.0000 ppm	300.0000 ppm	5.0000 ppm	20.0000 ppm
K 766.490 {44} (Radial)	5.0000 ppm	25.0000 ppm	30.0000 ppm	60.0000 ppm	100.0000 ppm	150.0000 ppm	200.0000 ppm	N/A	N/A
Mg 279.079 {121} (Radial)	5.0000 ppm	25.0000 ppm	45.0000 ppm	90.0000 ppm	150.0000 ppm	225.0000 ppm	300.0000 ppm	N/A	N/A
Mn 257.610 {131} (Axial)	0.0150 ppm	0.0750 ppm	1.5000 ppm	3.0000 ppm	5.0000 ppm	7.5000 ppm	10.0000 ppm	N/A	N/A
Mo 202.030 {467} (Axial)	0.0400 ppm	0.2000 ppm	0.3000 ppm	0.6000 ppm	1.0000 ppm	1.5000 ppm	2.0000 ppm	N/A	N/A
Na 589.592 {57} (Radial)	5.0000 ppm	25.0000 ppm	30.0000 ppm	60.0000 ppm	100.0000 ppm	150.0000 ppm	200.0000 ppm	N/A	N/A
Ni 231.604 {445} (Axial)	0.0400 ppm	0.2000 ppm	0.7500 ppm	1.5000 ppm	2.5000 ppm	3.7500 ppm	5.0000 ppm	N/A	N/A
Pb 220.353 {453} (Axial)	0.0100 ppm	0.0500 ppm	0.7500 ppm	1.5000 ppm	2.5000 ppm	3.7500 ppm	5.0000 ppm	N/A	N/A
Sb 206.833 {463} (Axial)	0.0200 ppm	0.1000 ppm	0.3000 ppm	0.6000 ppm	1.0000 ppm	1.5000 ppm	2.0000 ppm	N/A	N/A
Se 196.090 {472} (Axial)	0.0200 ppm	0.1000 ppm	0.3000 ppm	0.6000 ppm	1.0000 ppm	1.5000 ppm	2.0000 ppm	N/A	N/A
Sn 189.989 {477} (Axial)	0.0500 ppm	0.2500 ppm	0.3000 ppm	0.6000 ppm	1.0000 ppm	1.5000 ppm	2.0000 ppm	N/A	N/A
Ti 337.280 {100} (Axial)	0.0500 ppm	0.2500 ppm	0.3000 ppm	0.6000 ppm	1.0000 ppm	1.5000 ppm	2.0000 ppm	N/A	N/A
Tl 190.856 {476} (Axial)	0.0500 ppm	0.2500 ppm	0.3000 ppm	0.6000 ppm	1.0000 ppm	1.5000 ppm	2.0000 ppm	N/A	N/A
V 292.402 {115} (Axial)	0.0500 ppm	0.2500 ppm	0.3000 ppm	0.6000 ppm	1.0000 ppm	1.5000 ppm	2.0000 ppm	N/A	N/A
Zn 206.200 {463} (Axial)	0.0200 ppm	0.1000 ppm	1.5000 ppm	3.0000 ppm	5.0000 ppm	7.5000 ppm	10.0000 ppm	N/A	N/A
Si 251.611 {134} (Axial)	0.5000 ppm	N/A	N/A	1.5000 ppm	2.5000 ppm	3.7500 ppm	5.0000 ppm	N/A	N/A

Sr 421.552 {80} (Radial)	0.0100 ppm	0.0500 ppm	0.7500 ppm	1.5000 ppm	2.5000 ppm	3.7500 ppm	5.0000 ppm	N/A	N/A
W 224.875 {450} (Axial)	0.5000 ppm	N/A	N/A	1.5000 ppm	2.5000 ppm	3.7500 ppm	5.0000 ppm	N/A	N/A
U 409.014 {82} (Axial)	0.5000 ppm	N/A	N/A	1.5000 ppm	2.5000 ppm	3.7500 ppm	5.0000 ppm	N/A	N/A
Co 228.616 {447} (Axial)	0.0250 ppm	0.1250 ppm	0.3000 ppm	0.6000 ppm	1.0000 ppm	1.5000 ppm	2.0000 ppm	N/A	N/A

Table IV: ICP Calibration Verification Standards

ELEMENT	ICV (mg/L)	CCV (mg/L)
Aluminum	25	25
Antimony	0.50	0.50
Arsenic	0.50	0.50
Barium	2.5	2.5
Beryllium	2.5	2.5
Boron	0.50	0.50
Cadmium	0.50	0.50
Calcium	50	50
Chromium	2.5	2.5
Cobalt	2.5	2.5
Copper	2.5	2.5
Iron	25	25
Lead	0.50	0.50
Magnesium	50	50
Manganese	2.5	2.5
Molybdenum	2.5	2.5
Nickel	2.5	2.5
Potassium	50	50
Selenium	0.50	0.50
Silicon	2.5	2.5
Silver	0.50	0.50
Sodium	50	50
Strontium	2.5	2.5
Thallium	1.0	1.0
Tin	0.50	0.50
Titanium	0.50	0.50
Tungsten	2.5	2.5
Uranium	2.5	2.5
Vanadium	2.5	2.5
Zinc	2.5	2.5

Note: The concentrations are subject to change as long as the values are approximately mid-range of the calibration.

Table V: Interference Check Sample Concentrations

ELEMENT	SIC (ICSA) (mg/L)
Aluminum	300
Calcium	300
Iron	300
Magnesium	300

Table VII: TCLP LOQs, Regulatory Limits, and Matrix Spike Levels

ELEMENT	LOQ's (mg/L)	Regulatory Limit (mg/L)	Spike Level (mg/L)
Arsenic	0.15	5.0	5.0
Barium	0.25	100	10
Cadmium	0.050	1.0	1.0
Chromium	0.10	5.0	5.0
Lead	0.10	5.0	5.0
Selenium	0.20	1.0	1.0
Silver	0.10	5.0	1.0

Appendix B: ICP Data Review Checklist

Area of Review
• Analyzed per proper Method/SOP
• Analyzed within holding time
• Initial calibration within acceptance criteria.
• ICV/CCV criteria met
• Low level readback or LLICV/ LLOQ criteria met
• ICB/CCB criteria met
• CCV/CCB analyzed at the correct frequency and within acceptance criteria.
• ICSA met criteria
• Analysis within linear range
• LCS criteria met
• MB criteria met
• MS/MSD Recovery criteria met
• MS/MSD RPD criteria met
• *%RSD criteria met for samples
• Post digestion performed if required.
• Serial dilution performed if required.
• NCM generated if applicable

*Required by PAS-WCOL only, not a method requirement

Appendix C: Troubleshooting Guide

PROBLEM	POSSIBLE CAUSE/ SOLUTION
High Blanks	Increase rinse time. Clean or replace tip. Clean or replace torch. Replace sample tubing. Clean or replace nebulizer. Clean or replace mixing chamber.
Instrument Drift	RF not cooling properly. Replace torch (crack). Clean or replace nebulizer (blockage). Check room temperature (changing). Replace pump tubing. Room humidity is too high. Clean torch tip (salt buildup). Check for argon leaks.
Erratic Readings, Flickering Torch or High RSD	Check for argon leaks. Adjust sample carrier gas. Replace tubing (clogged). Check drainage (back pressure changing). Increase uptake time (too short). Increase flush time (too short). Clean nebulizer, torch, or spray chamber. Increase sample volume introduced. Check that autosampler tubes are full. Sample or dilution of sample not mixed. Increase integration time (too short). Realign torch. Reduce amount of tubing connectors.
Standards reading twice normal absorbance or concentration.	Incorrect standard used. Incorrect dilution performed.

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Inductively Coupled Plasma - Atomic Emission Spectroscopy
METHOD: Method 6010D
ISSUER: Pace ENV - Local Quality - WCOL

Appendix D: Contamination Control Guidelines

The following procedures are strongly recommended to prevent contamination:

- All work areas used to prepare standards and spikes need to be cleaned before and after each use.
- All glassware must be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.
- Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.
- Powdered gloves must not be used in the metals laboratory since the powder contains silica and zinc as well as other metallic analytes. Only Non-powered, vinyl, or nitrile gloves are to be used in the metals laboratory.
- Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

The following are helpful hints in the identification of the source of contaminants:

- Yellow pipette tips and volumetric caps can sometimes contain cadmium.
- Some sample cups have been found to contain lead.
- The marking on glass beakers have been found to contain lead.
- New glassware especially beakers can be a source of silica and boron.
- Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.
- Improper cleaning of glassware can cause contamination.
- Latex gloves contain over 500 ppb of zinc.

Appendix E: Preventative Maintenance

As part of the PAS-QCOL QAMP, a maintenance schedule has been developed for the ICP. This information is kept in a maintenance log to document any preventive maintenance or servicing to the instruments.

Preventive Maintenance - ICP

ITEM	FREQUENCY
Nebulizer	M
Auto Sampler Cleaning	W
Spray Chamber	M
Radial Bucket	BW
Plasma Torch	W
Vacuum System	SA
Water Filter	SA
Pump Tubing	BW

D = Daily
W = Weekly
BW = Bi-weekly
M = Monthly
SA =Semi Annual (January; July)

NOTE:

- Tubing should be checked daily and changed more often if necessary.
- Torch must be cleaned with aqua regia or a 25% Fluka solution. If multiple torches are being used on an instrument, each torch should be labeled and recorded in the maintenance logbook when it is used.
- Radial view bucket should be soaked in 10% nitric acid and then rinsed with di-water and allowed to air-dry. Care should be taken to not touch the bottom of the radial view bucket.
- Use only Kimwipes on ICP glassware and not paper towels.
- A 2.5% Fluka solution can also be used while ICP is warming up to help clean the sample introduction system.

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Inductively Coupled Plasma - Atomic Emission Spectroscopy
METHOD: Method 6010D
ISSUER: Pace ENV - Local Quality - WCOL

Appendix F: Cross-Reference of Terms Used in Methods 6010D and by PAS-WCOL

SW6010D	PAS-WCOL SOP
Calibration blank (S0)	Initial and continuing calibration blanks (ICB/CCB)
Dilution test	Dilution test
Instrument detection limit (IDL)	Instrument detection limit (IDL)
Continuing calibration verification (CCV)	Continuing calibration verification (CCV)
Internal standard	Internal standard (IS)
Laboratory control sample (LCS)	Laboratory control sample (LCS)
Matrix spike and matrix spike duplicate (MS/MSD)	Matrix spike and matrix spike duplicate (MS/MSD)
Method blank	Method or Prep blank (MB)
Check standard or Initial calibration verification (ICV)	Initial calibration verification (ICV)
Spectral Interference check solution (SIC)	(ICSA)

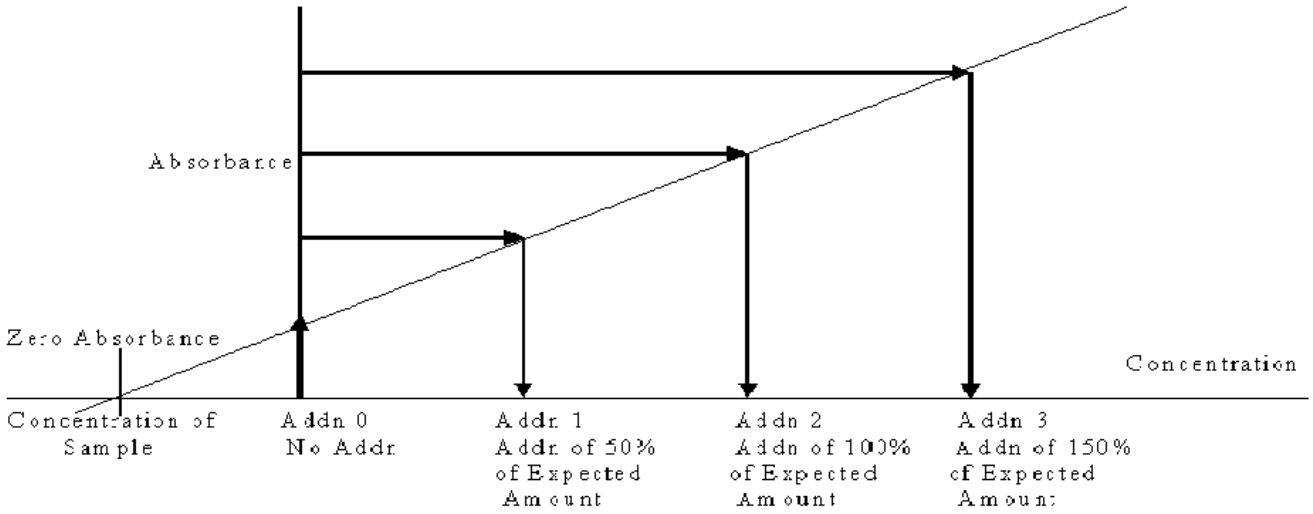
TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Inductively Coupled Plasma - Atomic Emission Spectroscopy
METHOD: Method 6010D
ISSUER: Pace ENV - Local Quality - WCOL

Appendix G: Method of Standard Addition (MSA) Guideline

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standard are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked standard should be the same.

In order to determine the concentration of analyte in the sample, the analytical value of each solution is determined and a plot or linear regression performed. On the vertical axis the analytical value is plotted versus the concentrations of the standards on the horizontal axis. An example plot is shown in figure below. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown.



For the method of standard additions to be correctly applied, the following limitations must be taken into consideration:

- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

Appendix H: DoD QSM Requirements

Sections found in this appendix supersede and/or supplement the existing sections of the SOP. These requirements must be met when analyzing samples for the Department of Defense- as stipulated in the DOD Quality Systems Manual.

Table B-8. Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Linear Dynamic Range (LDR) or high-level check standard	At initial set up and checked every 6 months with a high standard at the upper limit of the range.	Within ± 10% of true value.	Dilute samples within the calibration range or re-establish/ verify the LDR.	Flagging is not appropriate.	Data cannot be reported above the high calibration range without an established/passing high-level check standard.
Initial Calibration (ICAL) for all analytes	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, $r_2 \geq 0.99$.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	Minimum one high standard and a Calibration Blank. No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 10% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

Table B-8. Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All reported analytes within ± 10% of the true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without valid CCVs. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-8. Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Low-Level Calibration Check Standard (LLCCV)	Daily.	All reported analytes within ± 20% of true value.	Correct problem and repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed without a valid Low-Level Calibration Check Standard (LLCCV). LLCCV should be less than or equal to the LOQ. If the concentration of the lowest calibration standard is less than or equal to the LOQ, the lowest standard may be re-quantified against the calibration curve as a LLCCV. Otherwise, a separate standard must be analyzed as LLCCV prior to the analysis of any samples.

Table B-8. Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch.	The absolute values of all analytes must be < ½ LOQ or < 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid Method Blank. Non-detects associated with positive blank infractions may be reported. Sample results >10X the LOQ associated with negative blanks may be reported. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-8. Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial and Continuing Calibration Blank (ICB/CCB)	Immediately after the ICV and immediately after every CCV.	The absolute values of all analytes must be < ½ LOQ or < 1/10th the amount measured in any sample.	ICB: Correct problem and repeat ICV/ICB analysis. If that fails, rerun ICAL. All samples following the last acceptable Calibration Blank must be reanalyzed. CCBs may not be re-analyzed without re-analysis of the associated samples and CCV(s).	Flagging is not appropria te.	Results may not be reported without valid Calibration Blanks. Non-detects associated with positive blank infractions may be reported. Sample results >10X the LOQ associated with negative blanks may be reported. For CCB, failures due to carryover may not require an ICAL.
Interference Check Solutions (ICS) (also called Spectral Interference Checks)	After ICAL and prior to sample analysis.	ICS-A: Absolute value of concentration for all non-spiked project analytes <1/2 LOQ (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within ± 20% of true value.	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the failed ICS.	All analytes must be within the LDR. ICS-AB is not needed if instrument can read negative responses.

Table B-8. Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all reported analytes. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to the source(s) of difference (i.e., matrix effect or analytical error).

Table B-8. Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.
Dilution Test	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within ± 10% of the original measurement.	No specific CA unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	Only applicable for samples with concentrations > 50 x LOQ (prior to dilution). Use along with MS/MSD and PDS data to confirm matrix effects.
Post-Digestion Spike (PDS) Addition (ICP only)	Perform if MS/MSD fails. One per preparatory batch (using the same sample as used for the MS/MSD if	Recovery within 80-120%.	No specific CA unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	Criteria applies for samples with concentrations < 50 X LOQ prior to dilution.
Method of Standard Additions (MSA)	When dilution test or post digestion spike fails and if required by project.	NA.	NA.	NA.	Document use of MSA in the Case Narrative.

Table I-DoD: DoD Method 6010D ICP Target Analyte List

ELEMENT	Symbol	CAS #	LOQ (mg/L) Aqueous	LOQ (mg/kg) Soil
Aluminum	Al	7429-90-5	0.40	20
Antimony	Sb	7440-36-0	0.02	1.0
Arsenic	As	7440-38-2	0.015	0.75
Barium	Ba	7440-39-3	0.025	1.3
Beryllium	Be	7440-41-7	0.005	0.25
Cadmium	Cd	7440-43-9	0.005	0.25
Calcium	Ca	7440-70-2	5.0	250
Chromium	Cr	7440-47-3	0.01	0.5
Cobalt	Co	7440-48-4	0.025	1.3
Copper	Cu	7440-50-8	0.01	0.5
Iron	Fe	7439-89-6	0.10	5.0
Lead	Pb	7439-92-1	0.010	0.50
Magnesium	Mg	7439-95-4	5.0	250
Manganese	Mn	7439-96-5	0.015	0.75
Molybdenum	Mo	7439-98-7	0.040	2.0
Nickel	Ni	7440-02-0	0.040	2.0
Potassium	K	7440-09-7	5.0	250
Selenium	Se	7782-49-2	0.02	1.0
Silver	Ag	7440-22-4	0.01	0.5
Sodium	Na	7440-23-5	5.0	250
Thallium	Tl	7440-28-0	0.050	2.5
Tin	Sn	7440-31-5	0.050	10
Vanadium	V	7440-62-2	0.050	2.5
Zinc	Zn	7440-66-6	0.020	2.5

Table II-DoD: DoD LCS/MS Control Limits for ICP

ELEMENT	Symbol	Aqueous Control Limits		Solid Control Limits	
		Lower	Upper	Lower	Upper
Aluminum	Al	86	115	74	119
Antimony	Sb	88	113	79	114
Arsenic	As	87	113	82	111
Barium	Ba	88	113	83	113
Beryllium	Be	89	112	83	113
Cadmium	Cd	88	113	82	113
Calcium	Ca	87	113	81	116
Chromium	Cr	90	113	85	113
Cobalt	Co	89	114	85	112
Copper	Cu	86	114	81	117
Iron	Fe	87	115	81	118
Lead	Pb	86	113	81	112
Magnesium	Mg	85	113	78	115
Manganese	Mn	90	114	84	114
Molybdenum	Mo	89	113	82	116
Nickel	Ni	88	113	83	113
Potassium	K	86	114	81	116
Selenium	Se	83	114	78	111
Silver	Ag	84	115	82	112
Sodium	Na	87	115	83	118
Thallium	Tl	85	114	83	111
Tin	Sn	88	115	80	120
Vanadium	V	90	111	82	114
Zinc	Zn	87	115	82	113

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Inductively Coupled Plasma - Atomic Emission Spectroscopy
METHOD: Method 6010D
ISSUER: Pace ENV - Local Quality - WCOL

Appendix I: North Carolina Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be ≤1/2 LOQ (Table I)	Terminate analysis; Correct the problem; Recalibrate or NCM if applicable
CCB	Immediately following each CCV.	The result must be ≤1/2 LOQ (Table I)	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB or NCM if applicable
Method Blank	One per sample preparation batch of up to 20 samples.	The result must be ≤1/2 LOQ (Table1)	Redigest/reanalyze samples or NCM if applicable

Note: For a multi-point calibration, the concentrations of the standards must bracket the concentration of the samples analyzed. One of the standards must have a concentration equal to the laboratory's lower reporting concentration for the parameter involved. When sample results exceed the quantitation range (i.e. the concentration of the highest calibration standard), the laboratory shall dilute and reanalyze the sample (when sufficient sample volume and holding time permit) to bring results within the quantitation range.

Document Information

Document Number: ME001GA **Revision: -06**

Document Title: Reactivity - Reactive Cyanide and Reactive Sulfide

Department(s): |Wet Chem.|

Date Information

Effective Date: Friday, October 29, 2021

Notes

Document Notes:

All Dates and Times are in Eastern Standard Time Zone.

Signature Manifest

Document Number: ME001GA **Revision:** -06

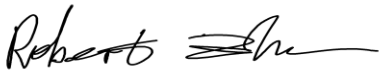
Title: Reactivity - Reactive Cyanide and Reactive Sulfide

All dates and times are in Eastern Standard Time Zone.

ME001GA-06

10/22/2021 8:21:55 AM
Daniel J. Wright
General Manager 1

10/14/2021 11:18:17 AM
Kelly M. Nance
Quality Manager



10/14/2021 4:25:52 PM
Robert Zhu
Technical Specialist

10/25/2021 1:33:20 PM
Bradley E. Belding
Operations Manager

10/14/2021 7:30:36 AM
Kristina P. Bouknight
Environmental Health and Radiation Safety Officer

10/21/2021 9:41:07 AM
Maria S. Gonzalez
Supervisor

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Reactivity - Reactive Cyanide and Reactive Sulfide
METHOD: SW-846 Method Guidance Section 7.3
ISSUER: Pace ENV - Local Quality - WCOL

Table of Contents

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Reactivity - Reactive Cyanide and Reactive Sulfide
METHOD: SW-846 Method Guidance Section 7.3
ISSUER: Pace ENV - Local Quality - WCOL

1.0 Scope and Application

This standard operating procedure (SOP) describes the laboratory procedure to determine the specific rate of release of hydrocyanic acid (or hydrogen cyanide – HCN) and hydrogen sulfide (H2S) upon contact with an aqueous acid.

This test measures only the hydrocyanic acid and hydrogen sulfide evolved at the test conditions. It is not intended to measure forms of cyanide or sulfide other than those that are evolvable under the test conditions.

1.1 Target Analyte List and Limits of Quantitation (LOQ)

The normal LOQ that can be achieved with this procedure is 50 mg/kg.

LOQ are established in accordance with Pace policy and SOPs for method validation and for the determination of detection limits (DL) and quantitation limits (LOQ). DL and LOQ are routinely verified and updated when needed.

1.2 Applicable Matrices

This method is applicable to all wastes, with the condition that wastes do not form explosive mixtures when combined with acids.

2.0 Summary of Method

500 mL of 0.01 N sulfuric acid is added to 10 g of sample in a closed system. The generated gas is swept into a scrubber containing 50 mL of 0.25 N sodium hydroxide for 30 minutes. The analyte is then quantitated using the procedures described in method 9014 for hydrogen cyanide and method 9034 for hydrogen sulfide.

3.0 Interferences

3.1 Interferences are undetermined.

4.0 Definitions

Refer to the *Laboratory Quality Manual* [QAMP ME0012K] for a glossary of common lab terms and definitions.

4.1 Non-conformance Memo (NCM) - A form used to document a non-conforming event. An analyst must document a non-conformance memo when a non-conforming event occurs. A non-conforming event may include the reporting of analytical data outside of method or SOP criteria, or when there is a deviation from a written policy or procedure. Information in an NCM may be used by project managers to flag data in the report narrative, or by the quality department to track trends and initiate corrective actions, where applicable. Additional

information on the NCM policy and procedure is located in the *Complaints and Non-conformances* SOP [QA SOP ME001BO].

5.0 Health and Safety

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

6.0 Sample Collection, Preservation, Holding Time, and Storage

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory performs samples collection for samples to be analyzed by this SOP in accordance with the *Field Services* SOP [FS SOP ME001BS]. Refer to this SOP for these instructions.

The laboratory will provide containers for the collection of samples upon client request for analytical services. Bottle kits are prepared in accordance with the *Sample Container Shipping* SOP [AD SOP ME001DS].

Requirements for container type, preservation, and field quality control (QC) for the common list of test methods offered by Pace are listed in the *Pace-WCOL Analytical Methods List* [ME002BS].

General Requirements

Matrix	Routine Container	Min. Sample Amount[1]	Preservation	Holding Time
Aqueous	250 mL amber glass	50 mL	Thermal: ≤6°C Chemical: None	Collection to Analysis: 28 days
Non-Aqueous	4 oz glass Teflon-lined lid	10 g	Thermal: ≤6°C Chemical: None	Collection to Analysis: 28 days
Solid	4 oz glass Teflon-lined lid	10 g	Thermal: ≤6°C Chemical: None	Collection to Analysis: 28 days

[1]Minimum amount needed for each discrete analysis.

Field / Matrix QC

Additional volume is required for duplicates. A full routine container is sufficient volume to analyze matrix QC.

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with the *Sample Receiving* SOP [AD SOP ME0013H]. Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are stored at 4 ± 2°C until sample analysis.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 28 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

7.0 Equipment and Supplies

NOTE: Refer to the *Major Operational Equipment List* [QA Control Log ME001PM] for specific details regarding the equipment utilized during this procedure.

7.1 Equipment

7.1.1 Round-bottom flasks – 500-mL, two-neck.

7.1.2 Gas scrubber.

7.1.3 Magnetic stirrer and stirring bars.

7.1.4 Funnel.

7.1.5 Flexible tubing – for connection from nitrogen supply to apparatus.

7.1.6 Balance – Analytical, capable of accurately weighing to the nearest 0.0001 g.

7.1.7 Flow meter – Humonics Model 420.

8.0 Reagents and Standards

NOTE: Other volumes of standards and reagents may be prepared to account for expected usage. As long as all ratios are kept constant, this is not considered a deviation from the approved procedure.

NOTE: Reagents may be purchased from any approved vendor that can provide a certificate of analysis. All standards must be prepared using certified reference materials. Follow manufacturer expiration date unless stated otherwise below.

NOTE: All stored reagents and standards must be labeled as required by the Preparation and *Documentation of Laboratory Standards and Reagents* SOP [QA SOP ME001HG], the *Contingency and Emergency Preparedness Plan* [HS SOP ME0012D], the *Safety Manual* [Corp Manual COR-MAN-HSE], and the *Laboratory Quality Manual* [QAMP ME0012K].

NOTE: All reagents and standards are prepared using reagent water unless otherwise noted.

8.1 Reagents

8.1.1 Reagent water – A series of in-house deionized (DI) tanks is employed to purify the incoming water. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks. See the Deionized Water System SOP [QA SOP ME0012S] for further information.

8.1.2 Nitrogen gas tank.

8.1.3 Sulfuric acid (H2SO4), 0.1 N – Add 2.8 mL concentrated H2SO4 to reagent water and dilute to 1 L.

8.1.4

NOTE: When preparing diluted acid, always add acid to water. If the water is added to acid, a violent reaction may occur.

8.1.5 Sodium hydroxide (NaOH) solution, 0.25 N – Dissolve 10 g of NaOH in reagent water and dilute to 1 L with reagent water.

8.1.6 Silver nitrate solution, 0.0192 N – Prepare by crushing approximately 5 g of AgNO3 crystals and drying to constant weight at 40°C. Weigh 3.265 g of dried AgNO3, dissolve in reagent water, and dilute to 1 L.

8.2 Standards

8.2.1 Sulfide reference solution – Dissolve 4.02 g Na2S*9H2O in 1 L of reagent water. This solution contains 570 mg/L H2S.

8.2.2 Cyanide reference solution (1000 mg/L) – In a 1000 mL volumetric flask, dissolve approximately 2.5 g of KOH and 2.51 g KCN in approximately 800 mL of reagent water. Dilute to the line and invert to mix. Standardize against silver nitrate solution (8.1.6).

9.0 Procedure

9.1 Equipment Preparation

9.1.1 Support Equipment

9.1.1.1 The balance is verified at the beginning of each analytical day using a certified weight set. Refer to the *Equipment and Instrumentation* SOP [QA SOP ME002JT] for balance verification procedures and acceptance criteria.

9.1.1.2 Pipettes are maintained and verified as required by the *Equipment and Instrumentation* SOP [QA SOP ME002JT].

9.2 Sample Preparation

9.2.1 Weigh out 10 g of sample and record the weight to the nearest 0.1 g.

9.2.2 Transfer the weighed sample to the round-bottom flask.

9.2.3 Place a magnetic stirring bar in the flask.

9.2.4 Add 50 mL of 0.25 N NaOH to the scrubber.

9.2.5 Assemble the system as follows:

9.2.5.1 Place the round-bottom flask on the magnetic stirrer.

9.2.5.2 Connect the cold finger and jacket to the flask.

9.2.5.3 Connect the scrubber containing NaOH to the cold finger and jacket.

9.2.5.4 Withdraw 50 mL of 0.1 N H2SO4 (7.3) and dilute to 500 mL with DI water to make 0.01 N H2SO4. Add 500 mL of 0.01 N sulfuric acid solution to the flask and seal the flask by inserting the inlet tube connected to a nitrogen gas tank.

9.2.5.5 Begin stirring. The stirring speed must remain constant throughout the test and should not be fast enough to create a vortex.

9.2.5.6 Measure the nitrogen flow rate using the flow meter and adjust to 60 mL/min.

9.2.5.7 Introduce nitrogen gas into the flask.

9.2.5.8 After 30 minutes, close off the nitrogen and disconnect the scrubber.

9.2.5.9 Determine the amount of cyanide or sulfide in the scrubber using appropriate determinative method.

9.2.6 Sufficient records must be maintained to allow for historical reconstruction of testing procedures. Refer to the Logbook and Data Recording SOP [QA SOP ME0012T] for

details regarding documentation requirements. The following quality documents are included in the technical record for this analysis:

- *Reactive Preparation Batch* [Wet Chem Form ME0021G]

10.0 Data Analysis and Calculations

10.1 Refer to the individual analysis SOPs: *Cyanide* [Wet Chem SOP ME0014R] and *Sulfide* SOP [Wet Chem SOP ME001GB].

11.0 Quality Control and Method Performance

11.1 Quality Control

The following QC samples are prepared and analyzed with each batch of samples.

QC Item	Frequency
Method Blank (MB)	1 per batch
Sample Duplicate	1 per 10 samples

11.2 Acceptance Criteria and Required Corrective Action

11.2.1 Method Blank (MB) – One method blank must be processed with each batch of 20 samples. The method blank consists of reagent water for water matrix batches and Ottawa sand for soil matrix batches containing all reagents specific to the method that is carried through the entire analytical procedure. The method blank is used to identify any system and process interferences or contamination that may lead to the reporting of elevated cyanide concentrations or false positive data. Refer to the Cyanide [INM SOP ME0014R] and Sulfide [INM SOP ME001GB] SOPs for blank acceptance criteria and corrective action.

11.2.2 Sample Duplicates – Sample duplicates are performed at a frequency of 10% and must be within 20% RPD. If the RPD is >20%, an NCM must be filed.

11.3 Method Performance

11.3.1 Method Validation

11.3.1.1 Detection Limits

Detection limits (DL) and limits of quantitation (LOQ) are established at initial method setup and verified on an on-going basis thereafter. Refer to Pace ENV corporate SOP

ENV-SOP-CORQ-0011 Method Validation and Instrument Verification and to the *Method Validation SOP* [QA Policy ME003BF] for these procedures.

11.4 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to the *Demonstration of Capability* SOP [QA SOP ME001F2] for more information.

12.0 Data Review and Corrective Action

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employee's complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to the *Data Review* SOP [QA SOP ME003LP] for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must

verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

13.0 Pollution Prevention

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

14.0 Modifications

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 Method Validation and Instrument Verification for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

For compliance samples, all procedures outlined in this SOP must be followed without exception. In the event that a deviation from this SOP cannot be avoided (i.e., demonstrated and uncorrectable matrix interference, non-compatible matrix, insufficient sample amount due to client or laboratory error) it is imperative that an NCM is completed that clearly documents the anomaly. This anomaly must be communicated to the client such that corrective action activity including sample recollection can be performed. Based upon the type and severity of the anomaly, the results may not be appropriate for compliance reporting. Discussions between the client, the appropriate regulatory authority, and the laboratory will be necessary to resolve certain anomalies. In any case, it is essential that any anomaly be documented with an NCM.

15.0 Responsibilities

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 Attachments

Not applicable

17.0 References

Note: Where reference exclude a date or edition, the latest edition of the referenced document adopted/recognized by the laboratory's accreditation bodies applies. Refer to the *Laboratory Quality Manual* [QAMP ME0012K] for details.

17.1 *Consolidated Quality System Manual (QSM) for Environmental Laboratories*. Department of Defense (DoD) / Department of Energy (DoE).

17.2 *General Requirements for the Competence of Testing and Calibration Laboratories*. International Standard ISO/IEC 17025.

17.3 *Laboratory Accreditation Standards*. TNI Standard. The NELAC Institute.

17.4 SW-846, *Test Method for Evaluating Solid Waste, Third Edition – Interim Guidance for Reactive Cyanide and Reactive Sulfide*, Section 7.3.3 and 7.3.4, Revision 3, December 1996.

18.0 Revision History

Revision #	Revision Date	Section Modified	Modification	Reason for Modification
-06	**10/29/2021**	All	Re-wrote and re-formatted entire document	Compliance with Pace policy

Document Information

Document Number: ME001IM **Revision: -07**

Document Title: Extraction of Chlorinated Herbicides

Department(s): |Organic Prep.|

Date Information

Effective Date: Thursday, February 10, 2022

Notes

Document Notes:

All Dates and Times are in Eastern Standard Time Zone.

Signature Manifest

Document Number: ME001IM **Revision:** -07

Title: Extraction of Chlorinated Herbicides

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1/28/2022 12:28:52 PM
Kelly M. Nance
Quality Manager

1/27/2022 1:40:20 PM
Bradley E. Belding
Operations Manager

1/27/2022 9:45:51 AM
Kristina P. Bouknight
Environmental Health and Radiation Safety Officer

2/4/2022 3:54:17 PM
David A. Lucas
Supervisor

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Extraction of Chlorinated Herbicides
METHOD: 8151A
ISSUER: Pace ENV - Local Quality - WCOL

Table of Contents

1.0 Scope and Application

This standard operating procedure (SOP) describes the laboratory procedure for the extraction of certain chlorinated acid herbicides and related compounds which will be analyzed by chromatographic procedures.

Method 8151A describes the procedure for the extraction of certain chlorinated acid herbicides and related compounds in aqueous, soil and waste materials. Because these compounds are produced and used in various forms (i.e., acid, salt, ester, etc.), this method describes a hydrolysis step that can be used to convert herbicide esters into the acid form prior to analysis. Herbicide esters generally have a half-life of less than one week in soil.

2.0 Summary of Method

2.1 For aqueous samples – A measured volume of sample, usually 1 liter, is acidified to pH < 2 with sulfuric acid. The sample is then extracted with diethyl ether and esterified with diazomethane.

2.2 For solid samples – 50 g of sample is acidified to pH < 2 with 1:1 hydrochloric acid. The sample is then extracted and esterified with diazomethane.

2.3 For Non-Aqueous samples-, 1g of sample is extracted and esterified with diazomethane.

2.4 If Herbicide esters are to be determined, hydrolysis conditions for the esters in water and soil extracts are described

3.0 Interferences

3.1 Solvents, reagents, glassware, and other sample processing 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.

3.2 Glassware must be scrupulously cleaned. Clean each piece of glassware as soon as possible after use by detergent washing with hot water, rinses with tap water, rinses with organic-free reagent water, and then with acetone (in that order). After rinsing and drying, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Immediately prior to use, glassware should be rinsed with acetone and pesticide-quality hexane.

3.3 The use of high purity reagents and solvents helps to minimize interference problems. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be necessary.

3.4 Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from waste to waste, depending upon the nature and diversity of the waste being sampled.

3.5 Alkaline hydrolysis and subsequent extraction of the basic solution removes many chlorinated hydrocarbons and phthalate esters that might otherwise interfere with the electron capture analysis. However, hydrolysis may result in the loss of dinoseb and the formation of aldol condensation products if any residual acetone remains from the extraction of solids.

3.6 The herbicides, being strong organic acids, react readily with alkaline substances and may be lost during analysis. Therefore, glassware must be acid-rinsed then rinsed to constant pH with organic-free reagent water. Sodium sulfate and glass wool must be acidified.

3.7 Sample extracts should be dried prior to methylation or else poor recoveries will be obtained.

4.0 Definitions

Refer to the *Laboratory Quality Manual* [QAMP ME0012K] for a glossary of common lab terms and definitions.

5.0 Health and Safety

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

6.0 Sample Collection, Preservation, Holding Time, and Storage

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory performs samples collection for samples to be analyzed by this SOP in accordance with the *Field Services* SOP [FS SOP ME001BS]. Refer to this SOP for these instructions.

The laboratory will provide containers for the collection of samples upon client request for analytical services. Bottle kits are prepared in accordance with the *Sample Container Shipping* SOP [AD SOP ME001DS].

Requirements for container type, preservation, and field quality control (QC) for the common list of test methods offered by Pace are listed in the *Pace-WCOL Analytical Methods List* [ME002BS].

General Requirements

Method	Matrix	Routine Container	Min. Sample Amount[1]	Preservation	Holding Time
8151A	Aqueous	1L amber glass Teflon-lined lid (2X)	1L	Thermal: ≤ 6°C Chemical: None	Collection to Prep: 40 days Prep to Analysis: 7 days
8151A	Soil	4 oz. glass Teflon-lined lid	50g	Thermal: ≤ 6°C Chemical: None	Collection to Prep: 40 days Prep to Analysis: 14 days

[1]Minimum amount needed for each discrete analysis.

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with the *Sample Receiving* SOP [AD SOP ME0013H]. Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are stored at ≤ 6°C until sample preparation. Prepared samples (extracts, digestates, distillates, other) are stored at ≤ 6°C until sample analysis.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 28 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

7.0 Equipment and Supplies

NOTE: Refer to the *Major Operational Equipment List* [QA Control Log ME001PM] for specific details regarding the equipment utilized during this procedure.

7.1 Equipment

7.1.1 Fume hood

7.1.2 Balance- Top-loading, capable of accurately weighing to the nearest 0.1 g

7.1.3 Timers – Capable of measuring seconds.

7.1.4 Water bath – Capable of holding temperature at 85 ± 2°C.

7.1.5 SONICATOR® Model 3000 ULTRASONIC Liquid PROCESSOR by MISONIX- This device is an ultrasonic disruptor that has 1500 V rms (max). The SONICATOR® has a dual horn apparatus, which accommodates two 3/4" diameter extenders for simultaneous sample extraction for the low concentration method or a 1/8" tapered microtip probe that attaches to a 1/2" tapped horn for the medium/high concentration method

7.1.6 SONICATOR Model Q700 by QSONICA- This device is an ultrasonic disruptor that has 1000 V rms (max).

7.1.7 SONABOX™ Acoustic Enclosure (Heat Systems – Ultrasonics, Inc., Model 432B or equivalent) – Recommended for use with the above ultrasonic disruptor. The SONABOX™ aids in the reduction of cavitation noise levels. The Sonabox Acoustic Enclosure reduces noise levels down to safe, comfortable levels.

7.1.8 Shaker – model/serial # 099A DPM12 / 11905743 manufactured by Glass-Col used to prep solids for the shaker method.

7.1.9 Turner – Used to tumble herbicides. Automated turner takes the place of manual shaking.

7.1.10 Kuderna-Danish (K-D) apparatus:

7.1.11 Concentrator tube – 10 mL graduated (Kontes K-570050-1025 or equivalent) with ground-glass stopper.

7.1.12 Evaporation flask – 500 mL (Kontes K-570001-500 or equivalent). Can be attached to concentrator tube with springs, clamps, or equivalent.

7.1.13 Snyder column – Three-ball macro (Kontes K-503000-0121 or equivalent).

7.2 Supplies

7.2.1 Beakers–150 mL, 400 mL, thick-walled

7.2.2 Spatula- Stainless steel, PTFE, or disposable wooden

7.2.3 Stainless steel filter funnels – 75-mm diameter with 35-mm stem and 12-mm stem diameter

7.2.4 Graduated cylinder- 1000 mL, 100 mL

7.2.5 Syringe- 1 mL, gas-tight.

7.2.6 Filter paper – 15-cm diameter (Whatman No. 1 or equivalent).

7.2.7 Glass Wool, acid washed

7.2.8 Clamps to hold K-D flasks, concentrator tubes, and Snyder columns together.

7.2.9 Pipet – 10 mL, sterile, disposable.

7.2.10 Pasteur glass pipets- 2 mL, disposable

7.2.11 Vials – 12 mL, glass with Teflon (PTFE)-lined screw caps – Each new lot of vials must be verified using the Container Precision by Weight spreadsheet [QA Spreadsheet ME001JL] prior to use.

7.2.12 Separatory funnel – 2-liter, with polytetrafluoroethylene (PTFE) stopcock.

7.2.13 Erlenmeyer flask – 500 mL, with a ground-glass joint at the neck.

7.2.14 Boiling chips – Teflon, approximately 10/40 mesh.

7.2.15 pH indicator paper – pH range including the desired extraction pH.

7.2.16 Diazomethane Bubbler – Assemble from two 20 mm × 150 mm test tubes, two Neoprene rubber stoppers, and a source of nitrogen. Use Neoprene rubber stoppers with holes drilled in them to accommodate glass delivery tubes. The exit tube must be drawn to a point to bubble diazomethane through the sample extract. The bubbler assembly is explained in Figure 4

8.0 Reagents and Standards

NOTE: Other volumes of standards and reagents may be prepared to account for expected usage. As long as all ratios are kept constant, this is not considered a deviation from the approved procedure.

NOTE: All stored reagents and standards must be labeled as required by the Preparation and *Documentation of Laboratory Standards and Reagents* SOP [QA SOP ME001HG], the *Contingency and Emergency Preparedness Plan* [HS SOP ME0012D], the *Safety Manual* [Corp Manual COR-MAN-HSE], and the *Laboratory Quality Manual* [QAMP ME0012K].

8.1 Reagents

8.1.1 ***Reagent water*** – Pace employs a series of in-house deionized (DI) tanks to purify the incoming water. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks. See the *Deionized Water System* SOP [QA SOP ME0012S] for further information.

8.1.2 ***Acidified Sodium sulfate (ACS, granular anhydrous), Na2SO4 -*** Purify by heating to 400°C, and maintaining that temperature for 4 hours. Acidify by slurrying 1000 g sodium sulfate with enough diethyl ether to just cover the solid, then add 1.0 mL of concentrated sulfuric acid in a drop wise manner and mix thoroughly. Place tray of acidified sodium sulfate on water bath heated at 65 ± 5°C and evaporate ether and acid until sodium sulfate is completely dried. Store in oven at 130°C. Good for up to 6 months.

Note: Heating must be done under a fume hood.

8.1.2.1 To check the pH of the acidified sodium sulfate, add 1 g of the acidified sodium sulfate to 5 mL of organic-free reagent water. The mixture must have pH of <4.

8.1.3 ***Ottawa sand*** - SiO2 (CAS 14808-60-7) – granular 30-40 mesh.

8.1.4 ***Sulfuric Acid (H2SO4) solution, 1:1 (v/v)*** – Slowly add 50 mL of concentrated H2SO4 to 50 mL of organic-free reagent water. Other concentrations of acid solutions may be used to adjust sample pH, provided that the volume added does not appreciably change (e.g., <1%) the total sample volume.

8.1.5 ***Sulfuric Acid (H2SO4) solution, 1:3*** - Slowly add 50mL of concentrated H2SO4 to 150 mL of organic-free reagent water. Store at 4°C ± 2.

8.1.6 ***Concentrated Hydrochloric Acid (HCl)***

Note: When preparing diluted acid, always add acid to water. If water is added to acid, a violent reaction may occur.

8.1.7 ***Solvents*** – All solvents must be pesticide quality or equivalent

8.1.7.1 Methylene chloride, CH2Cl2

8.1.7.2 Acetone, C3H6O

8.1.7.3 Hexane, C6H14

8.1.7.4 Diethyl Ether, C2H5OC2H5 – must be free of peroxides

Note: Diethyl ether used for this procedure should be stabilized with BHT, not with ethanol, as when ethanol-stabilized ether is used, the methylation reaction may not proceed efficiently, leading to low recoveries of target analytes.

8.1.7.5 Isooctane, (CH3)3CH2CH(CH3)2.

8.1.7.6 Methanol, CH3OH

8.1.7.7 Carbitol (diethylene glycol monoethyl ether), C2H5OCH2CH2OCH2CH2O.

8.1.8 Nitrogen gas.

8.1.9 Diazald – reagent grade.

8.1.10 Silicic Acid – reagent grade. -100 mesh powder stored at 130°C

8.1.11 Potassium Hydroxide (37% aqueous solution (w/v), KOH – Dissolve 37.0 g of potassium hydroxide pellets in reagent water in a 100 mL volumetric flask. Bring up to volume with reagent water.

8.1.12 Sodium Hydroxide (NaOH), (10N) solution – Weigh out 1000.0 grams of sodium hydroxide pellets. Place a 2000 mL beaker containing 1000-1200 mL of reagent water on a stir plate and add small portions of the pellets to beaker. Use caution when adding sodium hydroxide pellets; a substantial amount of heat will be generated by the addition of sodium hydroxide pellets. After the addition of the 1000.0 grams of sodium hydroxide pellets, transfer the solution to an appropriate storage container. Rinse the beaker with reagent water, and transfer to the storage container. Bring the final volume to 2500 mL with reagent water.

8.1.13 Comparator Vials – Make the appropriate comparator vial by transferring desired volume of applicable solvent using a gas tight syringe and place in the desired vial. Example: Transfer 1 mL applicable solvent using a gas tight syringe to a 2 mL vial or transfer 5 mL of applicable solvent using a gas tight syringe to a 12 mL vial. Mark the meniscus with a sharpie and date the vial. All comparator vials must be replaced at least monthly unless there are signs of evaporation or deterioration. Ensure the vials have been verified using the Container Precision by Weight spreadsheet [QA Spreadsheet ME001JL].

8.2 Standards

8.2.1 ***Stock Standard solutions*** – May be purchase from an approved vendor that can provide a certificate of analysis. Follow manufacturer expiration date of stock material.

8.2.2 ***Spike and Surrogate solutions*** – All matrix and surrogate spiking standards are in free acid form and are purchased from approved vender in solutions. All acid standards have a shelf life of 2 months. They must be replaced sooner if verification from an independent source indicates a problem. Refer to Table I for the appropriate concentration of spiking and surrogate solutions used. Table II shows the specific compounds in each spike and surrogate solution and the concentration of each compound that is added to the sample.

Note: All standards prepared for surrogate, LCS, and matrix-spiking solutions must be analyzed by GC or GC/MS prior to being used for sample extraction.

9.0 Procedure

9.1 Equipment Preparation

9.1.1 Support Equipment

9.1.1.1. The balance is verified at the beginning of each analytical day using a certified weight set. Refer to the *Equipment and Instrumentation* SOP [QA SOP ME002JT] for balance verification procedures and acceptance criteria.

9.1.1.2. Water bath – Capable of holding temperature at 85 ± 2°C.

9.1.1.3. SONICATOR® Model 3000 ULTRASONIC Liquid PROCESSOR by MISONIX- This device is an ultrasonic disruptor that has 1500 V rms (max). The SONICATOR® has a dual horn apparatus, which accommodates two 3/4" diameter extenders for simultaneous sample extraction for the low concentration method or a 1/8" tapered microtip probe that attaches to a 1/2" tapped horn for the medium/high concentration method

9.1.1.4. SONICATOR Model Q700 by QSONICA- This device is an ultrasonic disruptor that has 1000 V rms (max).

9.1.1.5. SONABOX™ Acoustic Enclosure (Heat Systems – Ultrasonics, Inc., Model 432B or equivalent) – Recommended for use with the above ultrasonic disruptor. The SONABOX™ aids in the reduction of cavitation noise levels. The Sonabox Acoustic Enclosure reduces noise levels down to safe, comfortable levels.

9.1.1.6. Shaker – model/serial # 099A DPM12 / 11905743 manufactured by Glass-Col used to prep solids for the shaker method.

9.1.1.7. Turner – Used to tumble herbicides. Automated turner takes the place of manual shaking.

9.1.1.8. Kuderna-Danish (K-D) apparatus:

9.1.1.9. Concentrator tube – 10 mL graduated (Kontes K-570050-1025 or equivalent) with ground-glass stopper.

9.1.1.10. Evaporation flask – 500 mL (Kontes K-570001-500 or equivalent). Can be attached to concentrator tube with springs, clamps, or equivalent.

9.1.1.11. Snyder column – Three-ball macro (Kontes K-503000-0121 or equivalent).

9.2 Aqueous and TCLP sample Preparation

Note: BEFORE ANY QUALITY CONTROL OR FIELD SAMPLES ARE PROCESSED, ALL GLASSWARE USED IN THE PROCEDURE FOR ALL SAMPLES MUST BE RINSED WITH HEXANE AS WELL AS BEING ACID RINSED WITH 1:1 SULFURIC ACID AND RINSED TO A CONSTANT pH WITH ORGANIC FREE REAGENT WATER.

9.2.1.1 Aqueous sample volume measurement:

9.2.1.1.1 Transfer 1 L of reagent water into each of 2 separatory funnels for aqueous method blank and laboratory control sample.

9.2.1.1.2 If aqueous sample is collected in a container larger than 1 L, use a properly cleaned graduated cylinder to measure 1 L of sample and transfer to a 2 L separatory funnel.

9.2.1.1.3 Alternatively, mark the sample level on the sample container and transfer the entire contents of the 1 L aqueous sample bottle into a separatory funnel. Refill the sample container with water to the mark and then measure using a 1 L graduated cylinder. Record the volume to the nearest 5 mL.

9.2.1.1.4 If high analyte concentrations are anticipated, a smaller sample volume may be taken and diluted to 1 L with reagent water.

Note: Any reduction in the volume of a sample should be approved by a supervisor, lab director, or QA Officer and completely documented using an NCM. The client shall be notified. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

9.2.1.2 TCLP Leachate/Aqueous TCLP Sample Volume Measurement: Refer to Sections 9.2.1.2.1 through 9.2.1.2.3 for TCLP leachate sample volume measurement. For aqueous TCLP sample volume measurement, refer to Sections 9.2.1.2.4 to 9.2.1.2.6.

9.2.1.2.1 Transfer 100 mL of TCLP blank leachate to each of 2 separatory funnels and dilute to 1000 mL with reagent water for TCLP method blank and laboratory control sample.

9.2.1.2.2 For TCLP samples, transfer 100 mL of the TCLP leachate to a 2 L separatory funnel and dilute to 1000 mL with reagent water.

9.2.1.2.3 An additional 100 mL aliquot of a sample designated for the MS should be transferred to a 2 L separatory funnel and diluted to 1000 mL with reagent water.

9.2.1.2.4 For aqueous TCLP herbicides, samples that are <0.05% solid, 1 L of reagent water is used for both the method blank and the laboratory control sample.

9.2.1.2.5 If the TCLP sample is aqueous, the sample must be filtered before preparation. The aqueous portion collected is defined as the TCLP filtrate. Transfer 100 mL of the aqueous filtrate to a 2 L separatory and dilute to 1000 mL with reagent water.

9.2.1.2.6 An additional 100 mL aliquot of a designated filtrate should be prepared in the same manner as the sample for the MS.

9.2.1.3 Using a syringe, add 1.0 mL of the surrogate spiking solution (refer to Section 8.2) to the method blank, LCS, each sample, and the MS (MSD, if applicable).

9.2.1.4 To each, the LCS and MS (MSD, if applicable) using a syringe, add 0.5 mL of the appropriate matrix spiking solution (refer to Section 8.2).

9.2.1.5 Record the unique chemical ID's of the surrogate and matrix spiking solutions, expiration dates, and volumes used on the prep batch sheet. These chemical ID's will be entered into LIMS during batch promotion. Any supplies used during extraction, which have an associated lot number or unique ID, are also recorded (pH strips, glass wool, etc.).

9.2.1.6 Add 250 g of NaCl to the method blank, LCS, MS/MSD, and samples, seal and shake to dissolve the salt.

9.2.1.7 If samples are being extracted for herbicide esters, hydrolysis must be performed. Refer to Section 9.3.1 for hydrolysis procedure.

9.2.1.8 If samples are being extracted for herbicide acids only, refer to Section 9.3.1.2.

9.3 Extraction of aqueous and TCLP samples:

9.3.1 HYDROLYSIS of Aqueous Samples: Use this step only if herbicide esters, in addition to herbicide acids, are to be determined.

Note: Quality Control (MB, LCS, and MS/MSD) samples are treated and extracted in the same manner as field samples.

9.3.1.1 Add 10-12 mL of 10N NaOH to the sample, seal, and shake. Check the pH of the sample with pH paper. If the sample does not have a pH ≥ 12, adjust the pH by adding more 10 N NaOH. Let the sample sit at room temperature until the

hydrolysis step is completed (usually 1-2 hours), shaking the separatory funnel and contents periodically.

9.3.1.2 Add 60 mL of methylene chloride to rinse the graduated cylinder used in transferring the sample (or sample bottle if the sample was transferred directly from the bottle). Transfer the methylene chloride to the separatory funnel and extract the sample by vigorously shaking the funnel for 2 minutes, with periodic venting to release excess pressure. Alternately, this can be done by using the automated turner for four minutes. Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between the layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration through glass wool, centrifugation, or other physical methods. Discard the methylene chloride phase.

Note: Record the chemical ID of the methylene chloride used for extraction for subsequent entry into LIMS.

9.3.1.3 Add a second 60 mL volume of methylene chloride to the separatory funnel and repeat the extraction procedure a second time, discarding the methylene chloride layer. Perform a third extraction in the same manner.

9.3.2 Add approximately 10 mL of COLD 1:1 sulfuric acid to the sample (or hydrolyzed sample), being extracted for herbicide acids seal, and shake to mix. Check the pH of the sample with pH paper. If the sample does not have a pH < 2, adjust the pH by adding more acid.

Note: Lesser strengths of acid solution may be used, if it does not result in a significant change (e.g., < 1%) in the volume of sample extracted.

9.3.3 Add 60 mL of diethyl ether directly to the separatory funnels for the hydrolyzed samples or use 60 mL of diethyl ether to rinse the cylinder used in transferring sample (or sample bottle if the sample was poured directly from the bottle) and transfer this rinse solvent to the separatory funnel. Record the chemical ID of the diethyl ether used for extraction for subsequent entry into LIMS.

9.3.4 Seal and shake the separatory funnel vigorously for 2 minutes with periodic venting to release excess pressure.

Note: Diethyl ether creates excessive pressure very rapidly; therefore, initial venting should be done immediately after the separatory funnel has been sealed and taken off the holding rack before being shaken once. The separatory funnel should be vented into a hood to avoid exposure of the analyst to solvent vapors.

9.3.5 Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between the layers is more than one third of the volume of the

solvent layer, the analyst must employ mechanical techniques to complete phase separation. The optimum technique depends upon the sample, but may include stirring, filtration through glass wool, centrifugation, or other physical methods.

9.3.6 Collect the aqueous layer (bottom layer) in a 1 L Erlenmeyer flask or original sample container. Drain the diethyl ether extract (top layer) into a 500 mL glass stoppered Erlenmeyer flask. Rinse the separatory funnel with a small portion of diethyl ether and collect the rinsate in the Erlenmeyer flask.

9.3.7 Return the aqueous phase to the separatory funnel. Using 60 mL of diethyl ether, repeat the extraction procedure (Section 9.3.3 - 9.3.6) a second time, combining the extracts in the 500 mL Erlenmeyer flask. Perform a third extraction with 60 mL diethyl ether in the same manner. After all three portions of extracts are combined, refer to Section 11.5.

9.4 Extraction of soil, sediment, or other solid samples by ultrasonic extraction.

9.4.1 Homogenization and Subsampling – See the Solid Sample and Sub-sampling for Analytical Preparation SOP [QA SOP ME001IU] for sub-sampling instructions before weighing up a sample.

9.4.2 Ultrasonic Extraction

9.4.2.1 Add 50 g of the well-mixed solid sample to a 400 mL thick walled beaker. To the MB and LCS, 50 g of Ottawa sand is used. Adjust the pH to ≤2 with concentrated hydrochloric acid and thoroughly mix the contents. Add acidified sodium sulfate, if needed to attain a sandy free flowing mixture.

9.4.2.2 Add 1.0 mL of the surrogate spiking solution to all samples, including the MB, and LCS and MS/MSD, if applicable. To the LCS and each of the sample aliquots weighed for MS/MSD, if applicable, add 1.0 mL of appropriate spiking solution.

9.4.2.3 Immediately after adding the surrogate and spiking solutions, to appropriate samples, add 100 mL of methylene chloride/acetone (80mL: 20mL), (v/v).

9.4.2.4 Place the bottom surface of the tip of the ¾" disruptor horn about ½" below the surface of the solvent, but above the sediment layer. Do not use a micro tip probe.

Note: Be sure the horn is properly tuned according to the manufacturer's instructions.

9.4.2.5 With the power control knob set at 10 (full power), press the START key to run the program. The sample will be extracted ultrasonically for three minutes. (Output control knob at 10 (full power), mode switch on Pulse (pulsing energy rather than continuous energy) and percent-duty cycle set at 50% (energy on 50% of the time and energy off 50% of the time). Allow the solids to settle. Transfer the organic

layer to a 500 ml Erlenmeyer flask through a filter paper containing 7-10 g of acidified sodium sulfate.

9.4.2.6 Ultrasonically extract the sample twice more using 100 mL of methylene chloride with the same ultrasonic conditions. Add 7-10 g of acidified sodium sulfate to each extract and allow the extracts to sit and dry for 2 hours, stirring occasionally.

9.4.2.7 After drying for 2 hours, transfer the extract to a 500 mL K-D flask with a 10 mL concentrator tube attached. Add 2-3 boiling chips and attach the macro Snyder column. Concentrate the extract, using a water bath set at 85°C, to a volume of approximately 5 mL. Add 50 mL of methylene chloride, and let the extracts concentrate to a volume of approximately 5 mL. Remove the flask from the water bath and allow them to cool. If hydrolysis is not needed proceed to 11.5.

9.4.3 HYDROLYSIS of Solid Samples: Use this step only if herbicide esters, in addition to herbicide acids, are to be determined.

9.4.3.1 Add 5ml of 37% aqueous potassium hydroxide and 30 mL of organic reagent water to the extract. Add an additional 2-3 boiling chips. Reflux the mixture on a water bath at 60-65°C until the hydrolysis step is completed (usually 1-2 hours). Remove the flasks from the water bath and allow to cool to room temperature.

CAUTION: the presence of residual acetone will result in the formation of aldol condensation products which will cause GC interference.

9.4.3.2 Transfer the hydrolyzed aqueous solution to a 2000 mL separatory funnel and extract the solution three times with 100 mL portions of methylene chloride allowing 10 minutes between extractions. Discard the methylene chloride phase. At this point, the basic (aqueous) solution contains the herbicide salts.

9.4.3.3 Adjust the pH of the solution to < 2 with cold (4°C) 1:3 sulfuric acid and add 40 mL of diethyl ether. Shake for 2 minutes and let stand for 10 minutes. Collect the aqueous phase in a clean 400ml beaker and the extract in a 500 mL Erlenmeyer flask with glass stopper. Extract and collect twice more with 20 mL of ether. Add 10 g of acidified sodium sulfate and swirl extract. Add acidified sodium sulfate slowly until free flowing and allow the extract to remain in contact with the drying agent for a minimum of 2 hours or leave overnight. Proceed to 11.6.

9.4.4 Shaker Extraction

9.4.4.1 Add 50 g (dry weight) of well mixed, moist solid sample to a 500 ml amber jar.

9.4.4.2 Adjust the pH to 2 with 6 mLs of 1:1 Hydrochloric acid. If necessary, add additional hydrochloric acid until the pH remains at 2.

9.4.4.3 Spike the sample with surrogate.

9.4.4.4 Add 80 mL diethyl ether to the same flask and shake for 20 minutes. Decant the extract and save in a jar. Extract the sample twice more by 80 mL of diethyl ether.

After addition of each solvent, the mixture should be shaken with the shaker for 10 minutes and the ether extract decanted.

9.4.4.5 After the third extraction, the volume of the extract recovered should be at least 75% of the volume of added solvent. If this is not the case, additional extractions may be necessary. Combine the extracts in a 2-L separatory funnel containing 250 mL of reagent water. Acidify reagent water using 400 µL of 1:1 Sulfuric acid. If an emulsion forms, slowly add 5 g of acidified sodium sulfate (anhydrous) until the solvent-water mixture separates. A quantity of acidified sodium sulfate equal to the weight of the sample may be added, if necessary.

9.4.4.6 Check the pH of the extract. If it is not at or below pH 2, add more concentrated HCl until stabilized at the desired pH. Gently mix the contents of the separatory funnel for 1 minute and allow the layers to separate. Collect the aqueous phase in a clean beaker and the extract phase (top layer) in a 500-mL ground glass-stoppered Erlenmeyer flask. Place the aqueous phase back into the separatory funnel and re-extract using 25 mL of diethyl ether. Allow the layers to separate and discard the aqueous layer. Combine the ether extracts in a 500-mL K-D flask.

9.4.4.7 Proceed to hydrolysis in section 9.4.3 for hydrolysis of soil, sediment, or other solid sample extracts. Use this step only if herbicide esters in addition to herbicide acids are to be determined.

9.5 Extraction of Non-Aqueous samples:

9.5.1 All samples must be homogenized prior to obtaining the aliquot to be used for extraction. Refer to *Solid Sample Sub-sampling for Analytical Preparation* SOP [QA SOP ME001IU].

9.5.2 Add 1g of sample to a 12 mL sample vial.

9.5.3 Two 1 g aliquots of the same designated sample must be weighed for the MS/MSD.

9.5.4 Designate two 12 mL vials, one for the method blank (MB) and one for the LCS.

9.5.5 Using a syringe, add 1.0 mL of surrogate spiking solution (refer to Section 8.2) into the method blank, LCS, each sample, and MS/MSD.

9.5.6 To the LCS and MS/MSD, using a syringe, add 1.0 mL of the appropriate matrix spiking solution (refer to Section 8.2).

9.5.7 Dilute the QC and samples to 10 mL with diethyl ether.

9.5.8 Record the unique chemical ID's and expiration date of the surrogate and matrix spiking solutions, and volumes used on the batch sheet. These chemical ID's will be entered into LIMS during batch promotion.

9.5.9 Add 2g of acidified sodium sulfate to the QC and each sample.

9.5.10 Cap and shake the QC and samples for 2 minutes.

9.5.11 If the sample is to be analyzed for both herbicide esters and acids, then the sample extract must be hydrolyzed. In this case, transfer 1.0 mL (a smaller volume or a dilution may be required if herbicide concentrations are large) to a 10 mL concentrator tube and attach it to a K-D Flask. Proceed to section 11.3.3 for hydrolysis. If the sample does not require hydrolysis, proceed to 11.7 for completion.

9.6 Drying of Aqueous extracts:

9.6.1 Add approximately 10 g of acidified sodium sulfate to the Erlenmeyer flask containing extract. Depending on the sample, more sodium sulfate may be necessary to ensure dryness. The amount of sodium sulfate is adequate when, some free flowing crystals are visible when swirling the flask. If the sodium sulfate solidifies or there is no free flowing sodium sulfate, add additional grams of acidified sodium sulfate and again test by swirling.

9.6.2 Allow the extract to remain in contact with the sodium sulfate for a minimum of 2 hours, but can be left in contact with the sodium sulfate overnight.

Note: The drying step is very critical to ensuring complete esterification. Any moisture remaining in the ether will result in low herbicide recoveries.

9.6.2.1 Concentration of extract using the Turbo Vap:

Note: IMMEDIATELY PRIOR TO USE, RINSE EACH piece of glassware, AS WELL AS FUNNELS AND TURBO VAP TUBES, WITH HEXANE.

9.6.2.2 Turbo Vap Concentration: Label Turbo Vap tubes accordingly. Filter the dried extract through a funnel plugged with acid washed glass wool into the corresponding Turbo Vap Tube. Place the Turbo vap tubes in the turbo vap at approximately 35°C. Concentrate the extract to approximately 1.0 mL - 1.5 mL. Transfer the extract to a 12 mL glass vial. Rinse Turbo Vap Tube with diethyl ether, and dilute to 2.5 mL.

9.6.2.3 Esterification of concentrated extract – Diazomethane derivatization – Bubbler method:

CAUTION: Diazomethane is a carcinogen and can explode under certain conditions.

9.6.2.4 The following precautions must be taken:

- Do not heat above 90°C – Explosion may result.
- Avoid grinding surfaces, ground-glass joints, sleeve bearings, and glass stirrers – Explosion may result.
- Store away from alkali metals – Explosion may result
- Solutions of diazomethane decompose rapidly in the presence of solid materials such as copper powder, calcium chloride, and boiling chips.

9.6.3 Add 1.0 mL of isooctane and 0.5 mL of methanol, using 10 mL sterile pipets, to the extract.

9.6.4 Assemble the diazomethane bubbler.

9.6.4.1 In the first test tube, add 5.0 mL of diethyl ether.

9.6.4.2 In the second test tube, add 1.0 mL of diethyl ether, 1.0 mL of carbitol, 1.5 mL of 37% KOH, and 0.1 – 0.2 g of Diazald.

Note: The amount of Diazald used is sufficient for esterification of approximately three sample extracts. An additional 0.1 – 0.2 g of Diazald may be added (after the initial Diazald is consumed) to extend the generation of the diazomethane. There is sufficient KOH present in the original solution to perform a maximum of approximately 20 minutes of total esterification.

9.6.4.3 Immediately place the exit tube into the 12 mL vial containing the sample extract. Apply nitrogen flow (10 mL/min) to bubble diazomethane through the extract for 10 minutes or until the yellow color of diazomethane persists.

9.6.4.4 Remove the 12 mL vial and cover the sample. Place in a hood at room temperature for 20 minutes.

9.6.5 Destroy any unreacted diazomethane by adding 0.1 g of silicic acid to the 12 mL vial. Allow samples to stand un-covered, until the evolution of nitrogen gas has stopped.

9.6.6 Adjust the extract volume to 10 mL with hexane. The final volume of the sample extract is compared to a comparator vial made in section 8.1.13. Label the vial with the sample ID, method, and prep batch number, and store away from light at 4 ± 2°C.

9.7 The extract may now be analyzed for herbicides.

9.8 ***Procedural Variations*** – For compliance samples, all procedures outlined in this SOP must be followed without exception. In the event that a deviation from this SOP cannot be avoided (i.e., demonstrated and uncorrectable matrix interference, non-compatible matrix, insufficient sample amount due to client or laboratory error) it is imperative that an NCM is completed that clearly documents the anomaly. This anomaly must be communicated to the client such that the corrective action activity including the sample recollection can be performed. Based upon the type and severity of anomaly, the results may not be appropriate for compliance reporting. Discussions between the client, the appropriate regulatory authority, and PAS-SC will be necessary to resolve certain anomalies. In any case, it is essential that any anomaly be documented in an NCM.

10.0 Data Analysis and Calculations

Not Applicable

11.0 Quality Control and Method Performance

11.1 Quality Control

The following QC samples are prepared and analyzed with each batch of samples. Refer to Appendix B for acceptance criteria and required corrective action.

QC Item	Frequency
Method Blank (MB)	1 per batch
Laboratory Control Sample (LCS)	1 per batch
Laboratory Control Sample Duplicate (LCSD)	As needed
Matrix Spike (MS)	1 per 10 samples
Matrix Spike Duplicate (MSD)	1 per 10 samples
LOQ Verification	At least annually

11.1.1. ***Extraction Batch*** - Composed of 1 to 20 environmental samples of the same matrix. The maximum time between the start of processing the first and last sample in the batch is 24 hours. The same analyst(s) using the same procedure and reagent(s) must process the samples. The batch must contain a method blank (MB), a laboratory control sample (LCS), if applicable, and a matrix spike/matrix spike duplicate (MS/MSD), when

requested. The batch may contain multiple MS/MSD pairs. If insufficient sample is available for MS/MSD, a LCSD may be extracted.

11.1.2. ***Sample Count*** - Laboratory generated QC samples (MB, LCS, and MS/MSD) are not counted towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

11.1.3. ***Method Blank (MB)*** - One method blank must be processed with each batch. The method blank consists of reagent water and carried through the entire analytical process. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated concentration of compounds or false positive data. Acceptance criteria of method blanks and corrective actions are contained within the individual analytical method SOP.

11.1.3.1. The aqueous method blank is prepared by adding 1.0 mL of surrogate spiking solution, containing all specific compounds, to 1000 mL of reagent water. For aqueous TCLP herbicides, samples that are <>0.05% solid, the method blank is prepared in the same manner. The method blank is then processed as described in Section 11.

11.1.3.2. The TCLP method blank is prepared by diluting 100 mL of the TCLP blank leachate to 1000 mL with reagent water and adding 1.0 mL of surrogate spiking solution. The TCLP method blank is then processed as described in Section 11. TCLP Extraction Fluid 1 and TCLP Extraction Fluid 2 must be extracted in separate leachate batches, as well as, prep batches containing the corresponding samples. One method blank is prepared from each type of extraction fluid.

11.1.3.3. The solid method blank is prepared by adding 1.0 mL of surrogate spiking solution, containing all compounds specific to the method, to the appropriate reagents. The solid method blank is then processed as described in Section 11.

11.1.4. ***Laboratory Control Sample (LCS)*** - One aqueous LCS must be processed with each batch. The LCS consists of all compounds specific to the method, and 1.0 mL of matrix spiking solution, containing known amounts of all compounds of interest. The LCS must be carried through the entire extraction procedure described in Section 11. The LCS is used to monitor the accuracy of the extraction process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. Acceptance criteria of LCS and corrective actions are contained within the individual analytical method SOP.

11.1.4.1. The aqueous LCS is prepared by spiking 1000 mL aliquot of reagent water with 1.0 mL of surrogate spiking solution, and 1.0 mL of the appropriate matrix spiking solution (see Table II). The LCS is then processed as described in Section 11. The aqueous

TCLP herbicide LCS is prepared in the same manner using the matrix spiking solution specific to TCLP herbicides.

11.1.4.2. The TCLP LCS is prepared by diluting 100 mL of TCLP blank filtrate to 1000 mL with reagent water and adding 1.0 mL of surrogate spiking solution and 1.0 mL of the appropriate matrix spiking solution (see Table II). The LCS is then processed as described in Section 11.

11.1.4.3. The solid LCS is prepared by adding 1.0 mL of the surrogate spiking solution, and 1.0 mL of the appropriate matrix spiking solution to the appropriate reagents. The LCS should be processed as described in Section 11.

11.1.5. ***Matrix Spike/Matrix Spike Duplicate (MS/MSD)*** - – One MS/MSD pair must be processed for each aqueous and solid batch. TCLP herbicide extraction requires each batch to contain a matrix spike (MS) only. A matrix spike (MS) is a field sample to which known concentrations of all compounds of interest have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in addition to MS/MSD. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. If MS/MSD are not possible due to limited sample volume, then a NCM is written. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits. Samples identified as field blanks cannot be used for MS/MSD analysis. Acceptance criteria of MS/MSD and corrective actions are contained within the individual analytical method SOP.

11.1.5.1. The aqueous matrix spike sample is prepared by spiking a 1000 mL aliquot of the designated sample with 1.0 mL of surrogate spiking solution and 1.0 mL of the appropriate matrix spiking solution (see Table II). The MS/MSD is processed as described in Section 11. The aqueous TCLP herbicide MS is prepared in the same manner using the matrix spiking solution specific to TCLP herbicides. A matrix spike duplicate (MSD) is not required for TCLP herbicide extraction.

11.1.5.2. The TCLP matrix spike is prepared by diluting 100 mL of TCLP designated sample filtrate to 1000 mL with reagent water and adding 1.0 mL of surrogate spiking solution and 1.0 mL of the appropriate matrix spiking solution (see Table II). The TCLP MS is processed as described in Section 11.

11.1.5.3. The solid matrix spike is prepared by adding 1.0 mL of the surrogate spiking solution, and 1.0 mL of the appropriate matrix spiking solution to 50 g of the designated sample containing specific reagents and solvents to the method. The MS should be processed as described in Section 11.

11.1.5.4. If MS/MSD are not possible due to limited sample volume, then an NCM will be written. The RPD of the LCS must be compared to the matrix spike RPD limits.

11.2 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to the *Demonstration of Capability* SOP [QA SOP ME001F2] for more information.

12.0 Data Review and Corrective Action

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employee's complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to the Data Review SOP [QA SOP ME003LP] for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must

be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

13.0 Pollution Prevention

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

14.0 Modifications

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 Method Validation and Instrument Verification for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

15.0 Responsibilities

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 Attachments

16.1 Appendix A: Tables

16.1.1 Table I: Spike and Surrogate Compounds and Vendor for Herbicides

16.1.2 Table II: Stock Standard Compounds and Vendors for Herbicides

16.1.3 Table III: Spike and Surrogate Compounds for Herbicides

17.0 References

Note: Where references exclude a date or edition, the latest edition of the referenced document adopted/recognized by the laboratory's accreditation bodies applies. Refer to the Quality Assurance Management Plan [QAMP ME0012K] for details.

17.1 Consolidated Quality System Manual (QSM) for Environmental Laboratories. Department of Defense (DoD) / Department of Energy (DoE).

17.2 General Requirements for the Competence of Testing and Calibration Laboratories. International Standard ISO/IEC 17025.

17.3 Laboratory Accreditation Standards. TNI Standard. The NELAC Institute

17.4 SW-846, Test Method for Evaluating Solid Waste, Third Edition – *Chlorinated Herbicides by GC Using Methylation or Pentafluorobenzylation Derivatization, Method 8151A*, Revision 1, December 1996.

17.5 Operation manual for the Sonication unit

17.6 Pace Quality Assurance Management Plan (QAMP) – ME0012K.

18.0 Revision History

Revision #	Revision Date	Section Modified	Modification	Reason for Modification
-07	**02/10/2022**	9.2.1.4	Updated volume for spiking LCS/MS	Corporate compliance audit Qualtrax WF # 31817C. SOP to match current practices.
		8.1.13	Add section about comparator vials	Corporate Compliance audit Qualtrax WF # 31817A.

APPENDIX A: TABLES

Table I: Spike and Surrogate Compounds and Vendors for Herbicides

	Concentration of Stock Solution (ug/ mL)	Concentration in Final Extract (µg/mL)	Amount Added (mL)	Diluted to (mL)	Vendor
Spike Compound				50.0	AccuStandard
2,4,5-T	100	10	5.0		
2,4-D	100	10	5.0		
2,4-DB	100	10	5.0		
Dalapon	100	10	5.0		
Dicamba	100	10	5.0		
Dichloroprop	100	10	5.0		
Dinoseb	100	10	5.0		
MCPA	10,000	1000	5.0		
MCPP	10,000	1000	5.0		
Silvex (2,4,5-TP)	100	10	5.0		
Pentachlorophenol	2000	10	0.25		
TCLP Spike Compound				100	Ultra Scientific
2,4-D	2000	20	1.0		
Silvex (2,4,5-TP)	2000	20	1.0		
Surrogate Compound					Ultra Scientific
DCAA	5000	50	1.0	100	

Table II. Stock Standard Compounds and Vendors for Herbicides

Standard/Solution	Concentration of Stock Solution (ug/ mL)	Concentration in Final Extract (µg/mL)1	Amount Added (mL)	Diluted to (mL)	Vendor
Dinoseb	100	10	1	10	Supelco
Chlorinated Herb Acids				10	Supelco
2,4-D	100	10.0	1		
2,4-DB	200	20.0	1		
2,4,5-T	25	2.5	1		
2,4,5-TP	25	2.5	1		
Dalapon	250	25.0	1		
Dicamba	50	5.0	1		
Dichloroprop	100	10.0	1		
MCPA	10,000	1000.0	1		
MCPP	10,000	1000.0	1		
DCAA	100	10.0	1		
Pentachlorophenol	10	1.0	1		

Table III. Spike and Surrogate Compounds for Herbicides

	Concentration of Stock Solution (µg/ mL)	Concentration in Final Extract (µg/mL)1	Amount Added (mL)	Diluted to(mL)	Vendor
Spike Compound				50.0	AccuStandard
2,4,5-T	100	10	5.0		
2,4-D	100	10	5.0		
2,4-DB	100	10	5.0		
Dalapon	100	10	5.0		
Dicamba	100	10	5.0		
Dichloroprop	100	10	5.0		
Dinoseb	100	10	5.0		
MCPA	10,000	1000	5.0		
MCPP	10,000	1000	5.0		
Silvex (2,4,5-TP)	100	10	5.0		

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Document Title: Acid Digestion of Aqueous Samples and TCLP/SPLP Extracts for Total Metals Analysis by ICP and ICP-MS Spectroscopy

Department(s): |Inorganic Metals|

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Signature Manifest

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General Manager 1

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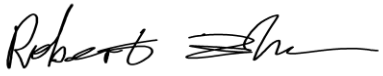
Kelly M. Nance
Quality Manager



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Robert Zhu
Technical Specialist

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Bradley E. Belding
Operations Manager

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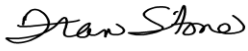
Kristina P. Bouknight
Environmental Health and Radiation Safety Officer



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Fran Stone
Supervisor

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Acid Digestion of Aqueous Samples and TCLP/SPLP Extracts for Total Metals Analysis by ICP and ICP-MS Spectroscopy
METHOD: Method 3010A
ISSUER: Pace ENV - Local Quality - WCOL

Table of Contents

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Acid Digestion of Aqueous Samples and TCLP/SPLP Extracts for Total Metals Analysis by ICP and ICP-MS Spectroscopy
METHOD: Method 3010A
ISSUER: Pace ENV - Local Quality - WCOL

1.0 Scope and Application

This standard operating procedure (SOP) describes the laboratory procedure for the preparation of aqueous and/or soil TCLP/SPLP samples for the analysis of metals by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) and Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) as specified in SW-846 Method 3010A.

Samples prepared by the protocols detailed in this SOP may be analyzed by ICP-AES and/or ICP-MS for any of the elements listed in Table I (Appendix A). Other elements and matrices may be analyzed following digestion by these protocols provided that the method performance criteria specified in this SOP are met.

This method is not a total digestion but will dissolve almost all metals that could become “environmentally available”. By design, metals bound in silicate structures are not dissolved by this procedure as they are not usually mobile in the environment.

2.0 Summary of Method

Method 3010A – A 50 mL extract of sample or 50 mL of aqueous sample is digested in nitric and hydrochloric acids. The digestates are then filtered and re-diluted to 50 mL.

3.0 Interferences

3.1 There are numerous routes by which samples may become contaminated. Potential sources of trace metal contamination include metallic or metal-containing lab ware (e.g., talc gloves which contain high levels of zinc), impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

3.2 Boron and silica from lab glassware may leach into the sample solution during and following sample processing. For critical low -level determinations of boron and silica, only quartz and/or plastic lab ware must be used.

3.3 Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents, and other matrices may not be digested using these methods if they are not soluble with acids. If physical interferences are present, they must be documented.

3.4 Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented.

3.5 Allowing samples to go dry during digestion may result in the loss of volatile metals. If this occurs, the sample must be redigested. Antimony is easily lost by volatilization from hydrochloric media.

3.6 Specific analytical interferences are discussed in each of the determinative methods.

4.0 Definitions

Refer to the *Laboratory Quality Manual* [QAMP ME0012K] for a glossary of common lab terms and definitions.

4.1 Total Metals – the concentration of metals determined on an unfiltered sample following vigorous digestion. This method is designed to determine total environmentally available metals.

4.2 TCLP Metals – the concentration of metals determined on the TCLP extract of a sample following digestion.

4.3 SPLP Metals – the concentration of metals determined on the SPLP extract of a sample following digestion.

4.4 Non-conformance Memo (NCM) - A form used to document a non-conforming event. An analyst must document a non-conformance memo when a non-conforming event occurs. A non-conforming event may include the reporting of analytical data outside of method or SOP criteria, or when there is a deviation from a written policy or procedure. Information in an NCM may be used by project managers to flag data in the report narrative, or by the quality department to track trends and initiate corrective actions, where applicable. Additional information on the NCM policy and procedure is located in the Non-Conformances, Corrective and Preventive Actions, Client Complaints, and Non-Conforming Items and Services SOP [QA SOP ME001BO].

5.0 Health and Safety

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids,

always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

6.0 Sample Collection, Preservation, Holding Time, and Storage

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory performs samples collection for samples to be analyzed by this SOP in accordance with the *Field Services* SOP [FS SOP ME001BS]. Refer to this SOP for these instructions.

The laboratory will provide containers for the collection of samples upon client request for analytical services. Bottle kits are prepared in accordance with the *Sample Container Shipping* SOP [AD SOP ME001DS].

Requirements for container type, preservation, and field quality control (QC) for the common list of test methods offered by Pace are listed in the *Pace-WCOL Analytical Methods List* [ME002BS].

General Requirements

Matrix	Routine Container	Min. Sample Amount[1]	Preservation	Holding Time
Aqueous	250 mL plastic	100 mL	Thermal: NA Chemical: HNO_3	Collection to Analysis: 180 days

[1]Minimum amount needed for each discrete analysis.

Aqueous TCLP/SPLP samples are preserved with concentrated HNO_3 to pH < 2 after they have been poured up for digestion and all spikes have been added. Aqueous samples are then stored at room temperature.

Soil TCLP/SPLP samples are not chemically preserved. The samples are stored at 4 ± 2°C until the time of leaching. After leaching, samples are filtered. Filtrates are preserved with concentrated HNO_3 to pH < 2 after they have been poured up for digestion and all spikes have been added. Filtrates are then stored at room temperature.

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with the *Sample Receiving* SOP [AD SOP ME0013H]. Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are stored at room temperature until sample preparation. Prepared samples (extracts, digestates, distillates, other) are stored at room temperature until sample analysis.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 28 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

7.0 Equipment and Supplies

NOTE: Refer to the *Major Operational Equipment List* [QA Control Log ME001PM] for specific details regarding the equipment utilized during this procedure.

7.1 Equipment

7.1.1 Environmental Express hot block (Cal 3300) or equivalent, capable of maintaining a temperature of 95 ± 2°C.

7.1.2 Hot plate, capable of maintaining a temperature of 95 ± 2°C.

7.1.3 Thermometer that covers a temperature range of 0 – 150°C.

7.1.4 Environmental Express disposable digestion tubes with screw caps, or equivalent.

7.1.5 Filter paper – Whatman No. 41 or equivalent.

7.1.6 Bottle top Dispensers or suitable reagent dispensers.

7.1.7 Calibrated automatic pipettes with corresponding pipette tips or class A volumetric pipettes

7.1.8 Class A volumetric flasks.

7.1.9 Watch glasses.

7.2 Supplies

7.2.1 pH paper, low range.

8.0 Reagents and Standards

NOTE: Other volumes of standards and reagents may be prepared to account for expected usage. As long as all ratios are kept constant, this is not considered a deviation from the approved procedure.

NOTE: All stored reagents and standards must be labeled as required by the Preparation and *Documentation of Laboratory Standards and Reagents* SOP [QA SOP ME001HG], the *Contingency and Emergency Preparedness Plan* [HS SOP ME0012D], the *Safety Manual* [Corp Manual COR-MAN-HSE], and the *Laboratory Quality Manual* [QAMP ME0012K].

8.1 Reagents

8.1.1 Reagent water – A series of in-house deionized (DI) tanks is employed to purify the incoming water. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks. See the Deionized Water System SOP [QA SOP ME0012S] for further information.

8.1.2 pH Adjusted Reagent Water – Reagent water of sufficient volume used to prepare the LCS (and LCSD, if necessary) and MB. To a volume of reagent water, and using pH strips to verify, add drop-wise concentrated nitric acid (HNO_3) until the pH is <2 S.U. Solution expires in 6 months, or when method blanks prepared with this solution do not meet acceptance criteria

8.1.3 Hydrochloric acid (HCl), 1 + 1 solution – Dilute concentrated HCl (trace metal grade or better) with an equal volume of reagent water.

Note: When preparing diluted acids, always add acid to water. If the water is added to the acid, a violent reaction may occur.

8.1.4 Nitric acid (HNO_3), concentrated, trace metal grade or better

8.2 Standards

8.2.1 Laboratory Control Samples (LCS) and Matrix Spike (MS)/Matrix Spike Duplicate (MSD) standards for ICP-AES and for ICP-MS are purchased as custom multi-element mixes or as single-element solutions. These standards are stock standards, and no further preparation is necessary. Refer to Tables II (ICP-AES) and III (ICP-MS) in Appendix A for details regarding the spike concentration of each element in the LCS/MS/MSD samples. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the standard solutions may be used for up to five years from the date of receipt but must be replaced sooner if verification from an independent source indicates a problem.

8.2.1.1 The LCS and MS/MSD samples must contain all the elements designated for analysis in each batch of samples. The preparation of the LCS must include all preservatives as well. If a non-routine element is required that is not contained in the custom solution, a solution must be purchased from a vendor that will cover the additional analyte(s) of interest and provide for a final spike concentration that is appropriate to the determinative method

9.0 Procedure

9.1 Equipment Preparation

9.1.1 Support Equipment

9.1.1.1 The hot block temperature must be recorded at the beginning and end of each batch and for each unit used by measuring the temperature of reagent water in a tube placed in the heated hot block using a NIST traceable calibrated thermometer. Record this temperature in the prep batch.

Note: Be careful not to allow the water in the thermometer digestion tube to evaporate too far. This could lead to incorrect temperature measurement and/or thermometer breakage.

9.1.1.2 Incubators, water baths, refrigerator units, freezer units, bottle top dispensers, pipettes, thermometers, and ovens are maintained and verified as required by the *Equipment and Instrumentation* SOP [QA SOP ME002JT].

9.2 Sample Preparation

Sample digestion must be carried out in a properly functioning hood.

Samples are typically logged as either aqueous or solid in LIMS. When initiating preparation, examine the sample to see if the sample matches the matrix designation. If the sample is logged as aqueous but appears more like a waste (biphasic, sludge like, organic liquid, lots of sediment, etc.), contact the group leader or project manager for further instructions. In some cases, it may be more appropriate to process these samples as solids.

Proper sample identification is extremely important in any preparation procedure. All digestion tubes must be labeled with sample number, method of preparation, and prep batch number.

9.2.1 **Preparation of aqueous and soil TCLP/SPLP samples for analysis by ICP-AES or ICP-MS by method 3010A:**

Note: Beakers and a hot plate can be used in place of digestion tubes and hotblock.

9.2.1.1 Transfer 50 mL of a homogenous mixture of sample directly into a digestion tube. Use an extra digestion tube for each of the MS and MSD samples.

9.2.1.1.1 Typically, a 10x dilution is performed prior to sample prep. Transfer 5mL of a homogenous mixture of sample and 45 mL of pH-Adjusted Reagent Water into a digestion tube.

9.2.1.1.2 A homogenous mixture is obtained by shaking the sample vigorously before transferring.

9.2.1.1.3 If there is not enough sample, dilute the sample with reagent water and follow all steps of the preparation as normal.

9.2.1.2 Aqueous TCLP/SPLP MB and LCS: Measure 50 mL of pH Adjusted Reagent Water into digestion tubes.

9.2.1.2.1 If a 10x dilution is performed prior to sample prep, measure 5mL of pH-Adjusted Reagent Water into a digestion tube.

9.2.1.3 Soil TCLP/SPLP MB and LCS: Measure 50 mL of extraction fluid into digestion tubes.

9.2.1.3.1 If a 10x dilution is performed prior to sample prep, measure 5mL of extraction fluid into a digestion tube.

Note: If there is more than 1 extraction fluid used in extracting samples in the batch (see Method 1311/1312), one method blank is prepared from each extraction fluid.

9.2.1.4 For ICP-AES, spike each of the LCS/MS/MSD with 1.0 mL of the ICP-AES stock standard(s). For ICP-MS, spike each of the LCS/MS/MSD with 1.0 mL of the ICP-MS stock standard(s).

9.2.1.5 Using a bottletop dispenser, add 1.5 mL of concentrated HNO_3 into each digestion tube.

9.2.1.6 Place a ribbed watch glass on each tube and heat on the hotblock at 95° ±2°C until the sample volume is reduced to about 5 mL.

Note: Do not allow samples to go dry during any part of the digestion. Doing so will result in the loss of analytes and the sample must then be redigested.

9.2.1.7 Remove the batch of samples from the hotblock and allow the samples to cool to room temperature.

9.2.1.8 Using a bottletop dispenser, add an additional 1.5 mL of concentrated HNO_3 to each digestion tube. Cover the digestion tubes with a non-ribbed watch glass and heat until digestion is complete. This is indicated when the digestate is light in color or does not change appearance. Add additional acid as necessary.

9.2.1.9 Cover each digestion tube with a ribbed watch glass and evaporate to 3 mL. Allow the samples to cool to room temperature.

9.2.1.10 Add 5 mL of 1 + 1 HCl to each digestion tube using a bottletop dispenser.

9.2.1.11 Replace the watch glass and reheat the samples on the hotblock at 95 ± 2°C for another 15 minutes.

9.2.1.12 Remove the batch of samples from the hotblock and allow the samples to cool to room temperature.

9.2.1.13 Rinse the sides of the digestion tube and the watch glass with reagent water down into the digestion tube.

9.2.1.14 Filter any sample that contains particulates in the bottom of the digestion tube through a Whatman No. 41 filter paper or equivalent.

9.2.1.14.1 If one or more sample in the batch requires filtration, the LCS and MB must also be filtered. If the sample used for the MS/MSD required filtering, then the associated MS/MSD must also be filtered.

9.2.1.15 Dilute the samples back up to 50 mL with reagent water.

9.2.1.16 The sample is now ready for analysis.

10.0 Data Analysis and Calculations

Not applicable

11.0 Quality Control and Method Performance

11.1 Quality Control

The following QC samples are prepared and analyzed with each batch of samples.

QC Item	Frequency
Method Blank (MB)	1 per batch
Laboratory Control Sample (LCS)	1 per batch
Matrix Spike (MS)	1 per batch
Matrix Spike Duplicate (MSD)	1 per batch

11.2 Acceptance Criteria and Corrective Action

11.2.1 Initial and Continuing Demonstrations of Capability (IDOC and CDOC) – To establish the ability of an analyst to generate acceptable accuracy, each analyst must make a satisfactory initial demonstration of capability prior to using any method and at any time there is a change in instrument type or method. Thereafter, a continuing demonstration

of capability is required annually. Refer to the *Demonstration of Capability* SOP [QA SOP ME001F2] for additional information.

11.2.2 Preparation Batch – A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain, at a minimum, a method blank (MB), a laboratory control sample (LCS), and a matrix spike/matrix spike duplicate (MS/MSD). Some client specific data quality objectives (DQO's) may require the use of an unspiked sample duplicate (DUP) in place of or in addition to the MS/MSD. A Laboratory Control Sample Duplicate (LCSD) must be processed along with the LCS in the absence of an MS/MSD. If the client specifies certain samples for the MS/MSD, then the batch may contain multiple sets of MS/MSD's.

11.2.3 Sample Count – Laboratory generated QC samples (MB, LCS, and MS) are not counted towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

11.2.4 Method Blank (MB) – One method blank must be processed with each preparation batch as described in Section 9. The method blank consists of pH Adjusted Reagent Water (for aqueous TCLP/SPLP) or extraction fluid (for soil TCLP/SPLP extraction batches) containing all reagents specific to the method, including preservatives, that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. Criteria for the acceptance of blanks and corrective actions are contained within the individual analytical method SOPs.

11.2.5 Laboratory Control Sample (LCS) – One aqueous LCS (and LCSD, if applicable) must be processed with each preparation batch as described in section 9. The LCS/LCSD consists of pH Adjusted Reagent Water (for aqueous TCLP/SPLP) or extraction fluid (for soil TCLP/SPLP extraction batches) The LCS/LCSD must contain all analytes of interest, including preservatives and must be carried through the entire analytical procedure. The LCS/LCSD is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS/LCSD results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. Criteria for acceptance of LCS/LCSD and corrective actions are contained within the individual analytical method SOP's. Tables II (ICP-AES) and III (ICP-MS) provide the details regarding final spike concentrations.

11.2.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One set of MS/MSD must be processed with each preparation batch as described in Section 9. An MS/MSD is a set of two preparations of a field sample to which known concentrations of target analytes have been added. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in addition to the MS/MSD. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process.

Samples identified as field blanks cannot be used for MS/MSD analysis. Criteria for acceptance of the MS/MSD and corrective actions are contained within the individual analytical method SOP's.

11.3 Method Performance

11.3.1 Method Validation

11.3.1.1 Detection Limits

Detection limits (DL) and limits of quantitation (LOQ) are established at initial method setup and verified on an on-going basis thereafter. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 Method Validation and Instrument Verification and to the Method Validation SOP [QA Policy ME003BF] for these procedures.

11.4 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to the Demonstration of Capability SOP [QA SOP ME001F2] for more information.

12.0 Data Review and Corrective Action

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employee's complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to the *Data Review* SOP [QA SOP ME003LP] for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

13.0 Pollution Prevention

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

14.0 Modifications

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 Method Validation and Instrument Verification for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

Procedural variations are not allowed for any regulatory compliance monitoring. One-time procedural variations are allowed for non-regulatory samples 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 Nonconformance Memo and is approved by a supervisor group leader and the QA Officer. If contractually required, the client shall be notified. Any unauthorized deviations from this procedure must also be documented as a nonconformance

15.0 Responsibilities

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 Attachments

16.1 Appendix A – Tables

16.1.1 Table I: Method 3010A Analyte List

16.1.2 Table II: ICP-AES TCLP/SPLP LCS/MS/MSD Spike Concentrations

16.1.3 Table III: ICP-MS TCLP/SPLP LCS/MS/MSD Spike Concentrations

16.2 Appendix B: Contamination Control Guidelines

17.0 References

Note: Where references exclude a date or edition, the latest edition of the referenced document adopted/recognized by the laboratory's accreditation bodies applies. Refer to the *Laboratory Quality Manual* [QAMP ME0012K] for details.

17.1 *Consolidated Quality System Manual (QSM) for Environmental Laboratories.* Department of Defense (DoD) / Department of Energy (DoE).

17.2 *General Requirements for the Competence of Testing and Calibration Laboratories*. International Standard ISO/IEC 17025.

17.3 *Laboratory Accreditation Standards.* TNI Standard. The NELAC Institute.

17.4 SW-846, Test Method for Evaluating Solid Waste, Third Edition – *Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP-AES Spectroscopy, Method 3010A,* Revision 1, July 1992

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Acid Digestion of Aqueous Samples and TCLP/SPLP Extracts for Total Metals Analysis by ICP and ICP-MS Spectroscopy
METHOD: Method 3010A
ISSUER: Pace ENV - Local Quality - WCOL

18.0 Revision History

Revision #	Revision Date	Section Modified	Modification	Reason for Modification
-07	**10/29/2021**	Signature Page	Updated to QM to Kelly Nance and added Kristina Bouknight as EHSO	Personnel update
		All	Re-wrote and re-formatted entire document	Compliance with Pace policy
		9.2.1.1, 9.2.1.2, & 9.2.1.3	Added 10x dilution prior to prep	Match current procedure
		Table I	Updated table	Match current procedure
		Table II	Added table for ICP-AES	Not included in previous version
		Table III	Removed old Table III	Not required for prep SOP

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Acid Digestion of Aqueous Samples and TCLP/SPLP Extracts for Total Metals Analysis by ICP and ICP-MS Spectroscopy
METHOD: Method 3010A
ISSUER: Pace ENV - Local Quality - WCOL

Appendix A: Tables

Table I: Method 3010A Analyte List

ELEMENT	SYMBOL	CAS NUMBER
Aluminum	Al	7429-90-5
Antimony	Sb	7440-36-0
Arsenic	As	7440-38-2
Barium	Ba	7440-39-3
Beryllium	Be	7440-41-7
Boron	B	7440-42-8
Cadmium	Cd	7440-43-9
Calcium	Ca	7440-70-2
Chromium	Cr	7440-47-3
Cobalt	Co	7440-48-4
Copper	Cu	7440-50-8
Iron	Fe	7439-89-6
Lead	Pb	7439-92-1
Magnesium	Mg	7439-95-4
Manganese	Mn	7439-96-5
Molybdenum	Mo	7439-98-7
Nickel	Ni	7440-02-0
Potassium	K	7440-09-7
Selenium	Se	7782-49-2
Silicon	Si	7440-21-3
Silver	Ag	7440-22-4
Sodium	Na	7440-23-5
Thallium	Tl	7440-28-0
Tin	Sn	7440-31-5
Titanium	Ti	7440-32-6
Uranium	U	7440-61-1
Vanadium	V	7440-62-2
Zinc	Zn	7440-66-6

Table II: ICP-AES TCLP/SPLP LCS/MS/MSD Spike Concentrations

ELEMENT	LCS/MS/MSD Stock Standard (mg/L)	LCS/MS Spike Concentration ** (mg/L)
Aluminum	1000	20
Antimony	50	1
Arsenic	250	5
Barium	500	10
Beryllium	100	2
Boron	50	1
Cadmium	50	1
Calcium	2000	40
Chromium	250	5
Cobalt	100	2
Copper	100	2
Iron	1000	20
Lead	250	5
Magnesium	2000	40
Manganese	100	2
Molybdenum	100	2
Nickel	100	2
Potassium	2000	40
Selenium	50	1
Silicon	100	2
Silver	50	1
Sodium	2000	40
Thallium	40	0.8
Tin	50	1
Titanium	50	1
Uranium	100	2
Vanadium	100	2
Zinc	100	2

** Represents the spike concentration in the final digestate of the ICP-AES LCS/MS/MSD based on the addition of 1.0 mL of the ICP-AES stock standard(s) with a final volume of 50 mL of sample.

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Acid Digestion of Aqueous Samples and TCLP/SPLP Extracts for Total Metals Analysis by ICP and ICP-MS Spectroscopy
METHOD: Method 3010A
ISSUER: Pace ENV - Local Quality - WCOL

Table III: ICP-MS TCLP/SPLP LCS/MS/MSD Spike Concentrations

ELEMENT	LCS/MS/MSD Stock Standard (mg/L)	LCS/MS/MSD Spike Concentration ** (mg/L)
Aluminum	10	0.20
Antimony	10	0.20
Arsenic	10	0.20
Barium	10	0.20
Beryllium	10	0.20
Boron	10	0.20
Cadmium	10	0.20
Calcium	100	2.0
Chromium	10	0.20
Cobalt	10	0.20
Copper	10	0.20
Iron	100	2.0
Lead	10	0.20
Magnesium	100	2.0
Manganese	10	0.20
Molybdenum	10	0.20
Nickel	10	0.20
Potassium	100	2.0
Selenium	10	0.20
Silicon	100	2.0
Silver	10	0.20
Sodium	100	2.0
Thallium	10	0.20
Tin	10	0.20
Titanium	10	0.20
Uranium	10	0.20
Vanadium	10	0.20
Zinc	10	0.20

** Represents the spike concentration in the final digestate of the ICP-MS LCS/ MS/MSD based on the addition of 1.0 mL of the ICP-MS stock standard with a final volume of 50 mL of sample.

Appendix B: Contamination Control Guidelines

The following procedures are strongly recommended to prevent contamination:

All glassware must be washed with detergent and tap water and rinsed with 1+1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered gloves must not be used in the metals laboratory since the powder contains silica and zinc as well as other metallic analytes. Only non-powdered, vinyl, or nitrile gloves are used in the metals laboratory.

Glassware must be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

The following are helpful hints in the identification of the source of contaminants:

Yellow pipette tips and volumetric caps can sometimes contain cadmium.

New glassware especially beakers can be a source of silica and boron.

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Latex gloves contain over 500 ppb of zinc.

Pace-WCOL Analytical Methods

Methods					Matrix											
	Analysis		Preparation		Aqueous						Solid					
Analytical Parameter	Method(s)	SOP #	Method(s)	SOP #	Container Size & Type	Temp Required (°C)	Chemical Preservation	Holding Time Collection to Prep	Holding Time Prep to Analysis	Minimum Volume	Container Size & Type	Temp Required (°C)	Holding Time Collection to Prep	Holding Time Prep to Analysis	Minimum Volume	Waste / Wipes & Notes
Alkalinity	SM2320 B -2011	ME0013Z	NA		250 mL plastic	≤ 6	NP	14 days	NA	100 mL	NA					
Carbon Dioxide (Calculation)	SM4500-CO2 D	ME0013Z	NA		250 mL plastic	≤ 6	NP	14 days	NA	100 mL	NA					
Ammonia Gas Diffusion	EPA 350.1	ME001GZ	NA		250 mL plastic	≤ 6	H2SO4	28 days	NA	100 mL	2 oz. glass teflon-lined lid	≤ 6	28 days	24 hrs.	10g	NA
Biochemical Oxygen Demand (BOD)	SM5210 B-2011	ME001ES	NA		2L plastic	≤ 6	NP	48 hrs.	NA	1L	NA					
British Thermal Unit (BTU)	ASTM D2382	ME001A7	NA		NA						2 oz. glass teflon-lined lid	≤ 6	28 days	NA	10g	Waste - same as solid
Anions by IC Br-, Cl-, F-, SO42-	EPA 300.0 EPA SW-846 9056	ME001J3	NA		250 mL plastic	≤ 6	NP	28 days	NA	10 mL	2 oz. glass teflon-lined lid	≤ 6	28 days	NA	10g	NA
Bulk Density	ASTM D5057	D5057	NA		NA						2 oz. glass teflon-lined lid	NA	NA	NA	10g	NA
Chemical Oxygen Demand (COD)	SM5220 D-2011	ME00145	NA		250 mL plastic	≤ 6	H2SO4	28 days	NA	100 mL	NA					
Total Residual Chlorine (TRC)	SM 4500CL G-2011	ME0016N	NA		250 mL plastic	NA	NP	15 min.	NA	100 mL	NA					

Pace-WCOL Analytical Methods

Methods					Matrix											
	Analysis		Preparation		Aqueous						Solid					
Analytical Parameter	Method(s)	SOP #	Method(s)	SOP #	Container Size & Type	Temp Required (°C)	Chemical Preservation	Holding Time Collection to Prep	Holding Time Prep to Analysis	Minimum Volume	Container Size & Type	Temp Required (°C)	Holding Time Collection to Prep	Holding Time Prep to Analysis	Minimum Volume	Waste / Wipes & Notes
Hexavalent Chromium (Cr+6)	SM3500-Cr B-2011 EPA SW-846 7196A	ME001G0	EPA SW-846 3060A	ME001G0	250 mL plastic	≤ 6	SM3500-Cr B-2011: Filtration (0.45µ) & pH adjustment buffer (ammonium sulfate/ammonium hydroxide) & NaOH to pH 9.3-9.7. EPA 7196A: NP	24 hrs.	SM3500-Cr B-2011: 28 days, if filtered and pH adjusted between 9.3 and 9.7 S.U. with appropriate buffer within 15 minutes of collection/ EPA 7196A: NA	100 mL	2 oz. glass teflon-lined lid	≤ 6	30 days	7 days	10g	NA
Hexavalent Chromium (Cr+6)	EPA SW-846 7199	ME0013B	EPA SW-846 3060A EPA SW-846 7199	ME0013B	250 mL plastic	≤ 6	Filtration (0.45µ) & pH adjustment buffer (ammonium sulfate/ammonium hydroxide) & NaOH to pH 9-9.5.	24 hrs.	NA	100 mL	2 oz. glass teflon-lined lid	≤ 6	30 days	7 days	2.5g	NA
Hexavalent Chromium (Cr+6)	EPA 218.6	ME0013B	EPA 218.6	ME0013B	250 mL plastic	≤ 6	Filtration (0.45µ) & pH adjustment buffer (ammonium sulfate/ammonium hydroxide) & NaOH to pH 9.3-9.7.	24 hrs.	28 days, if filtered and pH adjusted between 9.3 and 9.7 S.U. with appropriate buffer within 15 minutes of collection.	100 mL	NA					NA
Color Platinum Cobalt	SM2120 B-2011	ME001G5	NA		250 mL plastic	≤ 6	NP	48 hrs.	NA	200 mL	NA					NA
Color ADMI	SM2120 E-1993	ME00146	NA		250 mL plastic	≤ 6	NP	48 hrs.	NA	200 mL	NA					NA
Cyanide Amenable	SM4500-CN G-2011 EPA SW-846 9012B	ME0014R	SM4500-CN C-2011 EPA SW-846 9012B	ME0014R	250 mL plastic	≤ 6	NaOH	14 days	NA	100 mL	2 oz. glass teflon-lined lid	≤ 6	14 days	NA	10g	Waste - same as solid
Cyanide Total	SM4500-CN E-2011 EPA 335.4 EPA SW-846 9012B	ME0014R	SM4500-CN C-2011 EPA 335.4 EPA SW-846 9012B	ME0014R	250 mL plastic	≤ 6	NaOH	14 days	NA	100 mL	2 oz. glass teflon-lined lid	≤ 6	14 days	NA	10g	Waste - same as solid
Cyanide Total	Kelada-01	ME00341	NA		250 mL plastic	≤ 6	NaOH	14 days	NA	100 mL	2 oz. glass teflon-lined lid	≤ 6	14 days	NA	10g	Waste - same as solid

Pace-WCOL Analytical Methods

Methods					Matrix											
	Analysis		Preparation		Aqueous						Solid					
Analytical Parameter	Method(s)	SOP #	Method(s)	SOP #	Container Size & Type	Temp Required (°C)	Chemical Preservation	Holding Time Collection to Prep	Holding Time Prep to Analysis	Minimum Volume	Container Size & Type	Temp Required (°C)	Holding Time Collection to Prep	Holding Time Prep to Analysis	Minimum Volume	Waste / Wipes & Notes
Cyanide Total Microdistillation	SM4500-CN E-2011	ME001G1	10-204-00-1-X	ME001G1	250 mL plastic	≤ 6	NaOH	14 days	NA	100 mL	2 oz. glass teflon-lined lid	≤ 6	14 days	NA	0.5g	Waste - same as solid
Dissolved Organic Carbon (DOC)	SM5310 C-2011 EPA SW-846 9060A	ME0016Q	NA		250 mL plastic	≤ 6	NP	48 hrs.	28 Days	100 mL	NA					
Flashpoint-Ignitability Pensky-Marten	EPA SW-846 1010A EPA SW-846 1010B	ME00192	NA		250 mL glass	≤ 6	NP	28 days	NA	50 mL	4 oz. glass teflon-lined lid	≤ 6	28 days	NA	10g	Waste - same as solid
Hardness	SM 2340 C-2011	ME001ZZ	NA		250 mL plastic	≤ 6	HNO3	6 mo.	NA	50 mL	NA					
Ferrous Iron	SM3500-Fe B-2011	ME001G6	NA		40 mL amber glass (2X)	≤ 6	HCl	24 hrs.	NA	40 mL	NA					
Methylene Blue Active Substances (MBAS)	SM5540 C-2011	ME001FL	NA		500mL plastic or amber glass	≤ 6	NP	48 hrs.	NA	300 mL	NA					
Nitrate, Nitrite	EPA 353.2	ME001J4	NA		250 mL plastic	≤ 6	NP	48 hrs.	NA	50 mL	2 oz. glass teflon-lined lid	≤ 6	5 days	48 hrs.	10g	NA
Nitrate, Nitrite by IC	EPA 300.0 EPA SW-846 9056	ME001J3	NA		250 mL plastic	≤ 6	NP	48 hrs.	NA	10 mL	2 oz. glass teflon-lined lid	≤ 6	5 days	48 hrs.	10g	NA
Nitrate + Nitrite	EPA 353.2	ME001J4	NA		250 mL plastic	≤ 6	H2SO4	28 days	NA	50 mL	2 oz. glass teflon-lined lid	≤ 6	28 days	NA	10g	NA
Orthophosphate	EPA 365.1	ME001J5	NA		250 mL plastic	≤ 6	NP	48 hrs.	NA	50 mL	NA					
Paint Filter Test	EPA SW-846 9095B	ME001CH	NA		NA						4 oz. glass teflon-lined lid	NA	NA	NA	50g	NA

Pace-WCOL Analytical Methods

Methods					Matrix											
	Analysis		Preparation		Aqueous						Solid					
Analytical Parameter	Method(s)	SOP #	Method(s)	SOP #	Container Size & Type	Temp Required (°C)	Chemical Preservation	Holding Time Collection to Prep	Holding Time Prep to Analysis	Minimum Volume	Container Size & Type	Temp Required (°C)	Holding Time Collection to Prep	Holding Time Prep to Analysis	Minimum Volume	Waste / Wipes & Notes
pH	SM4500-H B-2011 EPA 150.1 EPA SW-846 9040C EPA SW-846 9045D EPA SW-846 9041A	ME0014S	NA		250 mL plastic	≤ 6	NP	15 min.	NA	50 mL	2 oz. glass teflon-lined lid	≤ 6	15 min.	NA	50g	Waste - same as solid
Phenolics	EPA 420.4 EPA SW-846 9065	ME001D2	EPA SW-846 9065	ME001D2	250 mL amber glass	≤ 6	H2SO4	28 days	NA	100 mL	2 oz. glass teflon-lined lid	≤ 6	28 days	NA	10g	NA
Phosphorus	EPA 365.1	ME001J5	EPA 365.1	ME001J5	250 mL plastic	≤ 6	H2SO4	28 days	NA	50 mL	2 oz. glass teflon-lined lid	≤ 6	28 days	NA	10g	NA
Reactivity (Sulfide)	SW-846 Method Guidance Section 7.3	ME001GA	NA		250 mL amber glass	≤ 6	NP	7 days	NA	50 mL	4 oz. glass teflon-lined lid	≤ 6	7 days	NA	10g	Waste - same as solid
Reactivity (Cyanide)	SW-846 Method Guidance Section 7.3	ME001GA	NA		250 mL amber glass	≤ 6	NP	14 days	NA	50 mL	4 oz. glass teflon-lined lid	≤ 6	14 days	NA	10g	Waste - same as solid
Salinity	SM2520 B-2011	ME0017W	NA		250 mL plastic	≤ 6	NP	28 days	NA	50 mL	2 oz. glass teflon-lined lid	≤ 6	28 days	NA	10g	NA
Specific Conductance	EPA 120.1 SM2510 B-2011	ME0017W	NA		250 mL plastic	≤ 6	NP	28 days	NA	50 mL	2 oz. glass teflon-lined lid	≤ 6	28 days	NA	10g	NA
Sulfide	SM4500-S2 F-2011	ME001GB	NA		250 mL plastic (3X)	≤ 6	NaOH & ZnC4H6O4	7 days	NA	50 mL	2 oz. glass teflon-lined lid	≤ 6	7 days	NA	10g	NA
Sulfide Acid Soluble & Acid In-Soluble	EPA SW-846 9034	ME001GB	EPA SW-846 9030B	ME001GB	250 mL plastic (3X)	≤ 6	NaOH & ZnC4H6O5	7 days	NA	50 mL	2 oz. glass teflon-lined lid	≤ 6	7 days	NA	10g	NA
Sulfite	SM4500 (SO3)2- B-2011	ME001MV	NA		250 mL plastic	≤ 6	NP	15 min.	NA	100 mL	NA					
Temperature	SM2550 B-2010	ME001HP	NA		250 mL plastic	≤ 6	NP	15 min.	NA	50 mL	NA					

Pace-WCOL Analytical Methods

Methods					Matrix											
	Analysis		Preparation		Aqueous						Solid					
Analytical Parameter	Method(s)	SOP #	Method(s)	SOP #	Container Size & Type	Temp Required (°C)	Chemical Preservation	Holding Time Collection to Prep	Holding Time Prep to Analysis	Minimum Volume	Container Size & Type	Temp Required (°C)	Holding Time Collection to Prep	Holding Time Prep to Analysis	Minimum Volume	Waste / Wipes & Notes
Total Dissolved Solids (TDS)	SM2540 C-2011	ME0014W	NA		250 mL plastic	≤ 6	NP	7 days	NA	100 mL	NA					
Total Halogens	EPA SW-846 9056A EPA 300.0	ME001J3	NA		250 mL plastic	≤ 6	NP	28 days	NA	50 mL	2 oz. glass teflon-lined lid	≤ 6	28 days	NA	10g	NA
Total Kjeldahl Nitrogen (TKN)	EPA 351.2	ME001BI	EPA 351.2	ME001BT	250 mL plastic	≤ 6	H2SO4	28 days	NA	50 mL	2 oz. glass teflon-lined lid	≤ 6	28 days	NA	10g	NA
Total Organic Carbon (TOC)	SM5310 C-2011 EPA SW-846 9060A	ME0016Q	NA		250 mL plastic	≤ 6	H2SO4	28 days	NA	50 mL	NA					
Total Organic Carbon (TOC)	Walkley-Black	ME001BG	NA		NA						2 oz. glass teflon-lined lid	≤ 6	28 days	NA	10g	NA
Total Solids (TS)	SM2540 B-2011	ME002YV	NA		250 mL plastic	≤ 6	NP	7 days	NA	100 mL	NA					
Total Suspended Solids (TSS)	SM2540 D-2011	ME0014X	NA		2L plastic	≤ 6	NP	7 days	NA	1000 mL	NA					
Tubidity	180.1	ME001HW	NA		250 mL plastic	≤ 6	NP	48 hrs.	NA	50 mL	NA					
Total Volatile Solid (TVS)	SM2540 B-2011 EPA 160.4	ME0014Y	NA		250 mL plastic	≤ 6	NP	7 days	NA	100 mL	NA					
Metals ICP-MS (excludes Hg)	EPA 200.8 EPA SW-846 6020B	ME0017U ME001FI	EPA 200.2 EPA SW-846 3005A EPA SW-846 3050B EPA SW-846 1311/3010A	ME001HA ME001J7 ME0019C ME001H8	250 mL plastic	NA	HNO3	6 mo.	NA	100 mL	2 oz. glass teflon-lined lid	NA	6 mo.	NA	10g	Waste - same as solid
Metals ICP-AES (excludes Hg)	EPA 200.7 EPA SW-846 6010D	ME0017T ME001FJ	EPA 200.7 EPA SW-846 3005A EPA SW-846 3050B EPA SW-846 1311/3010A	ME001IB ME001J7 ME0019C ME001H8	250 mL plastic	NA	HNO3	6 mo.	NA	100 mL	2 oz. glass teflon-lined lid	NA	6 mo.	NA	10g	Waste - same as solid Wipes - ghost wipe + 2 oz. jar

Pace-WCOL Analytical Methods

Methods					Matrix											
	Analysis		Preparation		Aqueous						Solid					
Analytical Parameter	Method(s)	SOP #	Method(s)	SOP #	Container Size & Type	Temp Required (°C)	Chemical Preservation	Holding Time Collection to Prep	Holding Time Prep to Analysis	Minimum Volume	Container Size & Type	Temp Required (°C)	Holding Time Collection to Prep	Holding Time Prep to Analysis	Minimum Volume	Waste / Wipes & Notes
Silica	EPA 200.7 EPA SW-846 6010D EPA 200.8 EPA SW-846 6020B	ME0017T ME001FJ ME0017U ME001FI	EPA 200.7 EPA SW-846 3005A EPA SW-846 3050B EPA SW-846 1311/3010A EPA 200.2	ME001IB ME001J7 ME0019C ME001H8 ME001HA	250 mL plastic	<6	NA	6 mo.	NA	100 mL	2 oz. glass teflon-lined lid	NA	6 mo.	NA	10g	Waste - same as solid Wipes - ghost wipe + 2 oz. jar
Mercury CVAA	EPA 245.1 EPA SW-846 7470A	ME0017R	EPA 245.1 EPA SW-846 1311/7470A	ME00II ME0019C	250 mL plastic	NA	HNO3	28 days	NA	100 mL	NA					
Mercury CVAA	EPA SW-846 7471B	ME0017R	EPA 245.5 EPA SW-846 7471B	ME001J6	NA						2 oz. glass teflon-lined lid	≤ 6	28 days	NA	10g	Waste - same as solid
Mercury LL CVAFS	EPA 1631E	ME0017X	NA		40 mL glass certified Hg-free vials w/ teflon-lined septa no headspace (3X)	NA	NP	28 days	NA	40 mL	NA					
EDB / DBCP	EPA SW-846 8011	ME00187	NA		40mL glass no headspace (3X)	≤ 6	HCl	14 days	NA	40 mL	NA					
EDB / DBCP	EPA 504.1	ME00187	NA		40mL glass no headspace (3X)	≤ 6	Na2S2O3	14 days	NA	40 mL	NA					
Explosives	EPA SW-846 8330A EPA SW-846 8330B	ME001JB ME001R4	EPA SW-846 3535A EPA SW-846 8330A EPA SW-846 8330B	ME001R4 ME001JB	1L amber glass teflon-lined lid (2X)	≤ 6	NP	7 days	40 days	1L	4 oz. glass teflon-lined lid	≤ 6	14 days	40 days	50g	NA
Herbicides	EPA SW-846 8151A	ME00157	EPA SW-846 8151A EPA SW-846 1311/8151A	ME001IM ME0019C	1L amber glass teflon-lined lid (2X)	≤ 6	NP	7 days	40 days	1L	4 oz. glass teflon-lined lid	≤ 6	14 days	40 days	50g	Waste - same as solid
Pesticides / PCBs	EPA 608.3 EPA SW-846 8081B EPA SW-846 8082A	ME0019A	EPA 608.3 EPA SW-846 3546 EPA SW-846 1311/3520C-RVE EPA SW-846 3520C-RVE EPA SW-846 3540C EPA SW-846 3550C EPA SW-846 3580A	ME00156 ME0019C ME00155 ME001LX ME00154 ME00150	250 mL amber glass teflon-lined lid (2X)	≤ 6	NP	7 days (PCB 8082 - 1 yr)	40 days (PCB 8082 - 1 year)	250 mL	4 oz. glass teflon-lined lid	≤ 6	14 days (PCB 8082 - 1 year)	40 days (PCB 8082 - 1 year)	50g	Waste - same as solid PCB Wipes - gauze + 4mL hexane in 4 oz. jar
Petroleum Hydro-carbons/ Oil & Grease (Hexane Extractable Material)	EPA SW-846 1664B	ME0014T	NA		500 mL glass wide-mouth (2X)	≤ 6	HCl	28 days	NA	500 mL	NA					

Pace-WCOL Analytical Methods

Methods					Matrix											
	Analysis		Preparation		Aqueous						Solid					
Analytical Parameter	Method(s)	SOP #	Method(s)	SOP #	Container Size & Type	Temp Required (°C)	Chemical Preservation	Holding Time Collection to Prep	Holding Time Prep to Analysis	Minimum Volume	Container Size & Type	Temp Required (°C)	Holding Time Collection to Prep	Holding Time Prep to Analysis	Minimum Volume	Waste / Wipes & Notes
Petroleum Hydro-carbons/ Oil & Grease (Hexane Extractable Material)	EPA SW-846 9071B	ME001IO	NA		NA						4 oz. glass teflon-lined lid	≤ 6	28 days	NA	100g	NA
Per- & Polyfluorinated Alkyl Substances	EPA 533	ME003UB	NA		250 mL polypropylene	≤ 10	Ammonium Acetate	28 days	28 days	250 mL	NA					
Per- & Polyfluorinated Alkyl Substances	EPA 537.1	ME002I6	NA		250 mL polypropylene	≤ 10	TRIZMA	14 days	28 days	250 mL	NA					
Per- & Polyfluorinated Alkyl Substances	QSM5.3 Table B-15	ME003NI	NA		250 mL HDPE	≤ 10	NP	28 days	28 days	250 mL	4 oz. HDPE	≤ 10	28 days	28 days	10g	NA
Per- & Polyfluorinated Alkyl Substances	QSM5.3 Table B-15 DAI	ME002I7	NA		250 mL HDPE	≤ 10	NP	28 days	28 days	8 mL	NA					
SVOC GC-MS	EPA SW-846 8270D EPA SW-846 8270E	ME0014Q	EPA SW-846 3546 EPA SW-846 1311/3520C-RVE EPA SW-846 1312/3520-RVE EPA SW-846 3520C-RVE EPA SW-846 3540C EPA SW-846 3550C EPA SW-846 3580A	ME00156 ME0019C ME001IY ME00155 ME001LX ME00154 ME00150	250 mL amber glass teflon-lined lid (2X)	≤ 6	NP	7 days	40 days	250 mL	4 oz. glass teflon-lined lid	≤ 6	14 days	40 days	50	Waste - same as solid
SVOC GC-MS	EPA 625.1	ME0014Q	EPA 625.1	ME0014Q	250 mL amber glass teflon-lined lid (2X)	≤ 6	Na2S2O3 (if chlorinated)	7 days	40 days	250 mL	NA					
DRO	EPA SW-846 8015C	ME00138	EPA SW-846 1311/3520C-RVE EPA SW-846 3520C-RVE EPA SW-846 3550C EPA SW-846 3580A	ME0019C ME00155 ME00154 ME00150	250 mL amber glass teflon-lined lid (2X)	≤ 6	HCl	7 days	40 days	250 mL	4 oz. glass teflon-lined lid	≤ 6	14 days	40 days	50	Waste - same as solid
Fl-PRO	FLO-PRO	ME001IQ	EPA SW-846 3520C-PRO EPA SW-846 3550C-PRO	ME00155 ME00154	1L amber glass teflon-lined lid (2X)	≤ 6	HCl	7 days	40 days	1L	4 oz. glass teflon-lined lid	≤ 6	14 days	40 days	50	Waste - same as solid
EPH	MADEP-EPH-04	ME001J1	MADEP-EPH-04	ME001IN	1L amber glass teflon-lined lid (2X)	≤ 6	HCl	14 days	40 days	1L	4 oz. glass teflon-lined lid	≤ 6	14 days	40 days	50	Waste - same as solid

Pace-WCOL Analytical Methods

Methods					Matrix											
	Analysis		Preparation		Aqueous						Solid					
Analytical Parameter	Method(s)	SOP #	Method(s)	SOP #	Container Size & Type	Temp Required (°C)	Chemical Preservation	Holding Time Collection to Prep	Holding Time Prep to Analysis	Minimum Volume	Container Size & Type	Temp Required (°C)	Holding Time Collection to Prep	Holding Time Prep to Analysis	Minimum Volume	Waste / Wipes & Notes
GRO	EPA SW-846 8015C	ME00137	EPA SW-846 5030B	ME00137	40 mL glass no headspace (3X)	≤ 6	HCl	14 days	NA	40 mL	40 mL pre-weighed VOA vial w/ 5 mL MeOH (2X)	≤ 6	14 days	NA	10g	2 oz. glass Teflon-lined lids
VPH	MADEP-VPH-04	ME00136	MADEP-VPH-04	ME00136	40 mL glass no headspace (3X)	≤ 6	HCl	14 days	NA	40 mL	40 mL pre-weighed VOA vial w/ 5 mL MeOH (2X)	≤ 6	28 days	NA	10g	Waste - same as solid
Glycols	EPA SW-846 8015C	ME001FZ	NA		40 mL glass no headspace (3X)	≤ 6	HCl	14 days 7 days if no HCl	NA	40 mL	2 oz. glass teflon-lined lid	≤ 6	14 days	NA	50g	2 oz. glass Teflon-lined lids
Alcohols	EPA SW-846 8015C	ME001FZ	NA		40 mL glass no headspace (3X)	≤ 6	HCl	14 days 7 days if no HCl	NA	40 mL	2 oz. glass teflon-lined lid	≤ 6	14 days	NA	50g	2 oz. glass Teflon-lined lids
VOC	EPA 624.1 EPA SW-846 8260B EPA SW-846 8260C EPA SW-846 8260D SM6200 B-2011	ME0012X	EPA 624.1 EPA SW-846 5035 EPA SW-846 1311/5030B EPA SW-846 5030B	ME0012X ME0019C	40 mL glass teflon-lined septa no headspace (3X)	≤ 6	HCl Na2S2O3 (if chlorinated)	14 days 7 days if no HCl	NA	40 mL	5035 Kit (2 x 40 mL pre-weighed vials containing reagent water and stir bar. 1x40 mL pre-weighed vial with 5 mL methanol, and one 40mL VOA.	≤ 6	Freeze vials containing regant water within 48 hrs/14 days	NA	15g	2 oz. glass Teflon-lined lids
VOC (w/ Acrolein & Acrylonitrile)	EPA 624.1 EPA SW-846 8260B EPA SW-846 8260C EPA SW-846 8260D	ME0012X	EPA 624.1 EPA SW-846 5035 EPA SW-846 1311/5030B EPA SW-846 5030B	ME0012X ME0019C	40 mL glass teflon-lined septa no headspace (3X)	≤ 6	NP	EPA 624.1 72 hrs. EPA SW-846 8260 7 days	NA	40 mL	NA	NA	NA	NA	NA	NA
VOC	EPA 524.2	ME001HI	EPA 524.2	ME001HI	40 mL glass teflon-lined septa no headspace (3X)	≤ 6	HCl Na2S2O3 (if chlorinated)	14 days	NA	40 mL	NA					

Pace-WCOL Analytical Methods

Methods					Matrix											
	Analysis		Preparation		Aqueous						Solid					
Analytical Parameter	Method(s)	SOP #	Method(s)	SOP #	Container Size & Type	Temp Required (°C)	Chemical Preservation	Holding Time Collection to Prep	Holding Time Prep to Analysis	Minimum Volume	Container Size & Type	Temp Required (°C)	Holding Time Collection to Prep	Holding Time Prep to Analysis	Minimum Volume	Waste / Wipes & Notes
VOC - 1,4-Dioxane	EPA 624.1	ME0012X	EPA 624.1	ME0012X	40 mL glass teflon-lined septa no headspace (3X)	≤ 6	NP	14 days	NA	40 mL	NA					
Dissolved Gasses (Methane, Ethane, and Ethene)	RSK-175	ME00139	NA		40 mL glass teflon-lined septa no headspace (3X)	≤ 6	HCl Na2S2O3 (if chlorinated)	14 days	NA	40 mL	NA					
VOC	SOM02.4	Low/Med ME001E3 Trace ME001E2	NA		40 mL glass teflon-lined septa no headspace (3X)	≤ 6	HCl Na2S2O3 (if chlorinated)	NA	10 days from VTSR	40 mL	LL: 40 mL pre-weighed closed-system vial 5mL DI H2O and 1 MeOH; can be 3 x 5g vials with stirbar	≤ 6	10 days from VTSR	NA	5g	NA
SVOC	SOM02.4	ME001E1	SOM02.4	Ultrasonic ME001DT, Liq-Liq ME001DU	1 L amber glass PFTE-lined screw caps	≤ 6	NP	5 days from VTSR	40 days	1L	4 oz. glass teflon-lined lid	≤ 6	10 days from VTSR	40 days	100g	NA
Pesticides/PCBs	SOM02.4	(PCB) ME001NS (Pest) ME001NT	SOM02.4	Ultrasonic ME001DT, Sep Funnel ME001DW, Liq-Liq ME001DU, Cleanup ME001DV	1 L amber glass PFTE-lined screw caps	≤ 6	NP	5 days from VTSR	40 days	1L	4 oz. glass teflon-lined lid	≤ 10	10 days from VTSR	40 days	100g	NA
TCLP/SPLP Herbicides	SOM02.4	ME0019C	Please see analysis and prep SOPs for each individual test		1L amber glass	≤ 6	NP	5 days from VTSR	7 days	1L	9 oz. glass teflon-lined lid	≤ 6	10 days from VTSR	7 days	100g	Waste - same as solid
ZHE Volatiles with ≥ 0.5% solid	SOM02.4	ME0019C	Please see analysis and prep SOPs for each individual test		NA						40 mL glass PTFE-lined septum capped vial	≤ 6	10 days from VTSR	7 days	40mL	NA
TCLP/SPLP Non-Volatiles or Waste Containing ≤ 0.5% solid	SOM02.4	ME0019C	Please see analysis and prep SOPs for each individual test		1 L amber glass PFTE-lined screw caps	≤ 6	NP	5 days from VTSR	7 days	1L	4 oz. glass teflon-lined lid	≤ 6	10 days from VTSR	7 days	100g	NA
Metals	ISM02.4	(ICP) ME0013K and (ICPMS) ME001DZ	NA		glass or polyethylene containers	≤ 6	HNO3	NA	180 days from VTSR	100 mL	2 oz. glass teflon-lined lid	≤ 6	NA	180 day from VTSR	1.0g	NA

Pace-WCOL Analytical Methods

Methods					Matrix											
	Analysis		Preparation		Aqueous						Solid					
Analytical Parameter	Method(s)	SOP #	Method(s)	SOP #	Container Size & Type	Temp Required (°C)	Chemical Preservation	Holding Time Collection to Prep	Holding Time Prep to Analysis	Minimum Volume	Container Size & Type	Temp Required (°C)	Holding Time Collection to Prep	Holding Time Prep to Analysis	Minimum Volume	Waste / Wipes & Notes
Mercury	ISM02.4	ME0013G	NA		glass or polyethylene containers	≤ 6	HNO3	NA	26 days from VTSR	100 mL	2 oz. glass teflon-lined lid	≤ 6	26 day from VTSR	NA	0.5g	NA
ICP-AES/ ICP-MS (TCLP/SPLP)	ISM02.4	ME0019C	Please see analysis and prep SOPs for each individual test		NA						2 oz. glass teflon-lined lid	≤ 6	NA	180 day from VTSR	100g	NA
Mercury (TCLP/SPLP)	ISM02.4	ME0019C	Please see analysis and prep SOPs for each individual test		NA						2 oz. glass teflon-lined lid	≤ 6	26 day from VTSR	NA	100g	NA
Cyanide	ISM02.4	ME001E0	NA		glass or polyethylene containers	≤ 6	NaOH	NA	12 days from VTSR	50 mL	2 oz. glass teflon-lined lid	≤ 6	NA	12 days from VTSR	1g	NA
SPLP Cyanide	ISM02.4	ME0019C	Please see analysis and prep SOPs for each individual test		NA						4 oz. glass teflon-lined lid	≤ 6	NA	12 days from VTSR	1g	NA
TCLP and SPLP VOC	Leach by TCLP/SPLP Method 1311/1312	ME0019C	Please see analysis and prep SOPs for each individual test		1L amber glass	≤ 6	NP	14 days	14 days	1L	2 oz. glass septum cap (no headspace)	≤ 6	14 days	14 days	100g	NA
TCLP and SPLP SVOC (BNAs, Pesticides, Herbicides)	Leach by TCLP/SPLP Method 1311/1312	ME0019C	Please see analysis and prep SOPs for each individual test		1L amber glass	≤ 6	NP	14 days	7 days	1L	9 oz. glass teflon-lined lid	≤ 6	14 days	7 days	100g	NA
TCLP and SPLP Metals (except Mercury)	Leach by TCLP/SPLP Method 1311/1312	ME0019C	Please see analysis and prep SOPs for each individual test		1L amber glass	≤ 6	NP	180 days	180 days	1L	9 oz. glass teflon-lined lid	≤ 6	180 days	180 days	100g	NA
TCLP and SPLP Mercury	Leach by TCLP/SPLP Method 1311/1312	ME0019C	Please see analysis and prep SOPs for each individual test		1L amber glass	≤ 6	NP	28 days	28 days	1L	9 oz. glass teflon-lined lid	≤ 6	28 days	28 days	100g	MA
Fecal Coliform	Colilert-18 ATP	ME001BL	NA		290 mL plastic sterile	≤ 6	Na2S2O3	8 hrs.	NA	100 mL	NA					
Total Coliform	SM 9223B-2004	ME0014Z	NA		120 mL plastic sterile	≤ 6	Na2S2O3	30 hrs.	NA	100 mL	NA					

Pace-WCOL Analytical Methods

Methods					Matrix											
	Analysis		Preparation		Aqueous						Solid					
Analytical Parameter	Method(s)	SOP #	Method(s)	SOP #	Container Size & Type	Temp Required (°C)	Chemical Preservation	Holding Time Collection to Prep	Holding Time Prep to Analysis	Minimum Volume	Container Size & Type	Temp Required (°C)	Holding Time Collection to Prep	Holding Time Prep to Analysis	Minimum Volume	Waste / Wipes & Notes
Heterotrophic Plate Count	SimPlate	ME001J0	NA		120 mL plastic sterile	≤ 6	Na2S2O3	8 hrs.	NA	100 mL	NA					
Fecal Coliform (MF)	SM 9222D-2006	ME0014U/ ME002L8	NA		290 mL plastic sterile	≤ 6	Na2S2O3	8 hrs.	NA	250 mL	120 mL plastic sterile	≤ 6	8 hrs (NC) 24 hrs (SC)	NA	10g	NA
Fecal Coliform (MPN)	SM 9221C E-2006	ME00143	NA		290 mL plastic sterile	≤ 6	Na2S2O3	8 hrs.	NA	250 mL	120 mL plastic sterile	≤ 6	8 hrs (NC) 24 hrs (SC)	NA	10g	NA
E. Coli (MPN)	SM 9223B-2004 (by Colilert-18)	ME001BL	NA		290 mL plastic sterile	≤ 6	Na2S2O3	8 hrs.	NA	100 mL	NA					

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Notes

Document Notes:

All Dates and Times are in Eastern Standard Time Zone.

Signature Manifest

Document Number: ME0013H **Revision:** -17

Title: Sample Receiving

All dates and times are in Eastern Standard Time Zone.

ME0013H-17

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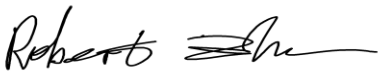
Daniel J. Wright
General Manager 1

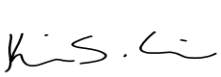
9/9/2021 12:01:26 PM
Kelly M. Nance
Quality Manager



9/7/2021 11:19:01 AM
Robert Zhu
Technical Specialist

9/20/2021 11:52:43 AM
Bradley E. Belding
Operations Manager

9/13/2021 4:09:15 PM
Kristina P. Bouknight
Environmental Health and
Radiation Safety Officer



9/10/2021 8:49:16 AM
Kevin S. Chavis
Supervisor

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Sample Receiving
ISSUER: Pace ENV - Local Quality - WCOL

TABLE OF CONTENTS

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Sample Receiving
ISSUER: Pace ENV - Local Quality - WCOL

1.0 PURPOSE

Sample control procedures are necessary in the laboratory from the time of sample receipt to the time the sample is discarded or returned to the client. Procedures in addition to those outlined in this SOP may be followed or implemented for special projects if and/or when requested by the client.

2.0 SCOPE AND APPLICATION

The procedures outlined in this SOP are applicable to all personnel at Pace-WCOL involved in the receipt of samples by the laboratory.

3.0 SUMMARY

This SOP outlines sample receiving procedures including receipt, prioritizing and logging, checking and adjusting preservation, subsampling, inter-laboratory (IR) transfer, subcontracting, and return or disposal of samples.

4.0 DEFINITIONS

Refer to the *Laboratory Quality Manual* [QAMP ME0012K] for a glossary of terms used in this procedure.

5.0 HEALTH AND SAFETY

5.1 Procedures shall be carried out in a manner that protects the health and safety of all Pace-WCOL personnel. All work must be stopped in the event of a known or potential compromise to the health and safety of a Pace-WCOL employee. The situation must be reported immediately to the Environmental Health and Safety Officer (EHSO).

5.2 As stated in the *Pace-WCOL Comprehensive Chemical Hygiene, Safety, and Hazard Communication Plan* [HS SOP ME0012D], eye protection that satisfies ANSI Z87.1, a laboratory coat, and at least latex gloves must be worn while samples, standards, and reagents are being handled. Contaminated disposable gloves are to be removed and discarded.

5.3 Be aware of sample container conditions upon inspecting coolers. If broken glass is discovered, cut-proof gloves must be worn while removing contents from the cooler.

5.4 The health and safety hazards of many of the chemicals used in these procedures have not been fully defined. Additional health and safety information can be obtained from safety data sheets (SDS) maintained in the public directory. Physical and health hazards specific to this procedure:

5.4.1 The following material(s) are classified as **corrosive**: sulfuric acid, hydrochloric acid, and nitric acid

5.4.2 The following material(s) are classified as **caustic**: sodium hydroxide

5.4.3 The following material(s) are classified as **irritant**: zinc acetate, methanol

5.4.4 The following material(s) are classified as **flammable**: methanol

5.5 All reagents must be carefully prepared inside of the fume hood located in the sample receiving area.

5.6 When preparing diluted acid, always add acid to water. If the water is added to the acid, a violent reaction may occur.

5.7 Samples requiring chemical preservation upon receipt must be carefully preserved inside of the fume hood located in the sample receiving area. If an adverse reaction occurs as a result of preservation attempts, this should be noted on the SRC and the EHSO should be contacted for further guidance.

5.8 Employees receiving samples must pay attention to the physical characteristics of the samples being received. Any samples that appear inherently hazardous, or are labeled as containing hazardous substances, must be stored in the fume hood and brought to the attention of the EHSO and the operations manager prior to sample processing. Indicators of a hazard include, but are not limited to:

5.8.1 A sample is received labeled with a pictogram, fire diamond, or other indication that the sample may pose some hazard;

5.8.2 The chain-of-custody is marked by the client denoting that the samples are hazardous;

5.8.3 Samples are accompanied by a safety data sheet (SDS);

5.8.4 Client sample IDs indicate a potential hazard (example: white phosphorus, asbestos, gasoline spill, etc.);

5.8.5 The sample is bi-phasic (multi-layered).

5.9 Samples comprised of commercially available chemical product, or mixtures thereof, must be submitted with an accompanying Safety Data Sheet (SDS) outlining the hazards associated with the materials received. SDSs must be forwarded to the Environmental Health and Safety Officer (EHSO) and Operations Manager, immediately upon receipt, for risk evaluation.

5.10 Any sample determined to pose a health or physical hazard to employees, must be identified and labeled in a conspicuous manner:

5.10.1 Hazard information must be noted in the comments section of the LIMS5 sample receiving module at the time of sample log-in (example: corrosive, toxic, caustic).

5.10.2 The appropriate hazardous (HAZ) sample container types must be chosen during log-in of samples.

5.10.3 OSHA pictograms, indicating the hazards presented by the sample, must be applied to each applicable sample container. Labels must be applied to the top and side of each bottle (refer to Appendix J for a list of OSHA pictograms and associated hazard classification).

5.10.4 If a SDS is received, each SDS must be scanned into LIMS5. The lot number associated with the samples must be sent to the EHSO.

5.11 Shipping containers must be opened in an area providing adequate ventilation to employees:

5.11.1 If container size permits, shipping containers are opened inside of the sample receiving fume hood.

5.11.2 If a shipping container is too large to fit inside of the fume hood, it must be opened directly in front of the fume hood. Containers must be placed on the lift jack, no more than approximately 12” from the face of

the fume hood sash, and raised such that the lid or opening of the container is approximately half way between the lip of the hood and the certified sash opening. The sash is to be opened at the certified sash height during this process.

5.12 All shipping containers from known radiological sites must be surveyed for radiological contamination, on all external surfaces, prior to sample processing. Radiological screening procedures are outlined in section 6.10. For additional information regarding the safe handling of samples from radiological sites, refer to the *Radiation Safety Plan* [HS SOP ME001I4].

6.0 PROCEDURE

6.1 Chain of Custody (COC) – Each sample received into the laboratory must be accompanied by a chain of custody. The chain of custody is a legal document which establishes an unbroken, continuous record of the physical possession, storage, and disposal of samples received into the laboratory. The chain of custody must include the following information:

- Client name
- Sample ID(s)
- Date and time of sample collection
- Sample type (grab or composite)
- Sample matrix (aqueous, solid, non-aqueous, etc.)
- Number of containers
- Sample preservation (H_2SO_4, NaOH, HCl, ice, etc.)
- Analysis requested
- Signatures of all persons who held custody of the sample(s) from the time the sample(s) was collected to the time received by the laboratory
- Signature of the custodian receiving the sample(s) in the laboratory with date and time

6.1.1 Two types of COC records are available for use at Pace-WCOL; one for CWA/NPDES sampling events and one for all other sampling events (Appendix A). The CWA/NPDES COC record includes space for the documentation of composite sampling activities (starting and ending date(s) and time(s) for compositing periods).

6.1.2 Chain of custody records are scanned into LIMS5 at the time of sample log-in and are available for viewing by employees who will process the sample(s).

6.2 Internal Chain of Custody (ICOC) – Security of samples and sample tracking is accomplished via the LIMS 5 internal chain of custody (ICOC) program. When removing a sample from the sample receiving area, any and all bar code labels attached to each sample container associated with tests must be scanned. The scanning process allows LIMS 5 to keep a record of the person who is taking the sample, the date taken, and the location in the laboratory to which the sample is being taken.

6.2.1 For 3M/Norfolk Southern samples, the vendor container ID/lot # must be scanned by sample receiving prior to sample distribution to/pick up by the lab for each sample bottle.

6.2.1.1 In the ICOC application in LIMS 5, sign in to the ICOC system and then click the Enable Vendor Container ID button. The source will default to “Sample Receiving Secure Area” and the cursor will default to the Destination field.

6.2.1.2 Scan in the ICOC location using the ICOC location list provided by QA. This list includes a description of the ICOC location and the sample types associated with that ICOC location. The cursor will move to the Sample ID field.

6.2.1.3 Scan in the Pace-WCOL sample ID. The cursor will move to the Vendor Container ID field.

6.2.1.4 Scan in the vendor container ID/vendor lot # (top/first bar code for QEC bottles).

6.3 Sample Receipt Checklist (SRC) - At the time of receipt of any samples into the laboratory, the custodian receiving the samples initiates a Sample Receipt Checklist (SRC; ME0018C; Appendix B). This form is used to assess sample acceptance criteria, to document the condition of the samples being received, and to note any non-conformances (and its resolution, if possible) associated with them.

6.3.1 When an anomaly is noted, the sample receiving custodian or the project manager (PM) must resolve the deviation internally and/or notify the client to resolve any discrepancy.

6.3.2 Resolutions to any anomalies found during sample receipt must be documented on the SRC to be provided with the final report.

6.4 Sample Storage - Samples must be stored appropriately to maintain preservation, minimize contamination, and ensure sample security within the laboratory.

6.4.1 All samples are stored in clean refrigeration units monitored and maintained between 2.0 and 6.0°C. The temperature of these refrigeration units must be monitored twice daily, except Sundays and holidays with at least a 4-hour interval between the two readings. Sunday and holiday temperatures are read only once. The temperatures of each of the three thermometers (corrected if applicable) are recorded in the Temperature Log (ME003K2, Appendix H).

6.4.1.1 An NCM must be written if the temperature falls outside of the requirements or if a required temperature is not taken. Add a note to the temperature log that an NCM has been written.

6.4.2 Volatile organic samples are stored separately from all other samples. Volatile samples are stored in separate boxes by test as well. These samples are ***ONLY*** stored in the volatiles laboratory and do not require transfer from sample receiving via the ICOC (section 6.2).

6.4.3 Sample security is maintained using an internal sample tracking system called the 'ICOC'. See section 6.2.

6.4.4 A refrigerated rental trailer will be acquired in case of catastrophic failure of all sample storage units. VOA samples will be segregated from non-VOA sample via the use of air-tight containers within the refrigerated unit.

6.4.5 *CLP Sample Storage* – All CLP samples are to be stored in a secure area, accessible only to authorized personnel. CLP samples are not to be tagged with colored dots in order to avoid disposing of the samples early (disposal is not permitted until 100 days have passed).

6.4.5.1 Aqueous CLP samples for Trace VOA analysis and solid samples for VOA testing are stored in their own designated refrigerator in the volatile organic laboratory. Samples for Low-Medium VOA analysis are stored in the VOA walk-in cooler. All other samples for non-VOA analysis are stored in Sample Receiving walk-in cooler.

6.4.5.2 Only Pace-WCOL employees that are required for sample receipt and analysis are listed as authorized personnel and have access to locked storage areas.

6.5 Prioritizing Samples – Upon receipt, coolers are prioritized based on holding times and rush requests. Higher priority is assigned to samples with short holding times (12 hours or less remaining) and samples requiring rush turn-around time. CLP samples shall be prioritized according to hold times (24 hours or less (encores) and turn-around-time noted on the COC.

6.5.1 If a sample holding time is expiring within 24 hours from the time of receipt, sample receiving must communicate sample expiration date and time to the appropriate departmental group supervisor. For samples received on Fridays, Saturdays, and Sundays, the notification extends to any samples expiring within 48 hours. Shorts holds are typically communicated to the department group email as well as the PM via email; however, or other forms of communication may be used.

6.5.2 Cooler contents are organized on the sample receiving counter according to the COC.

6.5.3 All information on the container labels is verified against the COC and this verification is documented on the SRC. If any of the information is absent or incomplete, the anomaly must be documented on the SRC.

6.6 Secure Area – A secure area is defined as an isolated room or refrigerated space that has restricted access limited to Pace-WCOL employees or visitors accompanied by Pace-WCOL employees only. The secure areas used are the sample receiving room, sample receiving walk-in cooler and the volatiles walk-in cooler.

6.6.1 In the absence of a sample receiving custodian, the COC is signed as relinquished to secure area and the date, time and temperature of each cooler are recorded. The sample receiving custodian will receive the samples from the secure area upon arrival.

6.7 Sample Acceptance Policy –This section outlines the necessary information and conditions required for samples to be deemed acceptable upon receipt. All samples received by Pace-WCOL must be compliant with the criteria listed on the Sample Receipt Checklist (Appendix B). If all of the criteria are not met, the laboratory will notify the client to determine if they wish to secure another sample, terminate the analysis of the sample, or continue the analysis. If the sample is processed, then a qualifier will be placed on the final report listing the non-conformance(s) or the non-conformance(s) will be documented in the report narrative. A copy of the SRC is included with each report of analysis. All known radioactive samples must be received with screening data and are to be evaluated for acceptance following the procedures outlined in Section 6.10. The sample acceptance policy includes but is not limited to:

6.7.1 All samples submitted must be accompanied by a Chain of Custody (COC). The COC must be completed and contain at a minimum:

6.7.1.1 Unique Sample ID, location of sampling site, date/time of collection, collector's (sampler's) name, preservation type, sample container type, sample type (grab, composite, solid, aqueous, etc.)

6.7.1.2 Any special remarks concerning the sample.

6.7.2 Sample labels must contain unique sample identification and notated using indelible ink. Sample labels must be durable enough to remain intact and on container in wet or cold conditions.

6.7.3 Samples must be submitted in appropriate containers and preservatives according to the analysis being requested. This includes temperature requirements.

6.7.4 Samples must be submitted with at least one half of the hold time remaining.

6.7.5 Samples must be submitted containing sufficient volume to perform the analysis requested.

6.7.6 Samples must be received in unbroken or undamaged containers. Broken or damaged containers can result in contamination of other samples as well as posing a potential health concern to Sample Receiving personnel.

6.7.7 Upon receipt by the lab, sample submittals are evaluated on the above-mentioned requirements. Any deviation from these requirements will result in the client being contacted by the PM who will inform the client that the data will be qualified if analysis goes forward. The client will then determine if the analysis should go forward.

6.7.8 Any sample submittals that are comprised of commercially available products must be accompanied by a Safety Data Sheet outlining the potential hazardous properties.

6.7.9 If there are any questions regarding container types, preservatives, hold times or any other aspects of sample requirements based on analysis, the PM should be contacted for guidance.

6.8 Reagents

Note: All reagent dilutions are made using reagent water.

Note: Other volumes of standards or reagents may be made to account for expected usage. As long as all ratios are kept constant this is not considered a deviation from this SOP.

Note: All stored reagents are labeled with the following information:

1. **Name of standard or solution**
2. **Concentration**
3. **Analyst's Initials**
4. **Prep date**
5. **Expiration date**
6. **Tracking number**
7. **Warning label of any hazards and/or concentration of acid or base**

6.8.1 Reagent water - Pace-WCOL employs a series of in-house deionized (DI) tanks to purify the incoming water. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks. See the deionized water system SOP (ME0012S) for further information.

6.8.2 Reagent grade chemicals are normally used in sample receiving. Other grades may be used, provided that the reagent is demonstrated to be of sufficiently high purity to permit its use without lessening the accuracy of the determination. Reagents should be stored in glass or Teflon to prevent the leaching of contaminants from plastic containers.

6.8.3 pH test strips – Wide-range, 0-14 SU. Commercially available.

6.8.4 Chlorine test strips – Commercially available, Hach Cat # 2745050 or equivalent.

NOTE: Other concentrations of NaOH, H_2SO_4, HNO_3 and HCl solutions may be used to adjust sample pH provided that the volume added does not change the total sample volume by more than 1%.

6.8.5 Sodium hydroxide solution (10 N), NaOH – Purchased from any manufacturer that can provide a certificate of analysis. Note manufacturer expiration date.

6.8.5.1 Alternatively, while stirring, carefully dissolve 200 g of NaOH in approximately 250 mL of reagent water. Allow the solution to cool and dilute to 500 mL. This reagent expires 6 months from the date of preparation.

6.8.6 Sulfuric acid solution (1:1), H_2SO_4, - Slowly add 250 mL of H_2SO_4 to 250 mL of reagent water. Allow the solution to cool. This reagent expires 6 months from the date of preparation.

6.8.7 Nitric acid solution (1:1), HNO_3 - Slowly add 250 mL of concentrated HNO_3 to 250 mL of reagent water. Allow the solution to cool. This reagent expires 6 months from the date of preparation.

6.8.8 Hydrochloric acid solution (1:1), HCl - Slowly add 250 mL of HCl to 250 mL of reagent water. Allow the solution to cool. This reagent expires 6 months from the date of preparation.

6.9 **Non-CLP Sample Receipt**

Note: Refer to *Foreign Soil and Regulated Domestic Soil Sample Receiving, Laboratory Handling, Disposal and Documentation* (HS SOP ME001J9) for special instructions regarding receiving foreign soils.

6.9.1 *Samples Received via Fed Ex, UPS, or Other Commercial Courier*

6.9.1.1 Examine the shipping containers prior to signing for receipt of packages.

6.9.1.1.1 Examine the shipping containers for damage and to ensure the integrity of the container has not been compromised. Inform the courier of any damages.

6.9.1.1.2 Physically verify that the parcel count matches the courier's parcel count.

3.9.1.1.3. Physically verify that all parcels are addressed to Pace-WCOL.

6.9.1.2 Inspect the shipping container for the presence of custody seals. Note the presence or absence of custody seals on the SRC. If custody seals are present, note their condition on the SRC.

6.9.1.3 Containers that are not securely fastened with tape, or other form of closure, should be noted on the SRC.

6.9.1.4 Open the shipping container under adequate ventilation as described in section 5.11. The container must be opened in this manner to reduce employee exposure to hazardous fumes resulting from potentially broken sample containers.

6.9.1.5 Locate the COC associated with the shipment and remove the courier shipping labels. The shipping labels are maintained along with the COC and SRC. If the label cannot be salvaged, the package tracking # is written on the SRC.

6.9.1.6 ***Immediately*** determine the receipt temperature before the shipping container is unpacked so that samples only stand at room temperature for a minimal amount of time. Receipt temperatures must be determined for each individual shipping container.

NOTE: Norfolk Southern samples (excerpt from Norfolk Southern TSM, Revision 1, Section 1.1.9.1) and 3M samples (excerpt from 3M TSM, Revision 1, Section 1.1.9) – "Samples requiring temperature preservation must not be allowed to reach temperatures > 6°C during sample receipt/login procedures (prior to being placed in laboratory cold storage)." To facilitate this requirement, Norfolk Southern and 3M samples will be placed in bins containing ice for the duration of the login process as soon as they are unpacked from coolers until they are transported to the appropriate storage area.

6.9.1.6.1 The temperature is determined using an infrared (IR) thermometer, or equivalent. The infrared thermometer should be aimed at the sample container label in order to avoid interference from ice or condensation.

6.9.1.6.2 If a temperature blank is provided inside of the shipping container, it ***must*** be used for temperature determination if it is representative of the samples in the shipping container. If the temperature blank is not representative of the samples, or if no temperature blank is present, a *single* representative sample container should be used. The method of determination must be noted on the SRC.

6.9.1.6.3 If a correction factor has been assigned to the IR thermometer, it must be added to or subtracted from the apparent reading every time a temperature is determined. The unadjusted temperature reading, adjusted temperature reading, IR thermometer ID, and correction factor must be noted on the SRC.

6.9.1.6.4 The adjusted temperature reading must be documented on the COC. Additionally, the presence or absence of ice and a temperature blank must be noted on the COC. The "Lab Use Only" section of the COC is designated for the documentation of this information. Information must be documented on each page of the CoC, the SRC, or a cooler temperature receipt label [SR Form ME003V8] which is applied to the SRC.

6.9.1.7 Adjusted temperature readings must be transferred into LIMS5 at the time of sample log-in. This information is recorded in the "edit coolers" window which appears onscreen during the lot creation process. The adjusted temperature must be recorded for each shipping container received. If a receipt temperature is greater than 6°C, LIMS will automatically notify the PM via email. **If samples are received from multiple shipping containers and one or more has a temperature of greater than 6°C, the custodian will isolate the sample containers from those coolers and must document the affected samples. The associated sample containers need to have the cooler number noted in LIMS 5.**

6.9.1.8 When samples are received via a commercial courier, both the "relinquished by" and "laboratory received by" portions of the COC must be completed by the employee receiving the samples. The "relinquished by" space should reflect the method of delivery (FedEx, UPS, etc.)

The relinquish date and time must be indicative of when the shipment was received. The employee receiving the samples must legibly sign the "laboratory received by" portion of the COC. The laboratory receipt date and time must exactly match the relinquish date and time. **NOTE: Initials are not an acceptable signature.**

6.9.2 *Samples Received Directly from The Client*

6.9.2.1 When samples are received directly from a client, the COC must be signed as relinquished by the client and signed as received by the laboratory. The relinquish date and time must reflect the date and time in which the samples are being dropped off. The laboratory receipt date and time must exactly match the relinquish date and time. The client is provided a copy of the CoC.

Note: Any Pace-WCOL **employee may receive samples from a client. Sample receiving summary procedures are posted in the sample receiving area and are included in Appendix E of this procedure.**

6.9.2.2 Open the shipping container under adequate ventilation as described in section 5.11. The container must be opened in this manner to reduce employee exposure to hazardous fumes resulting from potentially broken sample containers.

6.9.2.3 ***Immediately*** determine the receipt temperature as per section 6.9.1.6before the shipping container is unpacked so that samples only stand at room temperature for a minimal amount of time. Receipt temperatures must be determined for each individual shipping container.

6.9.3 *Coolers Delivered by Pace-WCOL Field Services (FS)*

Note: Refer to *Field Services* SOP (FS SOP ME001BS) for further information.

6.9.3.1 When samples are delivered by field personnel and a sample receiving custodian is present, the COC must be signed as relinquished by the field technician and signed as received by the sample receiving custodian. The relinquish date and time must reflect the date and time in which the samples are being dropped off. The laboratory receipt date and time must exactly match the relinquish date and time.

6.9.3.2 When samples are delivered by field personnel and a sample receiving custodian is not present, but it is during normal business hours, the COC must be signed as relinquished by the field technician and signed as received by "Pace-WCOL secure area". The relinquish date and time must reflect the date and time in which the samples are being dropped off. The received by date and time must exactly match the relinquish date and time. Both the "relinquished by" and "received by" portions of the COC must be completed by the field technician.

6.9.3.3 The field technician must immediately determine the receipt temperature of each individual shipping container as per section6.9.1.6.

6.9.3.4 If sufficient ice is present to keep the cooler at ≤ 6°C until the cooler is inspected and unpacked by a sample receiving custodian (no longer than overnight), the cooler(s) may be left in the middle of the sample receiving area. The COC must be left in a conspicuous manner.

6.9.3.5 If there is insufficient ice to maintain a temperature of ≤6°C, the cooler must be placed in the sample receiving walk-in cooler **unless volatile samples are present in the cooler. VOLATILE SAMPLES CANNOT BE STORED IN THE SAMPLE RECEIVING WALK IN COOLER**. **Volatile samples must be placed in the walk-in cooler located in the volatiles department or the cooler should remain in the sample receiving secure area after enough ice is added to the cooler to maintain a temperature of ≤ 6°C. The COC must be placed on the counter in sample receiving with instructions as to where the samples are stored.**

6.9.4 *Samples Received by Lab Personnel Outside Normal Business Hours*

6.9.4.1 Outside of normal business hours, on weekends, and/or on holidays samples may be received by lab personnel in the absence of a sample receiving custodian. The COC must be signed as relinquished by a field technician, commercial courier, or a client and signed as received by the employee receiving the samples. The relinquish date and time must reflect the date and time in which the samples are being dropped off. The received by date and time must exactly match the relinquish date and time.

6.9.4.2 ***The employee must immediately*** determine the receipt temperature of each individual shipping container. The temperature is determined using an infrared (IR) thermometer as per section6.9.1.6.

6.9.4.3 If sufficient ice is present to keep the cooler at ≤ 6°C until the cooler is inspected and unpacked by a sample receiving custodian (no longer than overnight), the cooler(s) may be left in the middle of the sample receiving area. The COC must be left in the sample receiving area in a conspicuous manner.

6.9.4.4 If there is insufficient ice to maintain a temperature of ≤6°C, the cooler must be placed in the sample receiving walk-in cooler **unless volatile samples are present in the cooler. VOLATILE SAMPLES CANNOT BE STORED IN THE SAMPLE RECEIVING WALK IN COOLER**. **Volatile samples must be placed in the walk-in cooler located in the volatiles department or the cooler should remain in the sample receiving secure area after enough ice is added to the cooler to maintain a temperature of ≤ 6°C. The COC must be placed on the counter in sample receiving with instructions as to where the samples are stored.**

6.9.5 *Using the SRC to Assess Sample Acceptance Criteria, Document Sample Condition, and Communicate Non-Conformances*

6.9.5.1 The SRC is a form used to assess sample acceptance criteria, to document the condition of the samples being received, and to note any non-conformances (and its resolution if possible) associated with them.

6.9.5.2 SRC records are scanned into LIMS5 at the time of sample log-in and are available for viewing by employees who will process the sample(s). If a sample condition warrants attention by laboratory personnel, the receiving employee must place a “SEE SRC” label on each applicable sample bottle to prompt the laboratory to reference the SRC for information.

6.9.5.3 The following sample conditions are checked and recorded on the SRC:

6.9.5.3.1 The presence or absence of custody seals. If custody seals are present, note their condition on the SRC. If custody seals are absent, note this on the SRC.

6.9.5.3.2 Sample receipt temperatures - The unadjusted temperature reading and adjusted temperature reading of the sample shipping container(s). The method of determination (temperature blank or against bottle) is also noted on the SRC.

6.9.5.3.3 Receipt of samples collected in containers appropriate for the analysis requested. Sample containers are also observed for condition (unbroken, lids intact, etc.).

6.9.5.3.4 Adequate sample volume for the analysis requested. **Note: Contact PM immediately if inadequate sample volume is received.**

6.9.5.3.5 Adequate hold time remaining for analysis - samples received within ½ the holding time.

6.9.5.3.6 Bubbles present in VOA/RSK-175 vials (compare to a 6 mm comparison device available in the sample receiving area).

6.9.5.3.7 Appropriate chemical preservation for the analysis requested (unless it is not technically acceptable to check preservation upon receipt (e.g. Oil and Grease VOA vials, PFAS drinking water, and microbiology samples)).

6.9.5.3.8 Absence of chlorine where applicable (when NH_3, TKN, Cyanide, Phenol, and/or BNA analysis is requested).

6.9.5.4 Additional quality elements reviewed and recorded on the SRC:

6.9.5.4.1 Receipt and maintenance of a courier packing slip (where applicable).

6.9.5.4.2 Proper custody procedures followed (samples properly relinquished by and received by each party involved).

6.9.5.4.3 The contents of each cooler is checked against the COC.

NOTE: If information on the bottles conflicts with the information on the COC, then the information on the COC takes precedence. All discrepancies must be noted on the SRC.

6.9.5.4.3.1 Sample IDs

6.9.5.4.3.2 Collection date and time (if collection date/time is not found on the COC, this information may be found on the bottles; document this on the SRC)

NOTE: If the sample is a short hold and the date/time is earlier on the bottle than on the COC, the PM must be notified immediately.

6.9.5.4.3.3 Tests to be performed (if tests are not listed on the COC, then log the samples for the tests as per the bottle label and note this on the SRC).

6.9.5.4.3.4 Missing or extra samples (Any extra sample containers received or any missing samples as compared to the COC should be noted on the SRC. Contact the PM for further instructions.)

6.9.5.4.4 Assurance that client requests such as turnaround time, dilutions, analysis hold and separate reports are transcribed from the COC to the comments in LIMS 5.

6.9.5.4.5 Presence of a quote number on sample container(s).

6.9.6 *Thermal Preservation of Samples –*

6.9.6.1 If the samples being received into the laboratory require thermal preservation, the temperature of the sample must be determined and documented upon arrival (Pace-WCOL Analytical Methods list [Administrative Form ME002BS] contains the thermal preservation required for each of the tests performed at Pace-WCOL).

6.9.6.2 Samples that require thermal preservation must arrive at a temperature of less than or equal to 6° C.

6.9.6.2.1 Samples that are hand delivered to the laboratory immediately after collection may not meet these criteria. In these cases, the samples shall be considered acceptable if there is evidence that the chilling process has begun, such as arrival on ice.

6.9.6.2.2 If samples are not logged the day of receipt, the sample custodian must notify the PM ***immediately*** either by phone, email or face-to face if the temperature is greater than 6°C. The PM will contact the client.

NOTE: LIMS 5 will automatically send an email to the PM when thermal preservation requirements are not met.

6.9.7 *Chemical Preservation of Samples –*

6.9.7.1 In addition to thermal preservation, chemical preservation is also required for some analyses. Chemical preservation must be checked at the time of sample receipt for all samples requiring chemical preservation, unless it is not technically acceptable to do so (e.g. Oil and Grease, VOA, PFAS drinking water and microbiology samples). Additionally, if a sample is received with documented information indicating that improper preservation has been used (sample bottle labels denote improper preservation for the tests being requested), the preservation must be checked. **Refer to the Pace-WCOL Analytical Methods list [AD Form ME002BS] for a list of the types of chemical preservation required for each test.**

6.9.7.2 In order to ensure that cross-contamination does not occur, an aliquot of sample is removed from the sample container and that aliquot is used to determine either the pH or chlorine content (TRC) of the sample. **Test strips must not be dipped directly into sample containers.**

6.9.7.2.1 pH paper is used to verify proper pH preservation of samples. Samples requiring acidic conditions are verified in the 0 – 6 SU range. Samples requiring basic conditions are verified with 7 to 14 SU range.

6.9.7.2.2 Chlorine test strips are used to detect the presence of chlorine. The strips are capable of a detection range of 0 – 10 mg/L of free and total chlorine.

6.9.7.3 During preservation verifications, care must be taken to ensure sample run-off is not allowed to spill from test strips onto the benchtop. Sample runoff must be collected in a waste container and managed as required by Pace-WCOL's *Hazardous and Non-Hazardous Waste Management Plan* (ME0012A).

6.9.7.4 If a sample requires adjustment to meet chemical preservation requirements, the sample receiving custodian must notify the PM to resolve the deviation. If agreed upon by the client. the appropriate preservative is added and the chemical ID(s) associated with the chemicals used must be documented on the SRC. Additionally, the ID(s) of the test strips used during preservation verifications and the time of sample preservation must be documented on the SRC.

6.9.7.5 If a sample is unable to be preserved, the receiving employee must note this on the SRC and perform the following actions:

6.9.7.5.1 Document "See SRC" in the comments section of LIMS5 followed by the applicable department. Example: "See SRC (IM)".

6.9.7.5.2 Place a "SEE SRC" label on the applicable sample bottles.

6.9.7.6 The following chemical preservations must be checked upon sample receipt:

6.9.7.6.1 A pH of <2 SU is required for all DRO, metals and nutrient samples.

6.9.7.6.1.1 Samples submitted for metals analysis that require preservation at the time of receipt will be noted in the comments section of the Sample Receiving module in LIMS 5 as "preserved at 'time' on 'date'". This comment will appear on the Metals Prep widget (see "Note" below).

NOTE: For samples to be analyzed by methods 200.7, 200.8 or 245.1 and collected the day of receipt, a 16 hour (drinking water) or 24 hour (non-potable water) waiting period is required after preservation. These samples are preserved by sample receiving and held in the sample receiving walk-in cooler. After the waiting period has expired, the samples are transferred to the metals department where the samples are tested for pH again. If the pH is >2, the samples are further preserved and held again in the metals department for the required waiting period listed above. This procedure will be followed until the pH is <2.

6.9.7.6.1.2 A pH of >12 SU is required for cyanide samples and a pH >9 SU is required for sulfide samples.

6.9.7.6.1.3 SC DHEC drinking water project samples require a pH of <2 SU.

6.9.7.6.1.4 Analyses including ammonia, TKN, cyanide, phenol, and BNA (625.1), (from chlorinated sources such as drinking water), must be verified to ensure that they are free of residual chlorine (< 0.5 mg/L).

6.9.7.6.1.5 If residual chlorine is present at > 0.5 mg/L in samples to be analyzed for cyanide, a "See SRC" label is adhered to the sample container lid. An INM analyst will treat the samples for chlorine according to the appropriate cyanide analysis SOP.

6.9.7.6.1.6 For all other samples, if > 0.5 mg/L of chlorine is present, the sample is de-chlorinated with sodium thiosulfate. Add a small amount of sodium thiosulfate to the sample, mix well and test the chlorine again. Continue until the chlorine is < 0.5 mg/L.

NOTE: Sample preservation adjustments must be performed inside of the fume hood located in the sample receiving area. If a sample reacts and fumes after addition of preservative, it should not be stored in the walk-in cooler.

NOTE: If a sample requires pH adjustment, care must be taken to ensure that the volume of acid or base added does not change the total sample volume by more than 1 %. [For 250 mL sample volumes, no more than 2.5 mL of acid or base may be used for adjustment. For 500 mL sample volumes, no more than 5 mL of acid or base may be used for adjustment. For 1000 mL sample volumes, no more than 10 mL of acid or base may be used for adjustment.]

6.9.8 *Other Preservation*

6.9.8.1 Dissolved Metals

6.9.8.1.1 For dissolved metals analysis, the sample must arrive in the appropriately preserved or non-preserved container according to whether filtration occurs in the field or in the lab.

6.9.8.1.2 For a field filtered dissolved metals sample, the container should be received preserved with nitric acid and the HNO_3 (dissolved) container will be selected in LIMS 5. "Field filtered" must be entered into the comments section of LIMS 5.

6.9.8.1.3 For a dissolved metals sample to be filtered in the lab, the container must be unpreserved (dissolved). The sample receiving custodian must log in the unpreserved container and a nitric bottle to be used when prepping the sample into LIMS 5. "Lab to filter" must be entered into the comments section of LIMS 5.

NOTE: If a sample container is to be logged for dissolved metals, but the sample is unpreserved and the client indicates that the sample has not been filtered in the field, then the sample should not be preserved. Leave the sample as is.

6.9.8.2 Orthophosphate Samples

6.9.8.2.1 Field Services must filter orthophosphate samples in the field and subsequently indicate on the chain of custody that the orthophosphate sample was field filtered. Sample receiving custodians must enter “field filtered” in the comments section of LIMS 5.

6.9.8.2.2 At times, orthophosphate samples may be received that have not been field filtered (client delivered or shipped samples). In this case, Sample Receiving custodians must enter “needs filtered” in the comments in the sample receiving module in LIMS 5 when the sample has not been field filtered.

6.9.9 *Client Requested Information and Special Sample Handling*

6.9.9.1 At times, clients may document requests for special treatment and/or handling of samples in the comments section of the associated chain of custody. When this occurs, the receiving employee should perform the following actions:

6.9.9.1.1 Document “See CoC” in the comments section of LIMS5 followed by the applicable department. Example: “See CoC (EXT)”.

6.9.9.1.2 Place a “See CoC” label on the applicable sample bottles.

6.9.9.1.3 Send an email to the applicable department supervisor.

6.9.10 *Storm Water Samples for Microbiological Analysis*

6.9.10.1 Storm water samples submitted for microbiological analysis require dilution during preparation procedures. If a sample container or chain of custody denotes that a sample is comprised of storm water, the receiving employee must place an “SW” label on the applicable bottle and put “stormwater” in the comments section of LIMS5.

6.9.11 *Inspection of Volatile Samples* - All aqueous volatile samples received in the laboratory must be inspected to verify the presence of air bubbles or headspace. If a sample vial or trip blank vial is found to contain an air bubble greater than “pea-sized” (1/4 inch or 6 mm diameter), document the sample number and number of vials affected on the SRC.

6.9.11.1 Trip blanks may be received with volatile samples. They are to be received in the same manner as volatile samples. Sometimes multiple trip blanks are received from several coolers, but are not labeled uniquely. The custodian will isolate the trip blanks from each cooler and log them in separately, so that one trip blank is received for each cooler that contains volatile samples. If the ID of each set of the trip blanks is not unique, contact the PM for resolution.

6.9.11.2 Several trip blanks may be received in one cooler in which case, consolidate the vials into one sample ID. If trip blanks are received without volatiles, then document this on the SRC.

6.9.11.3 In LIMS 5, trip blanks must be associated with the cooler they were received in. Also, the associated samples for each trip blank set need to have the cooler temperature and cooler number noted in LIMS 5.

6.9.11.4 At times, volatile samples may need to be prepared from samples submitted for testing which arrive in bulk (not in VOA vials). These samples may be poured up in the volatiles lab fume hood or in the sample receiving fume hood.

6.9.12 *Sample Containers* – Pace-WCOL Analytical Methods list [Administrative Form ME002BS] contains a listing of each method that Pace-WCOL performs along with pertinent information such as sample volume, sample containers, holding times, and preservatives. During sample receipt, this information must be confirmed to ensure that all of the method specific requirements are met before analysis can begin.

6.9.12.1 If a sample is received in an incorrect container, note this on the SRC and transfer the sample to the correct container.

6.9.12.2 If a sample container is provided by the client and does not conform to the preservation requirements, the custodian will adjust the pH and record incorrect preservation and adjustments made on the SRC. This is usually done when only one analysis is needed.

6.9.12.3 If multiple analyses requiring different preservatives are requested, the custodian will aliquot the sample into the appropriate containers needed for each analysis from the original after shaking the original container well for 5 seconds.

6.9.12.3.1 If the sample is to remain in the client's original container, the "client provided" container must be selected with the appropriate preservative in LIMS.

6.9.12.3.2 If the sample is transferred to the appropriately-preserved container(s) from the client's original, the final container(s) will be selected in LIMS.

6.9.12.4 For any dissolved analysis, the appropriately sized container labeled "dissolved" in LIMS 5 must be selected. This ensures that the analyst is taking the sample from the proper container.

6.9.12.5 Soil samples may be collected in Encore samplers. When such soil samples are received by the laboratory, the encore container will be selected in LIMS 5 and an extra label is printed for each Encore sampler received. These samples must be prepared and preserved within 48 hours of collection; therefore the ***volatiles lab will require prompt notification*** when any Encore samplers are received. If no one is working in the volatiles lab, then use the volatiles department phone list in order to ensure that a volatiles employee is informed that encore samples have been received. This must be accomplished prior to leaving for the day.

6.9.12.6 If volatiles analysis of soils has been requested and there is no separate sample (soil kit) appointed for this, then an unopened sample jar should be provided to the volatiles department and the volatiles lab should be notified.

6.9.12.7 Any non-conformance (and its resolution if possible) should be documented on the SRC.

6.9.13 *Sub-sampling* – If samples must be split because the client sent an insufficient number of containers for the required analysis, the following procedures apply:

6.9.13.1 Aqueous samples – Shake the parent sample vigorously for at least 5 seconds or more based upon the sample homogeneity. Quickly and carefully pour a portion of the sample into the proper container and preserve appropriately. Document this on the SRC.

NOTE: Refer to the Pace-WCOL Analytical Methods List [ME002BS] for the minimum volume required for analysis. Contact the PM to determine which tests should be poured up if there is not enough sample for all of the tests requested. Some analyses may be run from the same sample volume, but this option should be discussed with the PM and the department supervisor.

6.9.13.2 Non-aqueous liquid samples – Pour single phase liquid into appropriate container. If sample has multiple phases, notify the PM for instructions on how to proceed. If phase separation is required, notify the Operations Manager or Extractions Supervisor. The sample should be separated using an appropriately sized separatory funnel. The phases should be labeled (i.e. top layer, bottom layer, black layer, etc.), placed into the appropriate container and entered into the LIMS system using appropriate description. Document the phase separation on the SRC.

6.9.13.3 Solid samples – See section 6.9.14.

6.9.13.4 If the sample matrix is ambiguous and no matrix has been indicated on the COC, then the sample matrix must be tested. When discerning between aqueous and non-aqueous, a 50 mL digestion tube is filled with water and an aliquot of the sample is added to the water. Observe as to whether the sample mixes with the water or remains completely separate. The results must be reported to the PM who will assist in designating a matrix. If discerning between non-aqueous/aqueous and solid then the PM and the extractions department must be enlisted to test the matrix and a decision made accordingly.

6.9.14 *Handling of Solid Samples* – At times, solid samples may need to be transferred to other sample containers.

6.9.14.1 Free-flowing homogeneous samples such as sand can be transferred to other sample containers using a tongue depressor or by pouring directly into the appropriate containers. Document on the SRC.

6.9.14.2 Non-free-flowing samples and/or non-homogeneous samples such as clay, sediment, etc. are transferred to a larger container such as a one-liter wide mouth glass container and shaken or mixed and then transferred into appropriate containers. Document on the SRC.

6.9.14.3 Percent Solids Determination - All soil and sediment samples are subsampled from the largest parent sample container provided into a 0.5 oz snap-seal plastic container for percent solids determination.

6.9.14.3.1 Initial weight of snap seal container –

6.9.14.3.1.1 *For CLP samples:* Tare the weigh boat on the balance. Then place the % solid label on the weigh boat. Begin weighing each empty snap seal container by placing each one on the weigh boat and recording the weight on the bottom of each snap seal container.

6.9.14.3.1.2 *For non-CLP samples:* Give Inorganic Non-Metals analyst 10 random snap-cup from the new box (new Pace-WCOL lot #) of snap-cups for Inorganic Non-Metals analyst to verify the snap-cups. See ME0013F for specifics.

6.9.14.3.2 *To fill a snap seal container with sample for % solids determination:*

6.9.14.3.2.1 Using a tongue depressor, core a representative sample aliquot from the decanted sample container and transfer 5 to 10 g of sample into the snap-seal plastic container. In the event that there is not enough sample available, use the sample in the volatiles (VOA) screening vial to make a % solid sample. Document that the screening vial was used for % solids. At times a sample is such that a % solid sample cannot be made. When this occurs, document on the SRC that a % solid sample could not be made due to matrix.

6.9.14.4 Percent Moisture Determination – If standing water is observed in a parent sample container, the PM should be contacted prior to sub-sampling for % moisture. The standing water may need to be poured off.

6.9.15 *Compositing Samples* – Some samples are to be composited after they are received by the laboratory.

6.9.15.1 Aqueous samples must be shaken vigorously for at least 5 seconds. The volume required from each sample is poured into a clean, glass container and mixed thoroughly.

6.9.15.1.1 The resultant composite sample is then poured into the appropriate containers for the analyses requested.

6.9.15.2 Solid samples are transferred to a larger container, such as a one-liter wide mouth glass container and shaken or mixed and then transferred into appropriate containers.

6.9.15.3 The date and time that the sample was composited, along with the initials of the compositor will be documented on the SRC.

6.9.15.4 The samples used to make the composite sample are entered into LIMS 5 (as LOT#-001, LOT#-002, LOT#-003…) These samples will not be logged in for any tests, and a comment is added in the comments section identifying them as composite components. Then the composite sample is entered as a separate sample in the same lot with the tests required.

6.9.16 *Entering COC information into LIMS 5*

NOTE: See the *LIMS User Guide* [Admin SOP ME001IS] for instructions on using the LIMS system.

6.9.16.1.1 LIMS 5 is used for project management, scheduling, and for the internal tracking of samples from receipt, through analysis and to final disposal.

6.9.16.1.2 All information on the COC is entered, including the COC number, project name and numbers and PO numbers when applicable.

6.9.16.1.3 The “Notes to SR” section should be reviewed during login. PMs use this section in the quote to provide instruction for sample receiving.

6.9.16.1.4 Samples are assigned a lot number and a barcode label is printed for each sample container. The lot number provides receipt and login information for each sample that is received into the laboratory.

6.9.16.1.4.1 The lot number allows each sample to have unique sample identification (Example: QB27030-001)

6.9.16.1.4.1.1 The first letter of the lot number corresponds to the current year (i.e., P=2014, Q=2015, R=2016, etc.)

6.9.16.1.4.1.2 The second letter corresponds to the current month (i.e., A=January, B=February, C=March, etc.)

6.9.16.1.4.1.3 The first two numbers correspond to the day of the month.

6.9.16.1.4.1.4 The two numbers before the dash indicate the lot numbers received that day, in chronological order, as delivery groups of samples are received.

6.9.16.1.4.1.5 The last three numbers, which are preceded by a dash, reflect the total number of samples included in that lot. For example, JK06001-003 was in the first lot of samples received November 6, 2008 and is the third sample in that lot.

6.9.16.1.4.1.6 The labels also contain a unique bottle identifier that consists of (4) letters and (2) numeric values that change sequentially per container so that no containers have the same ID and can be tracked by the LIMS system to identify the location of each individual container.

6.9.16.1.5 The laboratory generated bar code labels are applied to the sample containers in sample receiving.

6.9.16.1.6 Further assessment is made concerning holding times, as it is imperative that samples are analyzed within the stipulated maximum holding time.

6.9.16.1.6.1 Pace-WCOL is responsible for meeting all holding times for properly preserved samples received within 48 hours of collection, or within one half the holding time, whichever is shortest.

6.9.16.1.6.2 Samples with short holding time left prior to expiration, such as those for BOD, fecal coliform, hexavalent chromium, etc., require immediate notification of the laboratory to ensure analysis within the holding time.

6.9.17 *Sample Receiving Review* – A final review by the sample receiving custodian is required after the samples have been entered into LIMS 5 and before proceeding to the next lot. The custodian will perform a cursory on-screen level I review of the lot and promote from "Lot Creation" to "Sample Receiving Review". A level 1 review of the lot includes:

6.9.17.1 Ensuring that all information from the COC has been entered into LIMS correctly and completely. When using the Clone Lot tool, extra care must be taken to review all information matches.

6.9.17.2 Ensuring that the SRC has been filled out completely and appropriately.

NOTE: Once a cooler has been emptied, remove all labels and packing materials (bubble wrap, etc.) from the outside of the cooler which were applied during prior usage of the cooler. It is especially important to remove any radiation safety labels prior to the next usage.

6.9.18 *Sample Receiving Communication* – During the login process, questions or concerns must be addressed verbally with the PM. When PMs are not available, notification of any questions or concerns are communicated via email or face-to-face. Any questions or concerns about rush or short holds must be verbally communicated to the PM immediately.

6.9.18.1 Email should be used at a minimum for questions or concerns that are not detrimental to the performance of the lab and will not hinder any analytical processes from occurring. This communication is not used for non-conformances. Non-conformances (and its resolution if possible) must be documented on the Sample Receipt Checklist.

6.10 **Radioactive Sample Handling: DOE / Savannah River Site (SRNS) H-16 Special Handling**

6.10.1 Based on screening done by SRNS at the H-16 site on samples intended for analysis at SES, notification is sent to the lab confirming that the tritium concentration in those samples is less than or equal to 20 micro curies/L. If this level is exceeded then the samples will not be sent to the lab and if they are inadvertently sent, they are to be *immediately* returned. All H-16 site samples are ultimately returned after analysis regardless of tritium levels.

6.10.2 In accordance with South Carolina Department of Health and Environmental Control (SCDHEC) Radioactive Materials License No 426, Pace-WCOL must perform surveys of all incoming samples authorized by the license.

6.10.2.1 Any sample measuring at or above 20 counts per minute (CPM) must be immediately returned to the original sender *without* being opened or processed. Samples must be segregated, in a designated location (beside the fume hood in SR), and held for pick-up by the sender.

6.10.3 When a shipping container is determined to contain samples requiring survey, the shipping container and samples may not be processed until the following procedure is complete and recorded using the Radioactive Materials Survey Log [HS Form ME001O3; Appendix I]. Once completed, save the survey log on the public drive (P:\Health and Safety\Radioactive Materials Receipt).

6.10.3.1 This procedure fulfills the requirements of Section RHA 3.26 of the State of South Carolina's Regulation 61-63 concerning radioactive materials.

6.10.3.2 The procedure should be followed as soon as possible after receipt but no more than 3 hours once the shipping container is received at the facility during normal business hours, or no more than three hours from the beginning of the next working day if received after working hours.

6.10.3.3 See Appendix G for details regarding the use of surveying equipment.

6.10.3.4 Before the shipping package is opened, all documentation such as the COC and shipping papers are reviewed to ensure the shipment is proper. Acknowledgement of complete documentation is noted on the attached Radioactive Materials Survey Log (RMSL).

6.10.3.5 The exterior of the shipping container is examined for integrity and signs of physical leakage. The condition is noted on the RMSL.

6.10.3.6 **Background** - Hold the pancake probe in the air to survey for background radiation using the survey meter as listed below. The results are recorded on the RMSL.

Survey A - Beta and Gamma
Meter - Pancake Probe Model 44-9 Geiger Counter

6.10.3.7 **Exterior -** The exterior of the package is surveyed for radiation "leakage" per 10 CFR 71.47 and “removable” radiation per 10 CFR 71.87 (i) with survey meter listed above. All external surfaces of the package must be surveyed by holding the pancake probe over the exterior of the cooler. The results are recorded on the RMSL.

6.10.3.8 If removable radiation readings are less than 20 CPM, the shipping package is opened in the following manner and the integrity of the sample containers is visually checked and noted on the RMSL.

6.10.3.9 If the shipping container size permits, shipping containers will be opened inside of the sample receiving hood. If a shipping container is too large to fit inside of the fume hood it must be opened directly in front of the fume hood. Shipping containers must be placed on the lift jack, no more than approximately 12” from the face of the fume hood sash, and raised such that the lid of the shipping container is approximately half way between the lip of the hood and the certified sash opening. This sash is to be opened at the certified sash height during this process.

6.10.3.10 **Interior** - The interior of the package is then surveyed for "removable" radiation by holding the pancake probe over the interior of the cooler 6.10.3.7 above). The results are recorded on the RMSL.

6.10.4 If all readings (steps6.10.3.7 and 6.10.3.10) are within limits (≤20 cpm), the samples are accepted and may be removed from the shipping container and received via the procedures above.

6.10.4.1 All sample containers must be logged into LIMS 5 using “RAD” bottle types, which allows for samples to be appropriate labeled with a “RAD” designation and prevents improper sample disposal. All sample containers must be logged into LIMS 5 using the “RAD” bottle type.

6.10.4.2 The designated storage and custody area for radiological samples is in sample receiving walk-in cooler #1.

6.10.5 If any of the meter readings from either step 6.10.3.7 or step 6.10.3.10 exceeds the limits, or other abnormalities are detected such as a damaged shipping container, the Laboratory General Manager and the Radiation Safety Officer must be summoned immediately. The Radiation Safety Officer must report the exceedance to the DHEC Bureau of Radiological Health in an expeditious manner. The shipping container and its contents will be properly secured and isolated awaiting disposition.

6.10.6 See section 6.12.3 for return of radioactive samples.

NOTE: Once a cooler or reusable shipping container has been emptied, remove all labels from the outside of the cooler which were applied during prior usage of the cooler. It is especially important to remove any radiation safety labels prior to the next usage.

6.11 **Subcontracted/IR Sample Transfer -** PACE-WCOL does not subcontract analytical analysis without prior notification to the client. When samples are subcontracted or IR transferred, a new COC is issued to the subcontract/IR laboratory with all pertinent information and a copy is kept for tracking purposes. PACE-WCOL retains the original COC and includes the new subcontracted COC with the project file. The samples should be scanned out as “Subcontracted” in the ICOC system prior to shipping.

6.11.1 Prior to subcontracting any analyses, PACE-WCOL will confirm that the laboratory being considered to receive samples is fully qualified and certified by the regulatory agency necessary to conduct the analyses on those samples. Prior to IR transfer, PACE-WCOL will confirm the IR laboratory has the appropriate certification. See the *Project Management* [Admin SOP ME001HD] for further information.

6.11.2 A sample that is being subcontracted or IR transferred should be placed in a cooler with sufficient ice to maintain the proper thermal preservation.

6.11.2.1 Ice should be double-bagged to prevent leakage and placed on the bottom and sides of the cooler, at a minimum.

6.11.2.2 A temperature blank must be added to each cooler and should be in direct contact with the ice.

6.11.2.3 The sample should be well protected by packing the cooler with enough packing material to ensure that the sample will not shift during shipment. This is to protect the sample from breakage.

6.11.2.4 Place the COC in a plastic bag and place inside the cooler.

6.11.2.5 Place custody seals on the cooler

6.11.2.6 Tape the cooler with a sufficient amount of packing tape to ensure that the cooler will not open during shipment.

6.11.2.7 Place the appropriate shipping label on the outside of the cooler and leave the cooler in the designated courier pickup area (FedEx Express is picked up from the front of the building outside the lobby, FedEx Ground / UPS is picked up from outside the SR doors at the side of the building)

NOTE: If the sample(s) to be subcontracted are hazardous, then contact the EHSO concerning the proper shipping placarding.

6.11.3 When samples are received that require a subcontracted or IR transfer analysis, the test is logged in with the appropriate subcontract test and a notification is sent to the PM via email. The PM fills out a Subcontract Shipping Record Form or an IR Form and returns it to Sample Receiving. Alternatively, the PM may provide the information in an email. The information should include the lot number, PM, the subcontractor lab, and the required analysis. SR will provide the courier's tracking number and the date the sample was subcontracted. This allows the PM to verify that the subcontracted analysis has been sent to the appropriate lab and to track the sample shipment.

6.11.3.1 If samples are subcontracted via field services, then the following must be done:

6.11.3.1.1 Columbia field service: Samples must be placed in a bag/box labeled with the name of the subcontract lab. The field services supervisor must be informed that subcontract is ready for pick up.

6.11.3.1.2 Charlotte and Greenville field services: The Charlotte or Greenville office must be contacted ahead of time in order to arrange for pick-up of the subcontracted samples.

6.12 **Sample Return/Disposal** - After preparation and analysis have been completed on a sample, it is returned to storage. Most samples are kept for a minimum duration of four (4) weeks before disposal. Others are kept longer per client request. The determination of what samples are ready to be disposed of is based on a color code system. Using a color code chart, the custodian removes and disposes of the samples received approximately during the sixth week prior to the current week (soils are dumped during the sixth week or afterwards). Exceptions to this include samples whose holding times are short and those that are consumed during analysis.

6.12.1 Regardless of the method of disposal or final disposition, the event is recorded via the LIMS 5 ICOC system.

6.12.2 Samples are either returned to clients or disposed of as either hazardous or non-hazardous waste via the ICOC. The PACE-WCOL *Hazardous and Non-Hazardous Laboratory Waste Management Plan* [HS SOP ME0012A] contains the proper procedure as to the handling of sample disposal or transfer back to the client. In the event samples are part of a litigation process, disposal of the samples must only occur with the written approval of the legal authority, sample submitter, or sample data user. The following types of samples are returned to the client: samples containing PCB hits between 50 and 500ppm, radioactive samples, or if the client has requested the return of their samples.

6.12.3 Radioactive samples, client requested samples - These samples should be labeled with a "Return to Client" label upon receipt of the sample. Such samples will be returned with a new COC, filled out by sample receiving, listing the sample IDs, collection time/date, reason for return and lot #. These samples must be scanned out as Returned to Client in the ICOC. Samples requiring return must be pulled out of circulation and isolated in the designated return to client bin and listed as being held on the return to client list. The PM should be notified of samples that need to be returned to the client.

6.12.3.1 All residual sample remaining for 3M samples must be returned to the client. A unique bottle type must be used for 3M samples. When a sample that is not consumed is scanned for disposal, LIMS will alert the user that this sample needs to be returned to the client.

6.12.4 Samples that have a PCB detection less than 500 ppm or samples for which the EHSO has given approval may be disposed at PACE-WCOL and placed in the PCB waste stream.

6.12.5 If returning H-16 samples to the Savannah River Site (SRS), then an H-16 form must be filled out, and the PM must be notified when samples are ready for return by field services.

6.13 **US EPA CLP Sample Receipt**

6.13.1 *Chain of Custody* – Each sample received must have a COC or a Traffic Report (TR). The information listed below should be available with each sample:

- Case Number
- CLP Sample Numbers
- Sample Matrix
- Concentration
- Sample Type
- Preservative
- Analysis
- Tag Numbers
- Station Location Identifier
- Collection Date/Time
- Sampler Initials
- Shipment for Case Complete
- Air bill Number
- COC Number

6.13.2 *Form DC-1* – At the time of sample receipt, an original Form DC-1 is completed contemporaneously for each sample shipping container. The form, as well as the air bill, is signed and dated by the sample custodian. The sample custodian records the cooler receipt temperature on both the DC-1, the chain of custody, and the associated air bill. Form DC-1 is completed to verify the presence or absence and the condition of the custody seals, air bills, COCs, TRs, sample tags, cooler temperature blanks and to verify the pH of any preserved samples. Any anomaly or discrepancy between the COC/TR and the sample labels is documented on Form DC-1. Also noted is the condition of the samples upon receipt, and the date and time of receipt.

NOTE: A separate DC-1 form must be filled out for ISM, SOM, and SFAM samples. See Appendix C for the ISM, SOM, and SFAM DC-1 forms.

6.13.3 *Sample Custody* – A sample is considered in custody if:

- It is in a person's possession; or
- It is in view after being in possession; or
- It is locked in a secure area after being in possession; or
- It is in a designated secure area, accessible only to authorized personnel.

6.13.4 *Sample Receiving* – A designated sample custodian (or designated backup custodian) is responsible for receiving all USEPA CLP samples.

6.13.4.1 A thorough exterior check of the shipping container is performed to ensure that the custody seals are present and intact. Before cutting the custody seals on a cooler, inspect the seal for any custody seal numbers. These custody seal numbers are required to be listed on the DC-1 form for the cooler shipment.

6.13.4.2 The sample cooler is then opened in the following manner to ensure that the person opening the cooler is not exposed to any fumes from potentially broken samples.

6.13.4.2.1 If the cooler size permits, coolers will be opened inside of the sample receiving hood. If a cooler is too large to fit inside of the fume hood it must be opened directly in front of the fume hood.

6.13.4.2.2 Coolers must be placed on the lift jack, no more than approximately 12” from the face of the fume hood sash,and raised such that the lid of the cooler is approximately half way between the lip of the hood and the certified sash opening. This sash is to be opened at the certified sash height during this process.

6.13.4.3 The temperature is immediately determined from the temperature blank, if present, before the cooler is unpacked. If a temperature blank is not present, the temperature will be taken from a *single* representative sample. The number of coolers must be recorded in LIMS along with their associated temperatures.

6.13.4.4 Prioritize CLP samples according to hold times (24 hours or less (encores) and turn-around-time required.

6.13.4.5 The cooler contents are organized according to the COC/TR and all information is verified on the container labels and sample tags if present.

6.13.4.6 The sample custodian signs, dates and records the time on all forms (COCs, original TRs and air bills). The sample custodian also completes Form DC-1.

6.13.4.7 In the “sample condition” field on the COC/TR and the DC-1, the sample receiving custodian must note if samples are missing, pH requires adjustment, VOA vials contain air bubbles greater than 6 mm, sample containers are received broken , if there is a label or tag discrepancy or if there is any other discrepancy.

Note: Initials are not acceptable for signing forms.

6.13.5 *Sample Delivery Group (SDG)* - The samples are assigned to a SDG at the time of receipt.

6.13.5.1 The SDG number is the lowest alphanumeric EPA sample number received in the SDG. When several samples are received together in the first shipment, the SDG number will be the lowest sample number (considering both alpha and numeric designations) in the first group of samples received under the SDG. The same sample may be in an SDG in an ISM and an SOM lot (for example: the SOM sample designation is D03A1 and the ISM sample designation is MD03A1).

6.13.5.2 An SDG is assigned for each lot of field samples received for a case, a maximum of 20 field samples within a case, or each 7-calendar day period (counting the receipt date) during which field samples in a case are received. For example: samples first received on Thursday are placed in an SDG which may also include samples received through the following Wednesday.

For EPA Cases scheduled for a 7-day TAT, samples can only be added to the SDG for a period of 4 calendar days (counting the receipt date).

NOTE: Samples must not be added into an SDG on the 8th day (counting the receipt date) unless given permission by the CLP PM or the Technical Director.

6.13.5.3 If an EPA case requires designated QC (noted on EPA schedule), then solid samples and aqueous samples need to be in separate SDGs.

6.13.5.4 If an EPA inorganic case has both total metals and/or mercury and dissolved/filtered metals and/or mercury, the total and dissolved samples need to be in separate lots/SDGs in LIMS5.

6.13.5.5 The sample receiving custodian will check for any e-mails from the technical director and/or CLP PM and if further information is required, the custodian will check the CLP Case Schedule located in the following location: P:\Lab Status\CLP_Case_Status\CLP_Scheduling before logging samples into LIMS.

6.13.5.6 If there are multiple modified analyses (MA) for one parameter, then each MA is placed into a separate SDG. Multiple MAs for different parameters may remain in the same SDG.

6.13.5.7 CLP Performance Evaluation (PE) samples **must be excluded** from the count of 20 samples comprising an SDG.

6.13.5.7.1 If PE samples arrive ahead of their associated samples, then PE sample(s) will be logged into LIMS5 without any tests until the associated samples arrive. The PE samples will be labeled and placed in the Receiving walk-in cooler. If the PE samples arrive on the same receipt date as the associated samples then log the PE samples into the SDG first. If there are over 20 samples and multiple PE samples have been received, then the PE samples are to be split among the sample lots. PE samples are not included in the designation of an SDG. The lowest alphanumeric regular sample must be used.

6.13.5.7.2 In LIMS 5 the sample type must be changed to PT Sample, the PE box should be checked, and “PT” needs to be written in the comments section.

6.13.5.8 Additionally, if a sample with the same sample ID, but different analysis is received while the SDG is open during a 7 calendar day period, it will always be added to the same SDG containing that same ID, but as a separate line item to indicate that it was received on a different date/time.

NOTE: If a sample with the same sample ID requiring % solids is received after the initial receipt date, then % solids is logged in for the sample as mentioned above and a note stating that the % solids result should be obtained from the original sample is added to the comments.

6.13.5.8.1 The information is then entered into the PACE-WCOL LIMS and a unique laboratory lot number is assigned to the samples. All sample tags, if present, are collected according to the SDGs and must remain with the COC/TR. Sample tags are recorded in the DC-1 form as well. Each tag is placed in a bag labeled with the SDG #. The tags are placed in order inside the bag as they are listed in the

SDG. Sample tags for the same Case/SDG for multiple receipt days will go into one labeled bag.

6.13.5.9 A storage blank associated with volatile organic samples is logged into the LIMS and prepared for each SDG. Note: If a SDG has two volatile analyses, then two holding blanks will need to be logged into the SDG (e.g. TVOA and TVOA SIM). A blank is made for each VOA analysis/fraction, matrix and MA (modified analysis).

6.13.5.9.1 Aqueous storage blanks are prepared as follows: Three preserved vials are filled with volatile-free water (from the VOA lab) and stored with the samples from the SDG.

6.13.5.9.2 Solid storage blanks are prepared as follows: 5 mLs of volatile free water (from the VOA lab) is placed into each of three unpreserved VOA vials. A stir bar is added along with 5 grams (not more than 5.10 g) of Ottawa sand.

6.13.5.10 Ensure that the sample type is changed in LIMS5 for any holding blanks, PT samples, equipment blanks, rinsate blanks, field blanks, and trip blanks.

6.13.5.11 For organic MS/MSDs, check the appropriate samples under the analysis MS column in order to designate which sample should have an MS/MSD run on it. This must also be recorded first in the comment section in LIMS. If more than one designated QC (MS/MSD) is provided, make sure that they are logged into two separate SDGs. If a case requires designated lab QC and nothing is designated on the COC/TR contact the CLP PM or the technical director before logging in samples.

NOTE: EPA does not use the letter “O” therefore CLP Sample IDs must always be entered into the LIMS as a zero (“0”)

6.13.6 *Verification of Aqueous Sample Preservation* **–** At the time of sample receipt, sample receiving checks the pH of the sample and notes on Form DC-1 if the pH is ≤ 2 SU for metals or is ≥ 10 SU for cyanide samples. If cyanide sample pH(s) are not in range, the sample custodian needs to alert the CLP PM right away before preserving. Note the exact pH of the sample (example 1.5 SU), record the chemical IDs of the preservative and the pH strips used as well as preservation time and volume of preservative added.

6.13.6.1 If a metals sample has not been properly preserved, sample receiving will adjust the pH of the metals sample. See section 6.9.7.4. This will be noted in the SDG narrative.

6.13.6.2 Cyanide samples are not pH adjusted. The Sample Management Office (SMO) will be contacted for further instructions before proceeding with the preparation and analysis.

6.13.6.3 The pH of TCLP and SPLP samples will not be adjusted.

6.13.7 *Sample Receiving Review* – A final review by the sample receiving custodian occurs after the samples have been entered into LIMS 5 and before proceeding to the next lot. The custodian will perform a cursory on-screen level I review of the lot but will not promote the lot. A level 1 review of the lot includes:

6.13.7.1 Ensuring that all information from the COC has been entered into LIMS correctly and completely.

Ensuring that the TR/COC and the DC-1 has been filled out completely and appropriately. The CLP PM or designee enters the SDG number, case number, and laboratory contract number on the COC.

7.0 RESPONSIBILITIES

7.1 The sample receiving department consists of four full-time designated sample receiving custodians. Several other PACE-WCOL employees are cross-trained in this department.

7.2 It is the responsibility of the sample receiving custodians, and anyone assisting in the receipt of samples, to adhere to the procedures summarized in this SOP.

8.0 ATTACHMENTS

8.1 Appendix A: Chain of Custody

8.2 Appendix B: Sample Receipt Checklist

8.3 Appendix C: Form DC-1

8.4 Appendix D: Sample Storage Locations

8.5 Appendix E: Sample Receiving – Summary of Procedures

8.6 Appendix F: Subcontract Shipping Record

8.7 Appendix G: Radiological Survey Meter Operations Guidance

8.8 Appendix H: Temperature Log

8.9 Appendix I: Radioactive Materials Survey Log

8.10 Appendix J: Hazard Communication Standard Pictogram

8.11 Appendix K: IR Gun Thermometer Quarterly Calibration Log

8.12 Appendix L: IR Gun Thermometer Daily Verification Log

8.13 Appendix M: Cooler Temperature Receipt Labels

8.14 Appendix N: Balance #13-1224 Verification Log

9.0 REFERENCES

9.1 Methods for the Chemical Analysis of Water and Wastes (MCAWW), EPA-600/4-79-020, March 1983.

9.2 US EPA CLP SOW SOM Organic Superfund Analysis, Multi-Media, Multi-Concentration, as referenced in the QAMP (ME0012K).

9.3 US EPA CLP SOW ISM Inorganic Analysis, Multi-Media, Multi-Concentration, as referenced in the QAMP (ME0012K).

9.4 US EPA CLP SOW SFAM, Multi-Media, Multi-Concentration, as referenced in the QAMP (MEK0012K).

9.5 PACE-WCOL Quality Assurance Management Plan (QAMP) – ME0012K.

9.6 EPA's SW-846 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Revision 3, December 1996.

9.7 DoD/DOE Quality Systems Manual (QSM) for Environmental Laboratories, as referenced in the QAMP (ME0012K).

9.8 "United States Department of Labor." *OSHA QUICK CARD: Hazard Communication Standard Pictogram*. Occupational Safety and Health Administration, n.d. Web. 24 Jan. 2017.

9.9 Norfolk Southern Technical Specifications Manual, Revision 1.

9.10 3M Technical Specifications Manual, Revision 1.

10.0 REVISION HISTORY

Revision #	Revision Date	Section Modified	Modification	Reason for Modification
-17	**09/20/2021**	6.10.6	Updated procedure for use of pancake probe and added sample storage location	ANAB audit finding #6

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Sample Receiving
ISSUER: Pace ENV - Local Quality - WCOL

APPENDIX A: CHAIN OF CUSTODY STANDARD

Pace Analytical® **Chain of Custody Record**

Pace Analytical Services, LLC.
106 Vantage Point Drive
West Columbia, South Carolina 29172
Telephone No. (803) 791-9700 Fax No. (803) 791-9111
www.pacelabs.com

Number

Client	Report to Contact	Telephone No. / E-mail	Quote No.
Address	Sampler's Signature	Analysis (Attach list if more space is needed)	Page ___ of ___
City / State / Zip Code	X _____ Printed Name		Lot # Bar Code (lab use only)
Project Name			

Project Number	P.O. No.		G=Grab C=Composite	Matrix				No of Containers by Preservative Type															
Sample ID / Description (Containers for each sample may be combined on one line)	Collection Date(s)	Collection Time (military)		Aqueous	Solid	Non-Aqueous		Unpres.	H2SO4	HNO3	HCl	NaOH	5035 Kit	Field Filtered									Remarks / Cooler I.D.

Turn Around Time Required (Prior lab approval required for expedited TAT)	Sample Disposal	Possible Hazard Identification (List any known hazards in the remarks)	QC Requirements
☐ Standard ☐ Rush (Please Specify)	☐ Return to Client ☐ Disposal by Lab	☐ Non-Hazardous ☐ Flammable ☐ Skin Irritant ☐ SDS provided ☐ Unknown	

1. Relinquished by	Date	Time	1. Received by	Date	Time
2. Relinquished by	Date	Time	2. Received by	Date	Time
3. Relinquished by	Date	Time	3. Received by	Date	Time
4. Relinquished by	Date	Time	4. Laboratory Received by	Date	Time

Note: All samples are retained for four weeks from receipt unless other arrangements are made	LAB USE ONLY Received on Ice (Check) ☐Y ☐N ☐Ice Pack	Receipt Temp. ____ ° C	Temp. Blank ☐Y / ☐N

Document Number: ME003Q3-01

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Sample Receiving
ISSUER: Pace ENV - Local Quality - WCOL

APPENDIX A CONT'D: CHAIN OF CUSTODY CWA/NPDES

CWA / NPDES Chain of Custody Record

Pace Analytical Services, LLC.
106 Vantage Point Drive
West Columbia, South Carolina 29172
Telephone No. (803) 791-9700 Fax No. (803) 791-9111
www.pacelabs.com

Number

Client	Report to Contact	Sampler (Printed Name)	Quote No.
Address	Telephone No. / Email	Field parameters (i.e., pH, temp, DO) can be recorded below	Page ___ of ___
City / State / Zip Code	**Preservative** 1. Unpres. 4. HNO3 7. NaOH/ZnA 2. NaOH 5. HCL 3. H2SO4 6. Sodium Thiosulfate	Analysis	Number of Containers Container Type: P=Plastic G=Glass Preservative (use code on left)
Project Name			Lot # Bar Code (lab use only)
Project Number / P.O. Number	G=Grab C=Composite / Collection Sample Temp °C / Chlorinated Y/N / **Matrix** (GW DW WW HW S=Solid)		
Sample ID / Description (Containers for each sample may be combined on one line) / Collection Date(s) / Collection Time (military)			Remarks / Cooler ID
Start / Finish			
Start / Finish			
Start / Finish			
Start / Finish			
Start / Finish			

Turn Around Time Required (Prior lab approval required for expedited TAT)	Sample Disposal	QC Requirements	Possible Hazard Identification (List any known hazards in the remarks)
☐ Standard ☐ Rush (Please Specify)	☐ Return to Client ☐ Disposal by Lab		☐ Non-Hazardous ☐ Flammable ☐ SDS Provided ☐ Skin Irritant ☐ Unknown

	Date	Time		Date	Time
1. Relinquished by / Sampler			1. Received by		
2. Relinquished by			2. Received by		
3. Relinquished by			3. Received by		
4. Relinquished by			4. Laboratory Received by		

Note: All samples are retained for four weeks from receipt unless other arrangements are made

LAB USE ONLY
Received on Ice (Check) ☐Y ☐N ☐Ice Pack

Receipt Temp. _____ ° C

Temp. Blank ☐ Y / ☐ N

Document Number: ME003Q4-01

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Sample Receiving
ISSUER: Pace ENV - Local Quality - WCOL

APPENDIX B: SAMPLE RECEIPT CHECKLIST

Samples Receipt Checklist (SRC) (ME0018C-15) Revised:9/29/2020
Issuing Authority: Pace ENV - WCOL Page 1 of 1

Sample Receipt Checklist (SRC)

Client: _____ Cooler Inspected by/date: _____ / _____ Lot #: _____

Means of receipt: ☐ Pace ☐ Client ☐ UPS ☐ FedEx ☐ Other: _____

☐ Yes	☐ No		1. Were custody seals present on the cooler?
☐ Yes	☐ No	☐ NA	2. If custody seals were present, were they intact and unbroken?

pH Strip ID: _____ Chlorine Strip ID: _____ Tested by: _____
Original temperature upon receipt / Derived (Corrected) temperature upon receipt %Solid Snap-Cup ID: _____
_____/_____°C _____/_____°C _____/_____°C _____/_____°C
Method: ☐ Temperature Blank ☐ Against Bottles IR Gun ID: _____ IR Gun Correction Factor: _____°C
Method of coolant: ☐ Wet Ice ☐ Ice Packs ☐ Dry Ice ☐ None

☐ Yes	☐ No	☐ NA	3. If temperature of any cooler exceeded 6.0°C, was Project Manager Notified? PM was Notified by: phone / email / face-to-face (circle one).
☐ Yes	☐ No	☐ NA	4. Is the commercial courier's packing slip attached to this form?
☐ Yes	☐ No		5. Were proper custody procedures (relinquished/received) followed?
☐ Yes	☐ No		6. Were sample IDs listed on the COC?
☐ Yes	☐ No		7. Were sample IDs listed on all sample containers?
☐ Yes	☐ No		8. Was collection date & time listed on the COC?
☐ Yes	☐ No		9. Was collection date & time listed on all sample containers?
☐ Yes	☐ No		10. Did all container label information (ID, date, time) agree with the COC?
☐ Yes	☐ No		11. Were tests to be performed listed on the COC?
☐ Yes	☐ No		12. Did all samples arrive in the proper containers for each test and/or in good condition (unbroken, lids on, etc.)? _____
☐ Yes	☐ No		13. Was adequate sample volume available?
☐ Yes	☐ No		14. Were all samples received within ½ the holding time or 48 hours, whichever comes first?
☐ Yes	☐ No		15. Were any samples containers missing/excess (circle one) samples Not listed on COC?
☐ Yes	☐ No	☐ NA	16. For VOA and RSK-175 samples, were bubbles present >"pea-size" (¼" or 6mm in diameter) in any of the VOA vials?
☐ Yes	☐ No	☐ NA	17. Were all DRO/metals/nutrient samples received at a pH of < 2?
☐ Yes	☐ No	☐ NA	18. Were all cyanide samples received at a pH > 12 and sulfide samples received at a pH > 9?
☐ Yes	☐ No	☐ NA	19. Were all applicable NH_3/TKN/cyanide/phenol/625.1/608.3 (< 0.5mg/L) samples free of residual chlorine?
☐ Yes	☐ No	☐ NA	20. Were client remarks/requests (i.e. requested dilutions, MS/MSD designations, etc…) correctly transcribed from the COC into the comment section in LIMS?
☐ Yes	☐ No		21. Was the quote number listed on the container label? If yes, Quote # _____

Sample Preservation (Must be completed for any sample(s) incorrectly preserved or with headspace.)

Sample(s) _____ were received incorrectly preserved and were adjusted accordingly in sample receiving with _____mL of circle one: H2SO4, HNO3, HCl, NaOH using SR # _____.
Time of preservation _____. If more than one preservative is needed, please note in the comments below.

Sample(s) _____ were received with bubbles >6 mm in diameter.

Samples(s) _____ were received with TRC > 0.5 mg/L (If #19 is ***no***) and were adjusted accordingly in sample receiving with sodium thiosulfate ($Na_2S_2O_3$) with Shealy ID: _____.

SR barcode labels applied by: _____ Date: _____

Comments:

APPENDIX C: FORM DC-1 -SOM02.4

FORM DC-1
SAMPLE LOG-IN SHEET

Lab Name		Page of
Received By (Print Name)		Log-in Date
Received By (Signature)		
Case Number	SDG No.	MA No.

Remarks:	
1. Custody Seal(s)	Present/Absent* Intact/Broken
2. Custody Seal Nos.	_____ _____
3. Traffic Reports/Chain of Custody Records or Packing Lists	Present/Absent*
4. Airbill	Airbill/Sticker Present/Absent*
5. Airbill No.	_____ _____
6. Sample Tags Sample Tag Numbers	Present/Absent* Listed/Not Listed on Traffic Report/Chain of Custody Record
7. Sample Condition	Intact/Broken*/ Leaking
8. Shipping Container Temperature Indicator Bottle	Present/Absent*
9. Shipping Container Temperature	_____
10. Does information on Traffic Reports/Chain of Custody Records and Sample Tags agree?	Yes/No*
11. Date Received at Lab	_____
12. Time Received	_____

	EPA Sample #	Corresponding Sample Tag #	Corresponding Assigned Lab #	Remarks: Condition of Sample Shipment, etc.
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				

* Contact SMO and attach record of resolution

Reviewed By	Logbook No.
Date	Logbook Page No.

SOM02.4 (10/2016) Form DC-1

APPENDIX C CONT'D: FORM DC-1 -ISM02.4

FORM DC-1
SAMPLE LOG-IN SHEET

Lab Name		Page of
Received By (Print Name)		Log-in Date
Received By (Signature)		
Case Number	SDG No.	MA No.

Remarks:	
1. Custody Seal(s)	Present/Absent* Intact/Broken
2. Custody Seal Nos.	_____ _____
3. Traffic Reports/Chain of Custody Records or Packing Lists	Present/Absent*
4. Airbill	Airbill/Sticker Present/Absent*
5. Airbill No.	_____ _____
6. Sample Tags Sample Tag Numbers	Present/Absent* Listed/Not Listed on Traffic Report/Chain of Custody Record
7. Sample Condition	Intact/Broken*/ Leaking
8. Shipping Container Temperature Indicator Bottle	Present/Absent*
9. Shipping Container Temperature	_____
10. Does information on Traffic Reports/Chain of Custody Records and Sample Tags agree?	Yes/No*
11. Date Received at Lab	_____
12. Time Received	_____

	EPA Sample #	Aqueous/ Water Sample pH	Corresponding		Remarks: Condition of Sample Shipment, etc.
			Sample Tag #	Assigned Lab #	
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					

* Contact SMO and attach record of resolution

Reviewed By	Logbook No.
Date	Logbook Page No.

ISM02.4 (10/2016) Form DC-1

APPENDIX C CONT'D: FORM DC-1 -SFAM01.1

FORM DC-1
SAMPLE LOG-IN SHEET

Lab Name		Page of
Received By (Print Name)		Log-in Date
Received By (Signature)		
Case Number	SDG No.	MA No.

Remarks:	
1. Custody Seal(s)	Present/Absent* Intact/Broken
2. Custody Seal Nos.	_____ _____
3. Traffic Report/Chain of Custody Records	Present/Absent*
4. Airbill	Airbill/Sticker Present/Absent*
5. Airbill No. and Shipping Container ID No.	_____ _____
6. Shipping Container Temperature Indicator Bottle	Present/Absent*
7 Shipping Container Temperature	_____
8. Sample Condition	Intact/Broken*/ Leaking
9. Sample Tags Sample Tag Numbers	Present/Absent Listed/Not Listed on Traffic Report/Chain of Custody Record
10. Does information on Traffic Report/Chain of Custody Records and Sample Tags agree?	Yes/No*
11. Date Received at Lab	_____
12. Time Received	_____

	EPA Sample #	Aqueous/ Water Sample pH	Corresponding Sample Tag #	Corresponding Assigned Lab #	Remarks: Condition of Sample Shipment, etc.
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					

* Contact SMO and attach record of resolution

Reviewed By	Logbook No.
Date	Logbook Page No.

SFAM01.0 (05/2019) FORM DC-1

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Sample Receiving
ISSUER: Pace ENV - Local Quality - WCOL

APPENDIX D: SAMPLE RECEIVING IMPORTANT NOTES

	Document Name: Sample Receiving Important Notes	Document Revised: 3/23/2021 Page 1 of 1
	Document No.: ME00238-03	Issuing Authority: Pace ENV – Local Quality - WCOL

Sample Receiving Important Notes

Test Method Information:

- Total Nitrogen = TKN+N/N
- Total Organic Nitrogen = TKN-NH3
- 8 RCRA Metals = As, Ba, Cd, Cr, Pb, Hg, Se, Ag
- TTO = VOC(624) + SVOC(625) + P/P(608)
- 3550 = DRO Soil
- 5030 = GRO Soil
- 9071 = O&G Soil

Sample Storage Locations:

Analysis Category	Storage Location
All analyses except volatiles	SR Walk-in Cooler #1
All inorganic non-metal analyses	INM Walk-in Cooler #3
Aqueous volatiles	VOA Walk-in Cooler #8
Solid volatiles (MeOH and Screening Vials)	VOA Refrigerator/Freezer #4
Solid volatiles (Encores and Low Level)	VOA Freezer #29
CLP trace volatiles	VOA Refrigerator #18
All other aqueous CLP volatiles	VOA Walk-in Cooler #8
CLP solid volatiles	VOA Freezer #29
CLP BNA/Pesticides/Aroclors	SR Walk-in Cooler #1

Critical Holding Times:

Test	Hold Time	Dept
Method 3030C	72 Hours	IM
BOD/CBOD	48 Hours	INM
Color [ADMI / Platinum Cobalt]		
MBAS		
Nitrate [NO3] / Nitrite [NO2]		
Ortho-phosphorous		
Turbidity		
Sulfite [SO3]	Immediately [15 mins]	
Total Residual Chlorine [TRC]		
Ferrous Iron	24 Hours	
Hexavalent Chromium [Aqueous]		
pH		
E.coli [MPN]	8 Hours	Micro / INM
Fecal Coliform [Colilert-18]		
Fecal Coliform [MF]		
Heterotrophic Plate Count [HPC]		
Fecal Coliform [MPN] Biosolids		
Total Coliform	30 Hours	
Encore Sampler Preservation	48 Hours	VOA

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Sample Receiving
ISSUER: Pace ENV - Local Quality - WCOL

APPENDIX E: SAMPLE RECEIVING - SUMMARY OF PROCEDURES

	Document Name: Sample Receiving - Summary of Procedures	Document Revised: 6/4/2021 Page 1 of 3
	Document No.: ME001KS-03	Issuing Authority: Pace ENV – Local Quality - WCOL

Sample Receiving – Summary of Procedures

NON-CLP CLIENT COOLER / SAMPLE LOGIN
See SOP for full instructions.

SAMPLE LOGIN PRIORITY

- COCs with **Short Holds**: Nitrate, Nitrite, Ferrous Iron, Hexavalent chromium (Cr^{6+}), BOD
- **Rush** work
- **EPA-CLP SOM/ISM**

1. Open all coolers, remove, and sign COCs – **RECORD TEMPERATURES. Notify PM immediately if temperature is above 6°C either by phone, email or face-to-face.**
2. Review COCs – prioritize log-in.
3. Inspect contents – **notify PM immediately if bottles are broken.**
4. Review collection dates/times and tests – **Notify lab immediately for samples with holding times that are in danger of expiration (less than 12 hours remaining).**
5. Place sample bottles on counter, organize bottles according to the COC.
6. Verify correct sample ID's and number and type of bottles. **Notify PM immediately if bottles are missing (compared to COC).**
7. **Aqueous Samples:**
   - Verify correct pH and TRC for each sample as required.
   - Adjust pH with the proper acid or base (cyanide only) if required.
   - Check VOC vials for bubbles. If vials have bubbles - record sample ID and number of vials w/bubbles.

   Soil Samples
   - Make up a **percent solids** snap cup for each sample. Consult PM before decanting any free-standing water from the sample container. Use a wooden tongue depressor and add 5-10 grams to the snap cup. Mark SOM sample snap cup labels with a yellow highlighter.
   - **VOC vials w/stir bar are stored on their side in freezer in VOA prep lab.**
   - **Notify** Volatiles lab immediately if **Encore** samplers arrive.
8. Fill out Sample Receiving Checklist (SRC).
9. Create a new lot in LIMS 5.
   - Use **Quote Number** on COC if provided. Contact PM if in doubt about which quote to use.
   - Enter **Project Name, Project Number and PO Number** from COC if provided.
   - Check COC for **Rush TAT – notify PM** that **Rush** samples have arrived.
   - Check COC for any **Special Instructions** – enter into comments in LIMS 5.
   - Enter Sample ID's, collection date & time, bottle and tests as requested on COC – **Call PM with discrepancies!! Do not login metals if the correct list is not available – send an email to appropriate PM!!**
   - Enter all COC numbers if more than one COC is sent.
   - Scan COC and completed SRC, print sample labels.
10. Label sample bottles and **% moisture** snap cups.

11. Store **non-VOC** sample bottles on the appropriately labeled shelves in the Sample Receiving Walk-in cooler.

12. VOC vial storage:

Aqueous samples

- The three-40 mL VOA vials are placed side by side according to sample ID starting at the lower left corner of the storage box and continuing from bottom to top.
- Store samples in walk-in cooler in Volatiles laboratory.

Soil Samples

- Store vials in each of three boxes: one box for methanol vials, one box for screening vials and one box for sets of two vials w/stir bars.
- Store in vials with stir bars (vials placed on their side) in freezer in Volatiles Prep Lab.
  - Alert VOC lab immediately when ENCORE soils arrive. If VOC personnel are not available place the samples in the VOA freezer.

13. Staple the COC and Sample Receiving Checklist (SRC), together and place in PM 'mail' box.

CLP COOLER / SAMPLE LOGIN
See SOP for full instructions.

USEPA CLP –SOM/ISM/SFAM SAMPLES

1. **Form DC-1**

- Sign and record date and time of sample receipt on DC-1 form – **document any sample anomalies or discrepancies**.

2. **Sample Receipt**

- **Inspect cooler**. Verify custody seals are present. **Note on DC-1 if custody seals are missing.**
- **Open cooler**. Inspect contents. Note any samples that are broken on DC-1.
- **Measure temperature** of temperature blank if present. If no temp blank is present measure temperature of a representative sample. **Notify PM if temp is > 10 °C.**
- Remove and setup samples according to COC or Traffic Report (TR)
- Make a copy of the TR. Sign and record date/time on all forms (COC, TR, and air bills). Fill out DC-1 form.
- **Assign SDG (Sample Delivery Group) number**. SDG number is the sample number of the first sample received in the SDG or the lowest sample number if multiple samples are received.

An SDG is assigned for:

- ***each case of field samples received,***
- ***each 20 field samples within a case,***
- ***or each 7-day calendar period during which field samples within a case are received***

If the same sample ID is received over multiple days while the SDG is open during the 7-day calendar period, it will always be added to the same SDG containing that ID.

- Create a new lot in LIMS 5. Enter sample information.
- Enter **entire** Traffic Report Number in COC field.

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Sample Receiving
ISSUER: Pace ENV - Local Quality - WCOL

- **Check the Test Key** at the bottom of the Traffic Report to verify the correct tests: i.e. VOC vs. Trace VOC.
- If there is a **discrepancy** between the tests requested on the Traffic Report and the Scheduling Notification, login **tests requested** for the Case as listed on the **Scheduling Notification**.

Modified Analysis (MA)

- Log in Modified Analysis test from quote**. If MA test is not available** or in doubt about which test to login – **contact the CLP project manager immediately.**
- Add MA number from Traffic Report to Comments.
  - **Soils: Log in pH for all SVOC, Pesticides, PCB's**

- Verify VOC soil sample vials received contain water and methanol as required. **Note the sample IDs on form DC-1** for the vials that do not contain the required water and methanol. The VOC department will **add DI water and/or methanol** to vials received empty as required.
- Print sample labels and label sample containers.
- Collect all sample tags according to SDGs and store with the Traffic Report and air bill. Store in **a Ziploc Bag** labeled with **SDG number**.
- Prepare VOC Storage Blank for each matrix and sample type: Soil, Aqueous and Trace VOC. Store with samples.

Aqueous & Trace VOC

1. 3-40 ml vials
2. Purged DI water from VOC lab

Soil VOC

1. 2-40 mL vials with stir bars
2. Purged DI water from VOC lab
3. 5 to 5.05 g of Ottawa sand

- Store Volatile samples according to sample matrix and type in the Volatiles lab:
  - Trace VOC refrigerator
  - Aqueous VOC refrigerator
  - Soil Freezer
- Alert VOC lab immediately when ENCORE soils arrive. If VOC personnel are not available place the samples in the VOA freezer.

Percent Moisture - Decant any standing water present and add 5 -10 grams soil to a labeled snap cup.

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Sample Receiving
ISSUER: Pace ENV - Local Quality - WCOL

APPENDIX F: SUBCONTRACT SHIPPING RECORD

Pace Analytical Services, LLC - South Carolina

Subcontract Shipping Record

Section 1: To be filled out by the PM

Date: _____ Project Manager: _____
Lot Number: _____
Subcontract Lab: _____
Address: _____
_____
_____
Phone Number: _____
Test(s): _____
_____
No. Samples: _____
Turnaround Time: _____

Shipping Method: _____ Select from drop-down menu

Special Instructions: _____

Report Level: _____
EDD: _____

Section 2: To be filled out by Sample Receiving

SR Custodian Initials: _____
Date: _____
COC Number: _____

Tracking Numbers: _____

(attach Subcontract COC if applicable)

APPENDIX G: RADIOLOGICAL SURVEY METER OPERATIONS GUIDANCE

Pace Analytical	Document Name: Radiological Survey Meter Operations Guidance	Document Revised: 3/23/2021 Page 1 of 1
	Document No.: ME0041U-01	Issuing Authority: Pace ENV – Local Quality - WCOL

RADIOLOGICAL SURVEY METER OPERATIONS GUIDANCE

Pancake Model 44-9 Geiger Counter

1. Turn the switch to the BATT check position. The meter should read within the BATT OK area.
2. To minimize fluctuation in readings, ensure that the RESPONSE knob is turned completely to the left (lowest setting).
3. Remove the red cover and perform an operation check by exposing the detector to a radiation check source. With the detector still exposed to the check source, press the [RESET] button. The reading should drop rapidly to zero, then climb back up to the source reading once the [RESET] button is released.
4. The meter reads in 200 cpm increments. Take readings with the meter set to the appropriate scale factor (0.1X for most sample containers encountered). The meter reading must be multiplied by the scale factor to obtain the proper number.
    a. Example 1: Meter needle at 200 cpm with meter set to the 0.1X scale factor signifies a reading of 20 cpm (200 x 0.1)
    b. Example 2: Meter needle at 200 cpm with meter set to the 10X scale factor signifies a reading of 2000 cpm (200 x 10)
5. Record the radiological survey on the Radioactive Materials Survey Log [HS Form ME001O3].

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Sample Receiving
ISSUER: Pace ENV - Local Quality - WCOL

APPENDIX H: Temperature Log

Temperature Log (ME003K2-02)
Issuing Authority: Pace ENV - WCOL
Revised:1/1/2021
Page 1 of 1

Temperature Log

Department:	Date:	Time:		Analyst:		Time:		Analyst:	
Support Equipment Information		Temp. (°C)	Adjusted Temp (°C)	Temp. Within Acceptable Range? Y/N	Comments	Temp. (°C)	Adjusted Temp (°C)	Temp. Within Acceptable Range? Y/N	Comments
Therm. ID:	Correction Factor: 0.0°C								
Therm. ID:	Correction Factor: 0.0°C								
Therm. ID:	Correction Factor: 0.0°C								
Therm. ID:	Correction Factor: 0.0°C								
Therm. ID:	Correction Factor: 0.0°C								
Therm. ID:	Correction Factor: 0.0°C								
Therm. ID:	Correction Factor: 0.0°C								
Therm. ID:	Correction Factor: 0.0°C								
Therm. ID:	Correction Factor: 0.0°C								
Therm. ID:	Correction Factor: 0.0°C								
Therm. ID:	Correction Factor: 0.0°C								
Therm. ID:	Correction Factor: 0.0°C								

Footnote(s):
Refrigerator Temperature Range: 2.0 to 6.0°C
Freezer Temperature Range: -10.0 to -20.0°C

Temperatures Must Be Taken Twice Daily, Monday - Saturday, At Least 4 Hours Apart And Once On Sunday And Holidays
IF TEMPERATURES FALL OUTSIDE OF ACCEPTABLE RANGE QA MUST BE NOTIFIED WITH AN NCM;
DOCUMENT NCM NUMBER IN COMMENT SECTION

Note: Only page 1 is displayed here.

APPENDIX I: RADIOACTIVE MATERIALS SURVEY LOG

RADIOACTIVE MATERIALS SURVEY LOG

[1] Gieger Counter with Pancake Probe (alpha, beta, and gamma measurements): Use 0.1X multiplier setting.
[2] **Shipment(s) cannot be accepted if survey results in readings at or above 20 CPM**

Survey Date	
Survey Time	
Survey Conducted By	
Lot Number	

Countrate Meter Information	**Geiger Counter / Pancake Probe**
Meter ID(s)	
Meter Calibration Due Date:	
Operation Check Performed? (Y/N)	

General Information	**Yes / No**
Client Screening Results and Shipping Information Present?	
Exterior Shipping Package/Cooler Intact?	
Interior Sample Containers Intact?	

Screening Information[1]	**Geiger Counter / Pancake Probe**
Background Reading (CPM)	
Exterior Reading (CPM)	
Interior Reading (CPM)	

Shipment Acceptance[2]	**Yes / No**
Shipment Accepted?	
If shipment not accepted, is the package properly isolated and has the RSO been contacted?	

APPENDIX J: Hazard Communication Standard Pictogram

HCS Pictograms and Hazards

Health Hazard  ▪ Carcinogen ▪ Mutagenicity ▪ Reproductive Toxicity ▪ Respiratory Sensitizer ▪ Target Organ Toxicity ▪ Aspiration Toxicity	**Flame**  ▪ Flammables ▪ Pyrophorics ▪ Self-Heating ▪ Emits Flammable Gas ▪ Self-Reactives ▪ Organic Peroxides	**Exclamation Mark**  ▪ Irritant (skin and eye) ▪ Skin Sensitizer ▪ Acute Toxicity (harmful) ▪ Narcotic Effects ▪ Respiratory Tract Irritant ▪ Hazardous to Ozone Layer (Non-Mandatory
Gas Cylinder  ▪ Gases Under Pressure	**Corrosion**  ▪ Skin Corrosion/Burns ▪ Eye Damage ▪ Corrosive to Metals	**Exploding Bomb**  ▪ Explosives ▪ Self-Reactives ▪ Organic Peroxides
Flame Over Circle  ▪ Oxidizers	**Environment (Non-Mandatory)**  ▪ Aquatic Toxicity	**Skull and Crossbones**  ▪ Acute Toxicity (fatal or toxic)

OSHA Occupational Safety and Health Administration

OSHA 3491-02 2012

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Sample Receiving
ISSUER: Pace ENV - Local Quality - WCOL

APPENDIX K: IR Gun Thermometer Quarterly Calibration Log

IR Gun Calibration Verification

Date: Analyst: IR Gun ID:

Temperature Point	NIST Therm. ID	NIST Reading (°C)	IR Gun Reading (°C)	Adjustment (+/- °C)	Comments (Retirement Date, High, Mid, Low)
Ambient				0.0	
SR Walk-in				0.0	
Water & ice				0.0	

**The adjustment for any temperature point should be no greater than ± 1°C. Notify QA if this criterion is not met.*

Adjustment Factor for IR Gun Thermometer = 0.0 (only report to one decimal place)

$$\text{Adj. Factor for IR Gun Therm.} = \frac{[(\text{ambient adjustment}) + (\text{SR walk-in adjustment}) + (\text{water \& ice adjustment})]}{3}$$

Calibration Instructions:

Obtain three 1-liter wide mouth amber bottles from the bottle prep area. Fill two 1-liter wide mouth amber bottles approximately 2/3 full of water for the 'ambient' and 'SR walk-in cooler' points. Fill one 1-liter wide mouth amber bottle with ice for the 'water and ice' point. Add approximately 50 grams of NaCl to the bottle containing ice and fill 2/3 full with water. The salt is used to depress the temperature below 0°C. Affix Shealy sample labels on each bottle below the water line. Place the SR walk-in cooler bottle in a safe area inside the SR walk-in cooler where it will not be disturbed. Put the ambient bottle and the ice/water/NaCl bottle in a safe area in SR. When taking the temperature with the IR gun, remember to aim the IR gun at the container label for the most accurate recording. Allow at least one hour for equilibration. Put the NIST traceable reference thermometer in the 'ambient' bottle and allow the reference thermometer to come to equilibrium. Read the temperature with the IR gun on the bottle by holding the gun ~8" from the label. Record all temperatures.

Place the reference thermometer in the 'water and ice' bottle and allow the reference thermometer to come to equilibrium. Read the temperature with the IR gun on the bottle by holding the gun ~8" from the label. Record all temperatures. Place the reference thermometer in the 'SR walk-in cooler' bottle in the walk-in and allow the reference thermometer to come to equilibrium for approximately thirty minutes. While physically in the walk-in cooler, read the temperature with the IR gun on the bottle by holding the gun ~8" from the label. Record all

Note: The last recorded digit represents the increments marked on that thermometer. If a reading appears to fall between two increments, the closest increment is chosen and reported.

Example: a thermometer with 0.5° increments will be recorded as 3.5°C with the knowledge that the reading was closest to the 3.5° increment.

Adjustments are derived from the difference between the subject thermometer and the NIST thermometer.

The Adjustment Factor for the IR Gun Thermometer must be added to the apparent reading of the thermometer for all subsequent recordings.

Note: Once verification is completed, please save document and send QA an email to let us know that it is finished.

QA Verified by:
Date:

APPENDIX L: IR Gun Thermometer Daily Verification Log

	Document Name: IR Gun Thermometer Daily Verification	Document Revised: 12/29/2020 Page 1 of 1
Pace Analytical	Document No.: ME001N9-05	Issuing Authority: Pace ENV – Local Quality - WCOL

IR Gun Thermometer Daily Verification

Date	Analyst	Reference Thermometer ID	Reference Thermometer Reading (°C)	IR Gun Thermometer ID	IR Gun Thermometer Reading (°C)	Pass / Fail (±0.5°C)	Comments

*Daily reading must be ±0.5°C or new quarterly verification must be performed.

APPENDIX M: Cooler Temperature Receipt Labels

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Sample Receiving
ISSUER: Pace ENV - Local Quality - WCOL

APPENDIX N: Balance #13-1224 Verification Log

	Document Name: Balance # 13-1224 Verification Log	Document Revised: 3/17/2021 Page 1 of 1
	Document No.: ME0024U-03	Issuing Authority: Pace ENV – Local Quality - WCOL

BALANCE # 13-1224 VERIFICATION LOG

MODEL # ML802E/03 SERIAL # B345970999 Manufacturer: Mettler Toledo

Weights				1.00 g	10.00 g	50.00 g	Pass Y = Yes N = No
Tolerance Limits				0.99 g – 1.01 g	9.99 g – 10.01 g	49.95 g – 50.05 g	
Date	Time	Analyst	Weight Set #				

(This balance is capable of weighing two places to the right of the decimal point.)

Note: If value is out of tolerance limits, re-zero and repeat balance verification. If still out of limits, contact QA.

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1.0 SCOPE AND APPLICATION

STATE NOTE: For samples analyzed in conjunction with the Ohio Voluntary Action Program (VAP) please utilize ENV-SOP-MTJL-0214.

1.1 Inductively coupled plasma-atomic emission spectrometry (ICP-AES) determines trace elements, including metals and some non-metals in solution. This procedure follows the guidelines established in EPA method 200.7 and SW-846 Method 6010B, 6010C, and 6010D for drinking water, waste water, ground water, TCLP, SPLP, and STLC leachates, soils, sludge, sediments, solid wastes, oils, and other digestates after appropriate preparatory procedure is performed.

This procedure is also applicable to reporting calculated values for Calcium, Magnesium, and Total Hardness from values determined using EPA methods 200.7 or 6010B/C/D from groundwater, wastewater and drinking waters. Reporting limits for Hardness are derived from the annual MDL studies for Calcium and Magnesium of the appropriate determinative EPA method. The routine reporting limits for each category of hardness are listed in Table 1.2b.

1.2 This method is applicable for the analytes listed in Table 1.2a and b. Detection limits, sensitivity, and the optimum and linear concentration ranges of the elements can vary with the wavelength, spectrometer, matrix, and instrument operating conditions. Table 1.2 also lists the Reporting Limits (RLs), used routinely by Pace Analytical National Center for Testing & Innovation (Pace National).

Table 1.2a: Environmental Analytes and Reporting Limits *(Subject to change, see section 13.1)*

Analyte	Aqueous				Sediment		
	Ground Water/ Wastewater 6010B/C/D/200.7	Drinking Water 200.7*	RL	Units	Solids 6010B/C/D	RL	Units
Aluminum	☜	☜	00.200	mg/L	☜	2.00	mg/Kg
Antimony	☜	☜	0.010	mg/L	☜	1.00	mg/Kg
Arsenic	☜	☜	00.010	mg/L	☜	1.00	mg/Kg
Barium	☜	☜	0.005	mg/L	☜	0.50	mg/Kg
Beryllium	☜	☜	0.002	mg/L	☜	0.20	mg/Kg
Boron	☜	☜	0.050	mg/L	☜	5.0	mg/Kg
Cadmium	☜	☜	0.002	mg/L	☜	0.20	mg/Kg
Calcium	☜	☜	1.000	mg/L	☜	100	mg/Kg
Chromium	☜	☜	0.010	mg/L	☜	1.00	mg/Kg
Cobalt	☜	☜	0.010	mg/L	☜	1.00	mg/Kg
Copper	☜	☜	0.010	mg/L	☜	1.00	mg/Kg
Iron	☜	☜	0.100	mg/L	☜	10.0	mg/Kg
Lead	☜	☜	0.005	mg/L	☜	0.50	mg/Kg
Lithium	☜		0.015	mg/L	☜	1.50	mg/Kg
Magnesium	☜	☜	1.000	mg/L	☜	100	mg/Kg
Manganese	☜	☜	0.010	mg/L	☜	1.00	mg/Kg
Molybdenum	☜	☜	0.005	mg/L	☜	0.50	mg/Kg
Nickel	☜	☜	0.010	mg/L	☜	1.00	mg/Kg

Analyte	Aqueous				Sediment		
	Ground Water/ Wastewater 6010B/C/D/200.7	Drinking Water 200.7*	RL	Units	Solids 6010B/C/D	RL	Units
Potassium	↳	↳	1.000	mg/L	↳	100	mg/Kg
Selenium	↳	↳	0.010	mg/L	↳	1.00	mg/Kg
Silicon	↳	↳	0.050	mg/L	↳	5.00	mg/Kg
Silver	↳	↳	0.005	mg/L	↳	5.00	mg/Kg
Sulfur	↳	↳	1.0	mg/L	↳		mg/Kg
Sodium	↳	↳	1.000	mg/L	↳	100	mg/Kg
Strontium	↳	↳	0.010	mg/L	↳	1.00	mg/Kg
Thallium	↳	↳	0.010	mg/L	↳	1.00	mg/Kg
Tin	↳	↳	0.050	mg/L	↳	5.00	mg/Kg
Titanium	↳	↳	0.050	mg/L	↳	5.00	mg/Kg
Vanadium	↳	↳	0.010	mg/L	↳	1.00	mg/Kg
Zinc	↳	↳	0.050	mg/L	↳	5.00	mg/Kg

**May not meet required Drinking Water Maximum Contamination Levels (MCLs) using this methodology.*

Table 1.2b: Hardness Categories and Reporting Limits

(Subject to change, see section 13.1)

Hardness:	RL (mg/L)
Calcium Hardness	1.25
Magnesium Hardness	0.41
Total Hardness	1.6

1.3 For the determination of total recoverable analytes in aqueous and solid samples, an acid digestion process is required. Environmental samples for analysis by Method 6010B, 6010C, or 6010D including, TCLP or EP leachates, soils, sludge, sediments, and other solid wastes require an acid digestion prior to analysis. Samples are digested by SW-846 methods 3005 (Acid Digestion of Waters for Total Recoverable Metals), 3010 (Acid Digestion of Aqueous Samples), 3015 (Microwave Digestion of Aqueous Samples), 3050 (Acid Digestion of Sediments, Sludge, Soil, and Oils) and 3051 (Microwave Assisted Digestion of Sediments, Sludge, Soil, and Oils). Digestion methods are found in ENV-SOP-MTJL-0217 and ENV-SOP-MTJL-0219.

1.4 The Clean Water Act has approved EPA Method 200.7 for demonstrating compliance on discharge monitoring for NPDES (National Pollution Discharge Elimination System) permits. 40 CFR136.3 has Guidelines for Establishing Test Procedures for Analysis of Pollutants. The National Primary Drinking Water Regulations for inorganic chemical sampling and analytical requirements can be found in 40 CFR141.23. Updates to these regulations can be found in the current Code of the Federal Register.

1.5 To determine dissolved analytes in aqueous samples, a 0.45µm filtration method is employed then the filtered samples are acidified. To reduce potential interferences, dissolved solids must be <0.2% (w/v).

1.6 Analysis without acid digestion can be used for drinking water samples if the samples have been properly preserved with acid and have turbidity of <1 NTU at the time of analysis. These samples must be acidified to match the acid matrix of the calibration standards and analyzed directly. This total recoverable determination procedure is referred to as "direct analysis". Silver concentration cannot be determined from direct analysis when chloride ions are present as a silver chloride precipitate may be formed. The sample must be acid digested to form a soluble silver chloride complex. Some primary drinking water metal contaminants may require sample concentration to meet regulatory drinking water reporting limits criteria[14.2].

Method 6010D – Samples that are not digested necessitate the use of either an internal standard or should be matrix-matched with the standards. If using the former option, the instrument software should be programmed to correct for the intensity differences of the internal standard between samples and standards. **NOTE**: All samples analyzed by Method 6010 are typically digested.

1.7 When determining boron and silicon in aqueous samples, only plastic, PTFE (Teflon™) sample containers and laboratory glassware must be used. For accurate determination of boron in solid samples, only quartz or PTFE tubes must be used during acid digestion with immediate transfer of an aliquot of the final volume of digestate to a plastic centrifuge tube[14.2].

1.8 For the determination of titanium, white plastic and white printed containers must be avoided as titanium dioxide is used as a white pigment.

1.9 The total recoverable sample digestion procedure dissolves and maintains in solution only minimal concentrations of barium in the presence of free sulfate. For the analysis of barium in samples having varying and unknown concentrations of sulfate, analysis must be completed as soon as possible following sample preparation[14.2].

1.10 Detection limits and linear ranges for the elements vary with the wavelength selected, the spectrometer, and the matrix. Table 1.11 provides a list of routinely used wavelengths and the type of spectrometer view used.

Method 6010D – IDLs are necessarily instrument-specific. Therefore, if needed, an IDL must be determined through a separate experimental study for each instrument. IDLs should be established, at a minimum, on an annual basis for each matrix and for each preparatory/determinative method combination used.

TABLE 1.10: WAVELENGTHS
(exact wavelengths vary slightly depending on the instrument)

Analyte	Wavelength (nm)	Type of View
Aluminum	308.215	Radial
Antimony	206.836	Axial
Arsenic	188.979	Axial
Barium	233.527	Axial
Beryllium	313.107	Radial
Boron	249.772	Radial
Cadmium	214.440	Axial
Calcium	317.933	Radial

Analyte	Wavelength (nm)	Type of View
	373.690	
Chromium	205.560	Axial
Cobalt	228.616	Axial
Copper	324.752	Radial
Iron	259.940 271.441	Radial
Lead	220.353	Axial
Lithium	670.784	Radial
Magnesium	279.077	Radial
Manganese	257.610	Axial
Molybdenum	202.031	Axial
Nickel	232.003	Axial
Potassium	766.490	Radial
Phosphorus	177.495	Axial
Selenium	196.026	Axial
Silicon	251.611	Axial
Silver	328.068	Axial
Sodium	589.592 818.326	Radial
Strontium	407.771	Radial
Sulfur	181.972	Axial
Thallium	190.801	Axial
Tin	189.927	Axial
Titanium	334.940	Radial
Vanadium	292.402	Radial
Zinc	213.857	Axial

1.11 Users of the data generated using this method must state the data-quality objectives (DQOs) prior to analysis.

1.12 Any deviations from this SOP must be documented. Deviations are reflected in a case narrative and the method is reported as modified. Per customer requirement, the procedure and QC criteria described in this SOP can be changed/modified. Authorization from the Operations Manager and Project Manager is required for each modification and Regulatory Affairs approval must also be secured for any deviation.

1.13 Method Detection Limits (MDLs) are performed based on ENV-SOP-MTJL-0016.

1.13.1 Limit of Detection (LOD) and Limit of Quantitation (LOQ) studies are completed at the frequency required by the TNI standard per the procedure identified in the ENV-SOP-MTJL-0016, *Method Detection Limits (MDL), Limits of Detection (LOD), and Limits of Quantitation (LOQ).* Should the procedure be utilized for DoD support; then the frequency of these studies must meet the requirements of the current DoD QSM.

1.14 Linear Dynamic Range (LDR) and Inter-element correction factor (IEC) studies must be analyzed semi-annually for each analytical instrument or when there are major changes/repairs to the instrument[14.5, 14.1]. Instrument Detection Limit studies must be analyzed at least quarterly for each analytical instrument[14.5].

2.0 METHOD SUMMARY AND DEFINITIONS

2.1 The analysis described in this method involves multi-elemental determinations by ICP-AES using sequential or simultaneous instruments. The instrument measures characteristic atomic-line emission spectra by optical spectrometry. Samples are aspirated into the nebulizer and the resulting aerosol is transported to the plasma torch. The emission spectra are dispersed by a grating spectrometer separating the light emitted into the distinct wavelengths generated by each element in the sample. A photosensitive device monitors the intensities of each wavelength line in the spectra. The intensity of light on the photosensitive device produces a signal that is measured and processed by a computer system. Due to the many possible wavelengths of light generated by each element and possible overlapping of high intensity peaks, a background correction technique is required for trace element determination. Background intensities must be measured adjacent to the analyte spectra lines during analysis. The position selected for background intensity measurement can be selected on either or both sides of the analyte wavelength line and must be determined by the complexity of the spectrum adjacent to the analyte line. The position used for background correction must be as free from spectral interference as possible and must reflect the same change in background intensity as occurs at the analyte wavelength. Background correction is not required in cases of line broadening where the background correction measurement would actually degrade the analytical result. The possibility of additional interferences should also be recognized and appropriate corrections made.

2.2 Dissolved Analyte - The concentration of analyte in an aqueous sample that has been passed through a 0.45µm membrane filter assembly prior to sample acidification and digestion.

2.3 Total (Total Recoverable) Analyte – The concentration of analyte determined either by ‘‘direct analysis’’ of an unfiltered acid preserved drinking water sample with turbidity of <1 NTU or by analysis of the solution extract of a solid sample or an unfiltered aqueous sample following digestion by refluxing with hot dilute mineral acid(s) as specified in the method

2.4 Instrument Detection Limit (IDL) - The concentration equivalent to the analyte signal which is equal to three times the standard deviation of a series of 10 replicate measurements of the calibration blank signal at the same wavelength. The IDL assures with 99% certainty that a value is above the instrument noise level.

Note: An IDL is a statistical determination without analytes present used to assess background correction protocols and an MDL is determined with low levels of analytes present to determine instrument sensitivity for each analyte.

2.5 Linear Dynamic Range (LDR) - The range over which the instrument response to analyte concentration remains linear.

2.6 Plasma Solution - A solution that is used to determine the optimum torch height relative to the radio frequency (RF) coil for viewing the spectrum.

2.7 Interference Check Sample (ICS) – A series of five solutions (ICSA, ICSAB, ICSA2, LA, & CE) to verify that inter-element interferences are correctly compensated. The ICS checks provide an adequate on-going test of inter-element correction (IEC) factors. These standards are referred to the Spectra Interference Check (SIC) in EPA Method 200.7

2.7.1 ICSA – A solution containing only the interfering analytes at high concentrations.

2.7.2 ICSAB – A solution containing interferents plus other method analytes at the level of concern, which corresponds to the project specific action limits.

2.7.3 ICSA2 – A solution containing interfering analytes not contained in the ICSA.

2.7.4 LA – A solution containing Lanthinum at a high concentration.

2.7.5 CE – A solution containing Cerium at a high concentration.

2.8 Water Sample - For the purpose of this method, a sample taken from one of the following sources: drinking water, surface water, ground water, storm water, industrial or domestic wastewater.

2.9 Preparation Batch - For method 6010B/C/D/ EPA 200.7 (WW only): A group of samples (not to exceed twenty) of a similar matrix, which have been digested at the same time using the same digestion process and have all necessary QC associated with them. For method 200.7 (DW only): A group of samples (not to exceed ten) of similar matrix, which have been digested at the same time using the same digestion process and have all necessary associated QC.

2.10 Analytical batch - A group of samples that are analyzed in the same sequence with all appropriate preparation and analytical QC.

2.11 Inter-element correction (IEC) coefficient - analyte concentration equivalent arising from a given interferent's concentration.

2.12 Serial Dilution - a dilution and reanalysis of a field sample that is performed once per batch of samples. One sample is diluted 5X and reanalyzed.

2.13 Post Spike – A second aliquot of a field sample that is spiked with known concentrations of target analytes and analyzed to assess recovery of the spike. A post spike must be analyzed when the MS and/or MSD fail due to a suspected matrix effect. One sample is spiked after digestion and analyzed per batch.

2.14 Lower Limit of Quantitation (LLOQ) - A term associated with analysis per the requirements of Method 6010D; the lowest point of quantitation which, in most cases, is the lowest concentration in the calibration curve.

2.15 See the current Quality Assurance Manual for other definitions associated with terms found in this document.

3.0 HEALTH AND SAFETY

3.1 The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

3.2 The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets (SDS) for all hazardous chemicals are available to all personnel. Employees must abide by the environmental, health, and safety (EHS) policies and procedures specified in this SOP and in the Pace National Chemical Hygiene / Safety Manual.

3.3 Personal protective equipment (PPE) such as safety glasses and/or side shields, gloves, a laboratory coat, and shoes that are not cloth, canvas, and/or perforated must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure. When handling glass, needles, knives, or any material with a potential sharp edge, employees must use cut-resistant gloves.

3.4 Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids, bases, or oxidizers in a fume hood whenever possible with the appropriate PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

3.5 Spill kits are located in each laboratory department. Employees are to familiarize themselves with the location and contents of each spill kit in their area.

3.6 Universal precautions should be observed when performing any tests or procedures. Hard surfaces, instrument surfaces may be contaminated and should be handled according to good laboratory practices.

3.7 Contact your supervisor or local EHS coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure. Any accidents involving personnel or sample supplies are to be reported immediately to either the Manager and/or to the Safety Officer.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

4.1 All samples must have been collected using a sampling plan that addresses the considerations of this method.

4.2 Samples submitted for analysis that do not meet the requirements contained within this section must be addressed before performing the logging process within the laboratory. In some cases, exceeding the appropriate preservation and storage criteria can cause significant bias in the resulting data. Clients may need to resubmit samples where the conditions during shipment cause uncertainty regarding sample integrity. If samples do not meet the requirements for preservation, sampling, shipment and storage and the client approves the completion of the analytical process, sample results can be qualified per the ENV-SOP-MTJL-0014, *Data Handling and Reporting*.

4.3 Prior to the collection of an aqueous sample, consideration must be given to the type of data required, (i.e., dissolved or total recoverable), so that appropriate preservation and pre-treatment steps can be taken. The pH of all aqueous samples must be assessed immediately prior to sample digestion or "direct analysis" to ensure the sample has been properly preserved. If the field sample is properly preserved, the sample can be held up to 6 months prior to analysis.

4.4 For the determination of dissolved elements, the sample must be filtered through a 0.45µm pore diameter membrane filter to remove the suspended elements or particles. This filtration must take place at the time of collection or as soon thereafter as practically possible. Glass or plastic filtering apparatus are recommended to avoid possible contamination. Only plastic apparatus must be used when the determinations of boron and silica are critical. Use a portion of the filtered sample to rinse the filter flask, discard this portion and collect the required volume of filtrate. Acidify the filtrate with (1:1) nitric acid: water immediately following filtration to pH <2.

4.5 For the determination of total recoverable elements in aqueous samples, samples must not be filtered, but acidified with (1:1) nitric acid: water to pH <2. Preservation may be done at the time of collection; however, to avoid the hazards of strong acid use in the field, possible transport restrictions, or possible contamination, it is recommended that the samples be returned to the laboratory within two weeks of collection and acid preserved upon receipt in the laboratory. Following acidification, the sample must be mixed and equilibrated for 24 hours. The pH is verified at <2 prior to withdrawing an aliquot for acid digestion or "direct analysis". If, for reasons such as high alkalinity, the sample pH is verified to be >2, more acid must be added and the sample equilibrated for another sixteen hours until verified to be pH <2.

4.6 Solid samples require no preservation prior to analysis. Solid samples can be held up to six months from the time of sample collection until preparation and analysis.

4.7 For aqueous samples, a field blank must be prepared and analyzed as required by the data user. Use the same container and preservative as is used in field sample collection. The sample holding time is six (6) months from the date and time of collection until analysis. Samples are preserved to pH <2 with nitric acid.

4.8 Samples prepared from concentrations must be run within three days of digestion to avoid evaporation.

5.0 INTERFERENCES

5.1 Spectral interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

5.1.1 Subtracting the background emission determined by measurement(s) adjacent to the analyte wavelength peak can usually compensate for background emission and stray light. The location(s) selected for the measurement of background intensity is determined by the complexity of the spectrum adjacent to the wavelength peak. The location(s) used for routine measurement must be free of off-line spectral interference (inter-element or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak. Changes in background correction must be saved in the instrument method. Background correction can be established by scanning the following three solutions: 1) blank (same as calibration blank); 2) solution, containing analytes at significant concentration to raise a signal above background signal (CCV solution may be used) at mid-range of the curve; 3) solution(s) containing most common interfering elements at high concentration and other interferents as well (ICSAB solution may be used).

5.1.2 Spectral overlaps can be compensated for by equations that correct for inter-element contributions, which involve measuring the interfering elements. When operative and uncorrected, these interferences produce false-positive determinations and are reported as analyte concentrations. Users may apply inter-element correction factors determined on their instruments within tested concentration ranges to compensate (offline or online) for the effects of interfering elements. Consult the method for specific identified interferences.

5.1.3 When inter-element corrections are applied, there is a need to verify their accuracy by analyzing spectral interference check solutions. The IEC's are established by analyzing a solution of the interfering element at a high concentration within the LDR limit, measuring the analyte concentration equivalents arising from the interfering element, calculating the interference factor as analyte reading in mg/L, then dividing by the interfering element concentration. The IEC's are changed in the stored ICP instrument method. Inter-element corrections vary for the same emission line among instruments because of differences in resolution, as determined by the grating plus the entrance and exit slit widths, and by the order of dispersion. Inter-element corrections also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line may appear should be avoided. Inter-element corrections that constitute a major portion of an emission signal may not yield accurate data. Users must not forget that some samples might contain uncommon elements that could contribute spectral interferences.

5.1.4 Interference effects must be evaluated for each individual instrument. For each instrument, intensities vary not only with optical resolution but also with operating conditions (such as power, viewing height and argon flow rate). To determine the appropriate location for offline background correction, the user must scan the area on either side of the peak adjacent to the wavelength and record the apparent emission intensity from all other method analytes. The location selected for background correction must be either free from offline inter-element spectral interference or a computer routine must be used for their automatic correction on all determinations.

5.2 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by such means as using a high-solids nebulizer, diluting the sample, using a peristaltic pump, or using an appropriate internal standard element. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, which affects aerosol flow rate and causes instrumental drift. This can be controlled using a high-solids nebulizer, wetting the argon prior to nebulization, using a tip washer, or diluting the sample. Also, it has been reported that better control of the argon flow rates, especially for the nebulizer, improves instrument stability and precision. This is accomplished with the use of mass flow controllers.

5.3 Chemical interferences include molecular-compound formation, ionization effects, and solute-vaporization effects. Normally, these effects are not significant with the ICP-AES technique. If observed, they can be minimized by careful selection of operating conditions (such as incident power and observation height), by buffering of the sample, by matrix

matching and by standards addition procedures. Chemical interferences are highly dependent on matrix type.

5.4 Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the buildup of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences must be recognized within an analytical run and suitable rinse times must be used to reduce them.

5.5 Linear Dynamic Range (LDR) study is performed by analyzing a solution of each element at maximal concentration unless the result falls outside 10% RPD. The highest calibration standard for each analyte cannot be greater than the LDR for that analyte. If an interferent is found greater than the LDR and an IEC factor is established between the interferent and analyte of interest, the sample must be diluted for proper correction of inter-element interferences. Instrument methods with different calibration standard concentrations require separate LDR studies.

5.6 Background correction is performed as needed and LDR and IEC studies are completed as required by each published analytical method and whenever significant changes to instrumentation are made. Background, or blank matrix, subtraction is not performed for environmental samples.

6.0 EQUIPMENT AND SUPPLIES

6.1 Inductively coupled plasma emission spectrometer:

6.1.1 Perkin Elmer Model 5300 or Thermo Model 7000 series ICP, or equivalent, with background correction and computer control

6.1.2 Cetac Autosampler or ESI autosampler

6.1.3 Argon gas supply - High purity grade (99.99%). When analyses are conducted frequently, liquid argon is more economical and requires less frequent replacement of tanks than compressed argon in conventional cylinders.

6.2 Narrow-mouth storage bottles, FEP (fluorinated ethylene propylene) with screw closure, 125mL to 1L capacities

6.3 One-piece stem FEP wash bottle with screw closure, 125mL capacity

6.4 Adjustable pipettes (Eppendorf or equivalent), ranges from 2µL to 5000µL

6.5 Class A volumetric flasks for standards preparations

6.6 Polypropylene (PP) conical tubes

6.7 Peristaltic pump

7.0 REAGENTS AND STANDARDS

7.1 All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See ENV-SOP-MTJL-0041, *Standard Logger – Tree Operation*. Additional information regarding reagent preparation can be found in the Standards Logger

(Tree) digital archive system. All spiking solutions and surrogate standard solutions should be replaced at least every six (6) months or sooner if a problem is detected unless otherwise noted.

7.2 Hydrochloric acid, concentrated (sp. gr. 1.19) - HCl

7.2.1 Hydrochloric acid (1+1) - Add 500mL concentrated HCl to 400mL reagent water and dilute to 1L with reagent water.

7.3 Nitric acid, concentrated (sp. gr. 1.41) - HNO_3

7.3.1 Nitric acid (1+1) - Add 500mL concentrated HNO_3 to 400mL reagent water and dilute to 1L with reagent water.

7.4 Laboratory Reagent water

7.5 ICP Standard List

Calibration	Stock Std Cat #	Working Standard Preparation
RL Standard #1	HP7013-500	50mL of Stock LL Std, 10% rinse to volume 1000mL
0.5ppm Standard #2	HP6928-1L	125mL of 2.0ppm Std, 10% rinse to volume 500mL
1ppm Standard #3		250mL of 2.0ppm, 10% rinse to volume 500mL
2ppm Standard #4		Direct pour
10ppm Standard #5	HP6409-500	Direct pour
250ppm Standard #6	HP4526-1L	500mL of 500ppm Std, 10% rinse to volume 1000mL
500ppm Standard #7		Direct pour
La Standard #8	VHG-PLAN-100	10mL of Stock La Std, 10% rinse to volume 1000mL

Standards	Stock Std Cat #	Working Standard Preparation
ICV	ESC-8	5mL of Stock ICV Std, 10% rinse to volume 500mL
CCV	HP6929-1L	50mL of Stock CCV Std, 10% rinse to volume 1000mL
ICVLL	HP7013-500	50mL of Stock LL Std, 10% rinse to volume 1000mL
CCVLL		50mL of Stock LL Std, 10% rinse to volume 1000mL
ICSA	ICL500-6	50mL of Stock A Std, 10% rinse to volume 500mL
ICSAB	ICL500-6,HP2739-500	50mL of Stock A Std, 5ml of Stock AB Std, 10% rinse to volume 500mL
CE	HP100010-1	5mL of Stock Ce Std, 10% rinse to volume 500mL

Standards	Stock Std Cat #	Working Standard Preparation
ICSA2	varies	5 mL of Stock Std, 10% rinse to volume 500mL
LA	VHG-PLAN-100	10mL of Stock La Std, 10% rinse to volume 1000mL.
IEC / LDR As	HP10003	10mL of Stock As Std, 10% rinse to volume 1000mL
IEC / LDR Co	HP100013	10mL of Stock As Std, 10% rinse to volume 1000mL
IEC / LDR Cr	HP100012	10mL of Stock As Std, 10% rinse to volume 1000mL
IEC / LDR Cu	HP100014	10mL of Stock As Std, 10% rinse to volume 1000mL
IEC / LDR Mn	HP100032	10mL of Stock As Std, 10% rinse to volume 1000mL
IEC / LDR Si	HP100050	10mL of Stock As Std, 10% rinse to volume 1000mL
Internal Std	HP10M67-1,HP10M24-1	1mL of Stock Y, 3mL of Stock In, 10% rinse to volume 1000mL
10% Blank and Rinse	A200C-212	Fill container to 90% with DI water, add HNO3 to volume

7.6 Blanks - Three types of blanks are required for ICP-AES analysis

7.6.1 The calibration blank is used to establish the baseline for the instrument prior to the analysis of the analytical curve. The calibration blank is prepared by acidifying reagent water to the same acid concentration as used for the standards (10% HNO3).

NOTE: The calibration blank must be stored in a FEP bottle to minimize leaching from other container materials that can cause an elevation in the target analytes leached causing an inherent bias in the calibration and quantitation of field samples when baselines are established prior to calibration of the ICP-AES.

7.6.1.1 Following calibration, the Initial Calibration Blank (ICB) is analyzed prior to field sample analyses. A Continuing Calibration Blank (CCB) is analyzed following the CCV after every ten samples and at the end of the analytical sequence to verify on-going acceptable instrument conditions.

7.6.2 The method blank is used to assess possible contamination from the sample preparation procedure. The method blank must contain all the reagents in the same volumes as used in sample preparation. The method blank must be prepared in the same manner as the samples including sample digestion, when applicable.

7.6.3 The rinse blank is prepared by acidifying reagent water to the same concentrations as the acids as used in the calibration blank (10% HNO3). This solution is stored in a convenient manner. The rinse blank is used for equipment “wash out” to flush the

sample delivery system and eliminate memory effects (carryover) from previous samples or standards.

7.7 Mixed Calibration Standard Solutions are used to make the following calibration solutions. All standards are prepared in Class A volumetric flasks using adjustable pipettes. The final acid concentration is matrix matched to digested field sample concentrations. See section 7.5 above for preparation of these standards.

Concentration of Target Analytes in Calibration Standards in mg/L

Analyte	STD 1	STD 2	STD 3	STD 4	STD 5	STD 6	STD 7	STD 8
Silver	0.005	0.5	1.0	2.0				
Aluminum	0.2				10	250	500	
Arsenic	0.01	0.5	1.0	2.0				
Boron	0.2		1.0	2.0				
Barium	0.005	0.5	1.0	2.0	10			
Beryllium	0.002	0.5	1.0	2.0				
Calcium	1.0		1.0	2.0	10	250	500	
Cadmium	0.002	0.5	1.0	2.0				
Cerium						10		
Cobalt	0.01	0.5	1.0	2.0				
Chromium	0.01	0.5	1.0	2.0				
Copper	0.01	0.5	1.0	2.0				
Iron	0.10	0.5	1.0	2.0	10	100	200	
Potassium	1.0			2.0	10	50	100	
Phosphorus	0.1	0.5	1.0	2.0				
Lanthanum								10
Lithium	0.015	0.5	1.0					
Magnesium	1.0				10	250	500	
Manganese	0.01	0.5	1.0	2.0				
Molybdenum	0.005	0.5	1.0	2.0				
Sodium	1.0		1.0	2.0	10	250	500	
Nickel	0.01	0.5	1.0	2.0				
Lead	0.005	0.5	1.0	2.0				
Antimony	0.01	0.5	1.0	2.0				
Selenium	0.01	0.5	1.0	2.0				
Silicon	0.2	0.5	1.0	2.0	10			
Strontium	0.01	0.5	1.0	2.0				
Sulfur	1.0		1.0	2.0	10	50	100	
Tin	0.05	0.5	1.0	2.0				
Thallium	0.01	0.5	1.0	2.0				
Vanadium	0.02	0.5	1.0	2.0				
Zinc	0.05	0.5	1.0	2.0				
Titanium	0.05	0.5	1.0	2.0				

7.8 Initial Calibration Verification (ICV) – The ICV is an analytical standard solution from a second source different from the calibration and CCV standards. The ICV is prepared at a mid-range concentration within the linear working range of the instrument. The ICV must have the same acid matrix as the Calibration Standards. See Section 7.5 above for the preparation of this standard.

All analytes are present in the mid-level ICV solution at the following concentrations (mg/L):

Analyte	Concentration
Silver	1.0
Aluminum	10.0
Arsenic	1.0
Boron	1.0
Barium	1.0
Beryllium	1.0
Calcium	10.0
Cadmium	1.0
Cobalt	1.0
Chromium	1.0
Copper	1.0
Iron	10.0
Potassium	10.0
Lithium	1.0
Magnesium	10.0
Phosphorus	1.0

Analyte	Concentration
Manganese	1.0
Molybdenum	1.0
Sodium	10.0
Nickel	1.0
Lead	1.0
Antimony	1.0
Selenium	1.0
Silicon	1.0
Strontium	0.4
Tin	1.0
Thallium	1.0
Vanadium	1.0
Zinc	1.0
Titanium	1.0
Sulfur	10.0

7.9 Continuing Calibration Verification (CCV) – The CCV is the mid-range calibration standard prepared from the same source as the initial calibration curve. The CCV is used to verify the regression of the initial calibration of the instrument and must be repeated following every ten samples and at the conclusion of the sequence. EPA Method 200.7 refers to this standard as the Instrument Performance Check (IPC) standard. See Section 7.5 above for the preparation of this standard.

All analytes are present in the mid-level CCV solution at the following concentrations (mg/L):

Analyte	Concentration
Silver	0.5
Aluminum	10.0
Arsenic	1.0
Boron	1.0
Barium	0.50
Beryllium	0.20
Calcium	50.0
Cadmium	0.50
Cobalt	1.0
Chromium	1.0
Copper	1.0
Iron	10.0
Potassium	50.0
Lithium	1.0
Magnesium	10.0
Phosphorus	1.0

Analyte	Concentration
Manganese	1.0
Molybdenum	0.25
Sodium	50.0
Nickel	1.0
Lead	0.50
Antimony	0.5
Selenium	1.0
Silicon	2.0
Strontium	1.0
Tin	0.5
Thallium	1.0
Vanadium	1.0
Zinc	1.0
Titanium	1.0
Sulfur	5.0

7.10 Low Level Initial/Continuing Calibration Verification for EPA 6010C (ICVLL/CCVLL) –The ICVLL/CCVLL is prepared at a low concentration within the linear working range of the

instrument and defines the lowest level of quantitation/reporting. The ICVLL/CCVLL must have the same acid matrix as the calibration standards. See Section 7.5 above for the preparation of this standard.

The concentration of the low-level ICVLL/CCVLL solution is listed in the table below:

Analyte	Concentration (mg/L)
Silver	0.005
Aluminum	0.2
Arsenic	0.01
Boron	0.2
Barium	0.005
Beryllium	0.002
Calcium	1
Cadmium	0.002
Cobalt	0.01
Chromium	0.01
Copper	0.01
Iron	0.10
Potassium	1
Lithium	0.015
Magnesium	1
Phosphorus	0.1

Analyte	Concentration (mg/L)
Manganese	0.01
Molybdenum	0.005
Sodium	1
Nickel	0.01
Lead	0.005
Antimony	0.01
Selenium	0.01
Silicon	0.2
Strontium	0.01
Tin	0.05
Thallium	0.01
Vanadium	0.02
Zinc	0.05
Titanium	0.05
Sulfur	1.00

7.11 Interference Check Solutions (ICSA, ICSAB, ICSA2, LA, CE) – The ICS checks are prepared to contain known concentrations of interfering elements that provides a test of the correction factors. The ICSA, ICSA2, LA, and CE solutions contains the interfering elements at a high concentration and the ICSAB contains both the interfering analytes at a high concentration and the analytes of interest at 0.5 to 1.0mg/L. EPA Method 200.7 and 6010D refers to this standard as the Spectral Interference Check (SIC) standard. See Section 7.5 above for the preparation of these standards.

7.11.1 The ICSA solution contains 5000mg/L of each Al, Ca, Mg and 2000mg/L Fe.

7.11.2 The ICSAB solution contains all the components at the same concentrations of the ICSA and other target analytes of interest spiked. In the working ICSAB solution, silver, boron, cadmium, nickel, lead, silica, and zinc are present at 1.0mg/L. All other analytes (arsenic, barium, beryllium, cobalt, chromium, copper, manganese, molybdenum, antimony, selenium, tin, thallium, vanadium and titanium) are present at 0.5mg/L.

7.11.3 The ICSA2 solution contains interfering elements that are not in the ICSA. In the working ICSA2 solution, all analytes are present at 10mg/L.

7.11.4 The LA solution contains 10mg/L of La.

7.11.5 The CE solution contains 10mg/L of Ce.

7.12 The IEC / LDR solutions are prepared to contain known concentrations of interfering elements that are used to adjust the IECs and are used for daily LDRs for 6010D. The interfering elements are present at 10mg/L.

7.13 Internal Standards – The internal standard response is used to measure the relative responses of other method analytes in each sample. Yttrium and Indium are used as internal standards. See Section 7.5 above for the preparation of this standard.

7.14 For the preparation of Laboratory Control Samples (LCSs) and Matrix Spikes (MSs) see the applicable sample preparation SOPs.

8.0 PROCEDURE

8.1 Sample Analysis

8.1.1 **Initializing the Instrument:** Prior to daily calibration of the instrument, inspect the sample introduction system including the nebulizer, torch, injector tube for salt deposits, dirt, and debris that would restrict solution flow and affect instrument performance.

8.1.1.1 Replace the uptake tubing daily.

8.1.1.2 If any of the sample introduction parts appear soiled, first remove the part from the instrument by following the maintenance procedure in the instrument manual. Once removed, attempt to clean the part with a dilute solution of 5% nitric acid. Cleaning may be performed using a cotton swab or by submersing the part in the acid solution for no longer than five (5) minutes. If cleaning is successful, dry the part using compressed air or argon and replace it in the instrument. If cleaning does not adequately remove the residue, the part must be replaced with a new one in accordance with the manufacturer's directions. Replacement parts are kept in the cabinet in the instrument lab.

8.1.2 **Instrument Stability:** The instrument must be allowed to become thermally stable before calibration and analyses. This usually requires at least 30 minutes of operation.

8.1.3 **Instrument Calibration:** For initial and daily operation, calibrate the instrument according to the instrument manufacturer's recommended procedures using mixed calibration standard solutions and the calibration blank. A peristaltic pump is used to introduce all solutions, samples, and the internal standard to the nebulizer. To allow adequate time for equilibrium to be reached in the plasma, aspirate all solutions for at least 30 seconds after the solution reaches the plasma before obtaining the sample analyte response.

8.1.3.1 Use the average value from three replicate analyte responses per sample to be correlated to the overall analyte concentration in the solution being sampled. Flush the system with the rinse blank for a minimum of 60 seconds between each standard.

8.1.3.2 The calibration regression is generated using first order linear regression of a calibration blank and three calibration standards where each element is present. The blank is included as a point in the calibration

curve to determine the baseline correction needed for the instrument to effectively quantitate target analyte concentrations.

8.1.3.3 Calibration acceptance criteria are described in section 10.4.

8.1.4 **Internal Standard:** All standards/samples/QC etc. contain yttrium and indium as the internal standards. The instrument adds the internal standards automatically. The instrument injects a constant volume into each solution being analyzed (i.e. standard, blank, field sample, LCS/LCSD/MS/MSD/DUP) and monitors the intensity at the sample level. An internal standard is the chosen alternative to the method of standard additions (MSA). If signal variation results from the sample introduction system (samples of different viscosity, matrix constitution), all the elements are corrected in the same way by an internal standard. If variation results from a variation of the energy transfer, the internal standard most accurately corrects elements of similar energy. Internal standard acceptance criteria are described in section 10.14.

8.1.5 **Calibration Accuracy:** Verify the acceptable initial calibration of the instrument using a standard source that is either an independent lot or entirely different manufacturer to ensure calibration accuracy. After calibrating and rinsing the instrument, analyze the ICV and, if analyzing samples using EPA 6010C, analyze the ICVLL standards. These standards are prepared as directed in section 7.7 and 7.9. Acceptance criteria are described in section 10.5.

8.1.6 **On-going Calibration Stability:** Verify the acceptable on-going instrument calibration by analyzing appropriate check standards during the sequence. Instrument calibration acceptability is demonstrated after every 10 samples using the CCV and CCB and at the end of the analytical run using the CCV, CCVLL (if analyzing EPA 6010C samples), and CCB that must meet the criteria described in sections 10.6 & 10.14.

8.1.7 **Accurate Background Corrections:** The interference check standards (ICSA, ICSAB, ICSA2, LA, and CE) are used to verify the inter-element and background correction factors at the beginning of an analytical run. The ICSA and ICSAB are verified during every 8-hour work shift. The interference check standards must meet the criteria found in section 10.10.

8.1.8 An Initial Calibration Blank (Section 7.5.1) is analyzed before sample analysis is initiated to verify the cleanliness of the analytical system. Acceptance criteria are described in section 10.14.

8.1.9 **Field Sample Analysis:** After completion of the above calibration requirements, samples must be analyzed in the same operational manner used in the calibration routine with the rinse blank also being used between all sample solutions, method blanks, Laboratory Control Standards, matrix spike, matrix spike duplicates, and check solutions.

8.1.10 **Dilutions:** If a sample analyte concentration is quantitated within 90% or greater of the upper limit of the analyte's determined Linear Dynamic Range (LDR), see section 10.4.2 for further guidance.

9.0 DATA ANALYSIS AND CALCULATIONS

9.1 Sample data should be reported in units of mg/L for aqueous samples and mg/kg dry weight corrected for solid samples.

9.2 For dissolved aqueous analytes, report the data generated directly from the instrument with compensation for sample dilution. Never report analyte concentrations below the MDL and if reporting between the MDL and the routine RL, results should be qualified with the appropriate indicator for estimated target analyte concentrations.

9.3 For total recoverable aqueous analytes, multiply solution analyte concentrations by the dilution factor 0.5 when a 100mL aliquot is used to produce the 50mL final digestate volume, and report data. If a different aliquot volume other than 100mL is used for sample preparation, adjust the dilution factor accordingly. Account for any additional dilution of the prepared sample digestate required to complete the determination of any analytes exceeding 90% or greater of the LDR upper limit. Never report analyte concentrations below the MDL and if reporting between the MDL and the routine RL, results should be qualified with the appropriate indicator for estimated target analyte concentration. Routine reporting limits are adjusted for any dilution required by the sample analysis.

9.4 Results are reported to Three significant figures by the laboratory LIMS. Analyte concentrations for solids data should be rounded in a similar manner following dry weight corrections.

9.5 For total recoverable analytes in solid samples, calculate the target analyte concentration using the equation below and do not report analyte data below the estimated solids RL or an adjusted RL based on additional dilutions required to complete the analysis:

$$\text{Sample Conc. (mg/Kg) dry-weight basis} = \frac{C \times V \times D}{W}$$

where: C = Concentration in extract (mg/L)
V = Volume of extract (L, 100mL = 0. 1L)
D = Dilution factor (undiluted = 1)
W = Weight in Kg of sample aliquot extracted (g x 0.001 = Kg)

9.6 Soil samples are routinely reported on a dry weight basis. Soil samples must be processed using the ENV-SOP-MTJL-0065, *Total Solids*. After a dry weight for each sample has been obtained, the calculations are performed automatically by the laboratory LIMS as follows:

$$\% \text{ solids (S)} = \frac{DW}{WW} \times 100$$

where: DW = Sample weight (g) dried
WW = Sample weight (g) before drying

9.7 Hardness calculations:

Total Hardness, mg equivalent $CaC0_3$/L = 2.497 [Ca, mg/L] + 4.118 [Mg, mg/L]
Calcium Hardness = 2.497 [Ca, mg/L]
Magnesium Hardness = 4.118 [Mg, mg/L]

9.8 To calculate the silica concentration from silicon analysis:

Silica (mg/L) = 2.14*[Silicon, mg/L]

9.9 Formula needed to calculate dilution of stock standards of known concentration to a known final volume, using the basic chemistry formula, $C_1*V_1=C_2*V_2$:

$$V_{stock} = V_{std} * C_{std} / C_{stock}$$

where: V_{stock} = volume of stock standard required (mL)
V_{std} = final volume of diluted standard required (mL)
C_{stock} = concentration of stock standard required (µg/mL or µg/L)
C_{std} = final concentration of diluted standard required (µg/mL or µg/L)

NOTE: Be sure to maintain consistent units for both concentration and volume during the use of the calculation and keep in mind that (1µg/mL = 1mg/L=1000µg/L, 1L=1000mL=1000000µL, and 1mL=1000µL)

9.10 Percent Relative Intensity (%RI) for internal standard assessment (ISTD):

$$\%RI = \text{Intensity of } ISTD_{sample} / \text{Intensity of } ISTD_{CalBlk} * 100\%$$

9.11 Relative Standard Error (RSE – expressed as a percentage)

$$RSE = 100 \times \sqrt{\sum_{i=1}^{n}\left[\frac{x'_i - x_i}{x_i}\right]^2 \Big/ (n-p)}$$

where:
x'_i = Measured amount of analyte at the calibration level *i*, in mass or concentration units
x_i = True amount of analyte at calibration level *i*, in mass or concentration units
p = Number of terms in the fitting equation (average – 1, linear = 2, quadratic = 3)

9.12 See the current Quality Assurance Manual for other equations associated with common calculations.

10.0 QUALITY CONTROL AND METHOD PERFORMANCE

10.1 All analysts must meet the qualifications specified in ENV-SOP-MTJL-0015, *Technical Training and Personnel Qualifications,* before approval to perform this method. Analysts must complete an initial demonstration of proficiency before being approved to perform this method. Continuing proficiency must be demonstrated using proficiency testing, laboratory control sample analysis and/or MDL studies. Method performance is assessed per analyst. Updated method performance records are filed and stored in a central location within the department.

10.1.1 Prior to using Method 6010D for quantitation of samples, an initial demonstration of performance packet must be completed. This packet must document:

- The selection criteria for background correction points
- Analytical dynamic ranges including the applicable equations and upper limits of ranges

- IDLs and Method LLOQs
- The determination and verification of interelement correction equations or other routines for correcting spectral interferences. These data must be generated using the same instrument, operating conditions, and calibration routine to be used for sample analysis. The data must be kept on file and available for review by the data user or auditor.

10.2 Use Prep Data to record batch order and standards/reagents used during analysis. See ENV-SOP-MTJL-0014, *Data Handling and Reporting*.

10.3 Batch Analyses

10.3.1 Environmental Preparation Batches: Preparation batches are defined as sets of 1-20 samples as defined in Chapter 1 of SW-846 and in section 9.3.1 of EPA 200.7. Preparation batch analysis must include the following: 1 Method Blank, 1 Laboratory Control Sample (LCS), 1 Laboratory Control Sample Duplicate (LCSD), 1 Serial Dilution, 1 Sample Post Digestion Spike, 1 Matrix Spike/Spike Duplicate (MS/MSD) pair. All batch information is maintained in Prep Data computer program.

10.3.2 Analytical Batches: Analytical batches are defined as a sequence of samples analyzed concurrently using the same calibrated instrument. Analytical batches include the QC samples produced in the Preparation Batches, in addition to: 1 Serial Dilution, 1 Sample Post Digestion Spike, 1 Initial Calibration Verification (ICV) following initial calibration, 1 Initial Calibration Verification-Low Level (ICVLL) following initial calibration (when analyzing EPA 6010C, 6010D and DOD only) 1 Initial Calibration Blank following the ICVLL, 1 Continuing Calibration Verification (CCV) following each 10 samples and at the conclusion of the sequence, 1 Continuing Calibration Verification-Low Level (CCVLL) at the conclusion of the sequence (when analyzing EPA 6010C only), 1 Continuing Calibration Blank (CCB) following each CCV, 1 Interference Check Sample A (ICSA), 1 Interference Check Sample AB (ICSAB), Interference Check Sample 2, 1 Lanthinum Interference Check Sample, and 1 Cerium Interference Check Sample following each initial calibration. All batch information is maintained in Prep Data computer program.

10.4 Supporting Analytical Studies

10.4.1 Instrument Detection Limits (IDL) Studies - IDLs in µg/L can be determined as the mean of the calibration blank results plus three times the standard deviation of 10 replicate analyses of the solution. Use zero for the mean if the mean is determined to be a negative value.

IDLs must be verified quarterly[14.13] or when major instrumentation change occurs.

10.4.2 Linear Dynamic Range (LDR) Studies – Linear dynamic ranges are established for each instrument to allow for quantitation above the highest level of calibration without qualification. ICP instruments are known to remain linear at high levels, but each upper limit of linearity is based on the target analyte being measured and the routine instrument operating conditions.

To perform a linear dynamic range study, the instrument must be calibrated normally as used with client field samples. The LDR is determined by the analysis of a minimum of three, but preferably five, different increasing

concentrations of standards containing each target analyte across a range. One concentration should be near the expected upper linear range for each analyte. The highest concentration, where the instrument calibration remains linear, is determined when the observed concentration of the increasing standards is no more than 10% below the expected concentration of the analyte. If more than a 10% deviation exists, the instrument is proven to no longer be linear at that value for that analyte. The upper linear range is therefore the next lower concentration of standards used in the determination. Samples quantitated above that upper determined LDR require dilution to quantitate within the proven linear range of the instrument.

LDR studies must be verified semi-annually[14.1] or when major instrumentation change occurs.

Method 6010D - LDR standards must be ran daily within ten percent of true value, or dilute all samples above the high standard in the curve.

STATE NOTE: For work performed in support of the NC Department of Natural Resources (15A NCAC 02H.0805(a)(7)(I)) for target analytes quantitated by ICP or ICPMS, a series of at least three standards must be analyzed along with each group of samples. The concentrations of these standards must bracket the concentration of the analytes in the field samples analyzed. Samples with target analyte concentrations above the highest level of calibration must be diluted to quantitate analytes within the calibration range. The use of the dynamic linear range studies to validate analyte/instrument calibration linearity must not be used for NC sample analysis.

10.4.3 Method Detection Limits – See also ENV-SOP-MTJL-0016, *Method Detection Limits (MDL), Limits of Detection (LOD) and Limits of Quantitation (LOQ)*.

MDL studies are required annually or when instrumentation change occurs. Method detection limit studies are performed on blank matrices most closely matching field sample matrices.

10.4.4 Inter-element Correction Factors – All inter-element spectral correction factors must be verified and updated every six months or when major instrumentation change occurs.[14.1,14.5,].

Criteria for determining an inter-element spectral interference is an apparent positive or negative concentration of an analyte that is outside the 3-sigma control limits of the calibration blank for the analyte. See Attachment II for a listing of potential interfering analytes and their contributions from SW-846 method EPA 6010B. Testing is performed using 100mg/L single element solutions; however, for analytes such as iron that may be found at high concentration, a more appropriate test would be to use a concentration near the upper analytical range limit.

Suggested analytes that are known to commonly interfere include: Ag, Al, As, B, Ba, Be, Ca, Cd, Ce, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Sb, Se, Si, Sn, Sr, Ti, Tl, V, and Zn.

10.4.5 Proficiency Testing (PT) – See also ENV-SOP-MTJL-0022, *Proficiency Testing Program*. Proficiency testing is performed in the metals department in support of

both environmental and industrial hygiene analyses. Environmental PTs are performed semi-annually for Water Supply (Safe Drinking Water Act), Water Pollution (Clean Water Act), and soils (RCRA) testing.

QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
ICAL	Daily	A calibration curve must consist of a blank and at least three calibration standards for each target analyte. The linear regression correlation coefficient must be ≥0.995 for 200.7, 6010B, and 6010D. Must be ≥0.998 for 6010C.	Identify and correct source of problem, repeat.	None. Do not proceed with analysis.
ICV	After Each ICAL	± 10% for method 6010B, 6010C and 6010D or ± 5% for method 200.7 The RSD of the standards must be below 5% for 6010B, 6010C and 6010D and below 3% for 200.7.	Identify source of problem, re-analyze. If repeat failure, repeat ICAL. Analysis may proceed if it can be demonstrated that the ICV exceedance has no impact on analytical measurements. For example, the ICV %R is high, CCV is within criteria, and the analyte is not detected in sample(s).	Qualify analytes with ICV out of criteria.
ICB	Immediately after the initial calibration verification	All elements of interest must be evaluated to a criteria of +/- ½ of the RL for method 6010D. All elements of interest must be evaluated to +/- the RL for method 6010B,6010C and 200.7. Criteria to be evaluated to method criteria unless otherwise specified by client.	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the ICB exceedance has no impact on analytical measurements. For example, the ICB has detections and the analyte is not detected in sample(s).	Qualify analytes with ICB out of criteria.
ICVLL/ CCVLL	The ICVLL must be analyzed at the beginning of each run for every analyte of interest. The ICVLL is analyzed at or below the RL. Additionally, the CCVLL must be analyzed after samples to bracket some specific client requirements and method 6010C samples.	± 50% (or specified by the client) For method 6010C, must be within ± 30% . For method 6010D, must be within.± 20%.	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the ICVLL/CCVLL exceedance has no impact on analytical measurements. For example, the ICVLL/CCVLL %R is high and the analyte is not detected in sample(s). For example, the ICVLL/CCVLL %R is high and the analyte detections exceed the continuing calibrations verification level (midpoint of the curve). If the ICVLL/CCVLL is biased low, no data can be reported for the target elements failing criteria.	Qualify outages and explain in case narrative.

QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
CCV	Daily, before sample analysis, after every 10, and at end of analytical window.	For method 6010B, 6010C, 6010D and 200.7, the CCV must be within ± 10% of the true value. The RSD of the CCV must be below 5% for 6010B.	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the CCV exceedance has no impact on analytical measurements. For example, the CCV %R is high, and the analyte is not detected in sample(s).	Qualify analytes with CCV out of criteria.
CCB	Daily, before sample analysis, after every 10, and at end of analytical window	All elements of interest must be evaluated to a criteria of +/- the RL for 200.7, 6010B, 6010C and 6010D. Depending on the data quality objective of individual clients different criteria may apply.	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the CCB exceedance has no impact on analytical measurements. For example, the CCB has detections and the analyte is not detected in sample(s).	Qualify analytes with CCB out of criteria.
Internal Standards	Every field sample, standard and QC sample	70-125% of its true concentration	Troubleshoot instrument performance. Reanalyze samples and dilute if needed.	Qualify outages and explain in case narrative.
Interference check solution (ICSA)	A mixed solution containing concentrations of Al, Ca, and Mg at 500 PPM and Fe at 200 PPM is analyzed at the beginning of each sample run sequence. In some specific client requirements the ICSA must bracket the run or the analytical batch.	Acceptance criteria for the spiked analytes are 80-120%. Unspiked analytes are evaluated at ±LLOQ.	Identify and correct source of problem, repeat performance verification(s). Note: The ICSA can be re-processed after appropriate SIC solutions are analyzed and the IECs are recalculated. If ICSA passes, continue.	None. Do not proceed with analysis for elements that cannot be verified.
Interference check solution (ICSAB)	A solution containing concentrations of Al, Ca, and Mg at 500 PPM and Fe at 200 PPM with low to mid-range concentrations of target analytes as outlined in ILM5.3. This is analyzed following the ICSA when requested. This is required by certain clients. It is not a method requirement and need be analyzed only for clients specifying this in the QAPP	Unspiked analytes are evaluated at ±LLOQ.	Identify and correct source of problem, repeat performance verification(s). Note: The ICSAB can be re-processed after appropriate SIC solutions are analyzed and the IECs are recalculated. If ICSAB passes, continue.	None. Do not proceed with analysis for elements that cannot be verified.
Spectral Interference	SIC solutions are single-element solutions used to	Unspiked analytes are evaluated at ±LLOQ.	If SIC fails, re-calculate IEC and re-process data.	None. Do not proceed with analysis for

QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
Check Solutions (SIC)	evaluate and correct IEC factors. Specific elements evaluated are listed in specific instrument methods.		If a sample level exceeds an SIC level and the interfering element affects target analytes, then: a) run a higher SIC or b) dilute the sample.	elements that cannot be verified.
Method Blank	One per 20 samples	Method 200.7: The method blank is considered to be acceptable if it does not contain the target analytes that exceed 1/2 LLOQ or project-specific DQOs. Method 6010B, 6010C and 6010D: The method blank is considered to be acceptable if it does not contain the target analytes that exceed the LLOQ or project-specific DQOs. WIDNR and West Virginia require samples to be reported to the MDL. The blanks must be clean to the data quality objectives.	Identify source of problem, re-analyze. If reanalysis of the MB fails, all samples affected by the failing MB elements need to be re-digested and re-analyzed. If the method blank exceeds the criteria, but the associated samples are either below the reporting level or other DQOs, or detections in the sample are >10x MB detections then the sample data may be reported. J-flag qualification will be applied for blank detections between the LOQ and LOD when DQOs require evaluation to the MDL.	Qualify outages and explain in case narrative.
LCS	One per 20 samples	80-120% for 6010B,6010C and 6010D 85-115% for 200.7	Identify source of problem, re-analyze. If reanalysis of the LCS fails, all samples affected by the failing LCS elements need to be re-digested and re-analyzed. If LCS recovery is > QC limits and these compounds are non-detect in the associated samples	Qualify analytes with LCS out of criteria.
LCSD	An LCSD must be substituted in the event of insufficient sample volume for a matrix spike duplicate sample.	80-120% for 6010B,6010C and 6010D 85-115% for 200.7 %Diff ≤ 20%	Identify source of problem, re-analyze. If reanalysis of the LCS fails, all samples affected by the failing LCS elements need to be re-digested and re-analyzed. If LCS recovery is > QC limits and these compounds are non-detect in the associated samples	Qualify analytes with LCS out of criteria.
MS/MSD	One per 20 samples for 6010 / 6010C / 6010D One per 10 samples for 200.7	75-125% for 6010B, 6010C, and 6010D 70-130% for 200.7 % RPD: 20%	Perform a SD and PDS on any elements that fail to meet criteria for method 6010(C)(D).	Qualify analytes with MS out of criteria.
Sample Duplicate	Per client request	%Diff ≤ 20%	Qualify outages	Qualify outages.
Serial Dilution	One SD per batch. Method suggestion / Pace Policy, if reporting by 6010B, 6010C, or 6010D.	6010B/C: 1:5 dilution of sample, SD RPD should agree within +/- 10% of the original result when the original sample is greater than 10x the RL.	Data is qualified.	Qualify outages.

QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
		6010D: 1:5 Dilution of sample or MS, for concentrations 25x > LLOQ in parent sample, resultant RPD should agree within +/- 20%.		
Post Digestion Spike	Method suggestion / Pace policy if reporting by 6010B, 6010C, 6010D and MS/MSD fail outside 75-125%	80-120% for 6010C 75-125% for 6010B and 6010D.	Data is qualified.	Qualify outages.
Laboratory Filter Blank (FB)	Analyzed only with batches of lab filtered dissolved metals, one per batch of 20 or less.	All elements of interest must be evaluated to a criteria of +/- ½ the RL for method 6010D. All elements of interest must be evaluated to a criteria of +/- the RL for method 6010B,6010C and 200.7. If the FB does not contain target analytes at a level that interferes with project-specific DQOs, then the FB would be considered acceptable.	Identify source of problem, re-analyze. If reanalysis of the MB fails, all samples affected by the failing MB elements need to be re-digested and re-analyzed. If sample(s) non-detect, report the data. If sample result >10x MB detections, report the data.	Qualify outages and explain in case narrative.
Linear Dynamic Range	If a SIC/LDR standard is not analyzed for any specific element, the highest standard in the calibration becomes the linear range. See Appendix C.	The standard must recover within 10% of the true value, and if successful, establishes the linear range. In each scenario, the data reporting range is established using 90% of the highest calibration level or LDR sample.	The linear range of the instrument must be adjusted until 90% recovery of the reference standard can be achieved.	N/A

10.8 Method/Calibration/Rinse Blanks

10.8.1 Method Blank

NOTE: Per DoD QSM, version 5.0, Section 1.7.4.1, DoD/DOE require that method blanks be evaluated to ½ RL (LOQ) for target analytes and RL (LOQ) for common laboratory contaminants. If contaminants are present in the blank above this level, samples must be re-prepared and re-analyzed or reported with appropriate qualification.

NOTE: Method 6010D – The method blank is considered to be acceptable if target analyte concentrations are less than ½ the LLOQ or are less than project-specific requirements.

State Note: For Wisconsin samples, the method blank must not contain analytes more negative than the MDL value. If target analytes are more negative than the MDL, the instrument must be recalibrated or a new LOD study performed.

State Note: For West Virginia samples analyzed by Method 6010D, blanks are generally considered to be acceptable if target analyte concentrations are less than ½ the lower limit of quantitation (LLOQ) or are less than project-specific requirements. Blanks may contain analyte concentrations greater than acceptance limits if the associated samples in the batch are unaffected (i.e., targets are not present in samples or sample concentrations are ≥10X the blank). Other criteria may be used depending on the needs of the project.
For West Virginia samples analyzed by Method 200.7, blank values that exceed the MDL indicate laboratory or reagent contamination should be suspected.

10.16 Lower Limit of Quantitation (LLOQ) – When analyzing samples according to Method 6010D, the LLOQ is initially verified by the analysis of at least seven (7) replicate samples, spiked at the LLOQ and processed through all preparation and analysis steps of the method. The mean recovery and relative standard deviation of these samples provide an initial statement of precision and accuracy at the LLOQ. In most cases the mean recovery should be +/- 35% of the true value and RSD should be < 20%. In-house limits may be calculated when sufficient data points exist. Monitoring recovery of LLOQ over time is useful for assessing precision and bias.

10.16.1 Ongoing LLOQ verification, at a minimum, is on a quarterly basis to validate quantitation capability at low analyte concentration levels. This verification may be accomplished either with clean control material (e.g., reagent water, method blanks, Ottawa sand, diatomaceous earth, etc.) or a representative sample matrix (free of target compounds). Optimally, the LLOQ should be less than the desired regulatory action levels based on the stated project-specific requirements.

11.0 DATA VALIDATION AND CORRECTIVE ACTION

11.1 All data must undergo a primary review by the analyst. The analyst must check the performance of the initial calibration, mid-point check standard and continuing calibrations to ensure that they meet the criteria of the method. The analyst should review any sample that has quantifiable compounds and make sure that they have been confirmed, if needed. The analyst must also verify that reported results are derived from quantitation between the RL and the highest standard of the initial calibration curve. All calculations must be checked (any dilutions, %solids, etc.). Data must be checked for the presence or absence of appropriate flags. Comments should be noted when data is flagged.

11.2 All data must then undergo a second analyst review. This review must be performed according to ENV-SOP-MTJL-0014, *Data Handling and Reporting* and ENV-SOP-MTJL-0038*, Data Review*.

State Note:
For West Virginia samples analyzed by Method 200.7, when blank values constitute 10% or more of the analyte level determined for a sample or is 2.2 times the analyte MDL whichever is greater, fresh aliquots of the samples must be prepared and analyzed again for the affected analytes after the source of contamination has been corrected and acceptable LRB values have been obtained.

NOTE: Method 6010D - If the method blank fails to meet the necessary acceptance criteria, it should be reanalyzed once. If still unacceptable, then all samples associated with the method blank must be re-prepared and re-analyzed along with all other appropriate analysis batch QC samples. If the method blank results do not meet the acceptance criteria and reanalysis is not practical, then the laboratory should report the sample results along with the method blank results and provide a discussion of the potential impact of the contamination on the sample results. However, if an analyte of interest is found in a sample in the batch near its concentration confirmed in the blank, the presence and/or concentration of that analyte should be considered suspect and may require qualification.

NOTE: If the sample concentration for an analyte is greater than four times (4x) the spike concentration, a "V" qualifier is used. The "V" qualifier indicates that the high concentration of analyte in the sample interfered with the ability to make an accurate spike recovery determination.

11.9 ISTD – The intensity of the Yttrium internal standard response in each sample is monitored and compared to the intensity of the response for that internal standard in the calibration blank. The Percent Relative Intensity (%RI) in each sample must meet the criteria in section 10.14. If the %RI of the response in the sample falls outside of these limits, the laboratory must immediately re-analyze the calibration blank and monitor the internal standard intensities. If the %RI for that calibration blank is within the limits, the laboratory must re-analyze the original sample at a two-fold dilution due to a possible interference from the matrix on the ISTD. If the %RI for the re-analyzed calibration blank is outside the limits, the analysis must be terminated, the problem corrected, the instrument recalibrated, the new calibration verified, and the samples reanalyzed.

11.10 Interference Check Standards (ICSA/ICSAB/ICSA2/LA/CE) - Evaluate the ICSA, ICSAB, ICSA2, LA, and CE. The analyst must verify that the ICS checks have been analyzed at the required frequency. If the criteria in section 10.11 are not met, check the background correction protocols currently in place for appropriateness. If these are the initial ICS checks run after daily calibration, re-analyze the CALBLANK and re-calibrate the instrument. If the ICSA and/or ICSAB did not agree at the end of an 8-hour shift, re-analyze the ICSA and ICSAB. If failure persists, perform instrument maintenance as needed, recalibrate and re-analyze any samples in the previous run that may have been affected.

11.11 Serial Dilution/Post-digestion Spike – The analyst must verify that the SD and PS have been analyzed at the required frequency. If either of these tests fails to meet the required criteria in sections 10.12 & 10.13, the possibility of a matrix interferent should be suspected. An O1 qualifier is used when either sample type fails due to matrix interferences.

11.11.1 Serial Dilution - An analysis of a 1:4 dilution must agree within 10% of the original determination. If not, a chemical or physical interference effect is suspected.

11.11.2 Post-digestion Spike - The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits, a matrix effect is suspected.

CAUTION: If spectral overlap is suspected, use of computerized compensation, an alternate wavelength, or comparison with an alternate method is recommended.

11.12 Data that does not meet acceptable QC criteria may be acceptable for use in certain circumstances.

11.12.1 If a method blank contains an amount of target analyte, but all samples are non-detected, the data may be reported with a “B3” flag. If a method blank contains an amount of target analyte, but the samples contain analyte at a level that is 10 times the level present in the method blanks, the data may be reported with a “B” flag.

11.12.2 If the MS/MSD fails (recovery less than 30% or greater than 150% and/or RPD greater than 30%) in an initial analysis and again upon re-analysis, the data is released with an appropriate qualifier as the failure is accepted as matrix related.

11.12.3 If a calibration verification standard is above the acceptable QC criteria and all samples being bracketed are below the reporting limit, the data is acceptable based on a high calibration bias with undetectable levels in the field samples. Any positive samples require re-analysis.

11.12.4 If the target analyte spiked in the quality control samples (LCS, LCSD, MS, MSD) exhibits high recovery and the target analytes in the field samples are below the reporting limit, the data may be released with a J+ qualifier indicating the high bias with no impact on the field sample analysis due to the bias present.

11.12.5 If the target analyte spiked into the QC pair (LCS/LCSD, MS/MSD) exhibit acceptable recoveries, but high calculated RPD values for precision, and the target analytes in the field sample are flagged with a J3 for the precision beyond acceptable quality control limits.

11.12.6 Sample results can be qualified and possible bias is narrated per the ENV-SOP-MTJL-0014, *Data Handling and Reporting*.

11.12.7 Samples with multiple elements above the LDR must be diluted to verify interferences are not present

STATE NOTE: Drinking water samples analyzed using this procedure for compliance cannot be qualified.

12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

12.1 The EPA requires that a laboratory waste management practice be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner. See ENV-SOP-MTJL-0051*, Waste Management Plan.*

12.2 See ENV-SOP-MTJL-0046, *Environmental Sustainability & Pollution Prevention*.

13.0 METHOD MODIFICATIONS/CLARIFICATIONS

13.1 Adjustments to the concentrations of standards/spiking solutions, standards providers, and quality control are subject to change to better meet client/project/regulatory needs or to improve laboratory method performance.

13.2 Modifications to this method are noted in the body of the text as state notes. Compliance analyses performed in conjunction with specific state requirements must be performed as noted within the specific state(s) note listed.

13.3 Superscripts are provided where necessary to indicate the reference in Section 14.0 where the requirement/information can be found. Subscripts noted identify the most frequent/restrictive cases, but requirements may also be included at different frequencies/conditions in other references noted in section 14.0.

13.4 In the May 2012 Methods Update Rule, the EPA revised the previous interpretation of EPA 200.7 to include the use of axial torch orientation in the published method. Either axial or radial orientation is acceptable.

14.0 REFERENCES

14.1 *Inductively Coupled Plasma-Atomic Emission Spectrometry*, SW-846 Method 6010B, Revision 2, December 1996.

14.2 *Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry*, EPA Method 200.7, Revision 4.4, May 1994.

14.3 *Identification of Test Procedures*, 40 CFR §136.3.

14.4 *Inorganic Chemical Sampling and Analytical Requirements*, 40 CFR §141.23.

14.5 *Inductively Coupled Plasma-Atomic Emission Spectrometry*, SW-846 Method 6010C, Revision 3, February, 2007.

14.6 *Hardness by Calculation*, Standard Methods 2340B, 20th Edition.

14.7 *Hardness by Calculation*, Standard Methods 2340B, 2011.

14.8 *Hardness by Calculation*, Standard Methods 2340B, 1997.

14.9 *Inorganic Analytes*, SW-846 Chapter 3, Revision 4, February, 2007.

Attachment I: Revision History

Current Version (Pace National):

Date	Description of Revisions
4/11/2022	Technical and quality review and update. Revised sections 1.13, 4.8, 10.3.2, 10.5, 10.6.5, 10.8.1,10.8.2, 11.3, 11.7,

Superseded Versions (ESC Lab Sciences SOP#340386):

Version	Date	Description of Revisions
0	5/1/95	Origination
1	7/25/95	
2	3/11/97	
3	8/18/99	
4	2/11/00	
5	8/21/00	
6	3/28/01	
7	12/14/01	
8	4/11/03	
9	1/26/04	
10	8/2/04	
11	10/15/05	Corrected CCV criteria for EPA 200.7
12	10/29/08	Technical and Quality Review and update. Corrected acceptance criteria in Section 10.6. Updated format and re-organized sections 8.0, 10.0 and 11.0 based on new format.
13	1/23/09	Technical and Quality Review and update.
14	2/2/09	Technical and Quality Review and update. Clarification of holding times, Inclusion of cross-references. Inclusion of section 13.1 and section 7.1.
15	4/15/11	Technical and Quality Review and update. Added state notes where applicable; Added Tables 1.2b & 1.2c; Revised Table 1.2a and Sections 1.1, 1.3, 1.6, 1.11, 2.18, 2.22, 5.6, 7.1, 7.6.2, 7.9, 8.1.3, 8.1.5, through 8.1.10, 9.1, 9.7 through 9.12, 10.3, 10.0 & 11.0, 12.1; Added Sections 2.13.1, 2.14.1, 2.31 through 2.35, 3.1.1, 4.1, 4.7, 7.17, 13.2, 13.3, 14.5 through 14.10.
16	6/27/14	Complete Rewrite and update.
17	12/7/2015	Technical and quality review and update. Header and signature block re-formatting. Revised Sections 1.13.1, 2.13.1, 2.23, 5.1.3, 6.1.1, 6.1.2, 7.1, 7.6, 7.7, 7.8, 7.9, 7.10.1, 7.10.2, 7.11, 7.12, 7.13, 7.14, 7.16, 8.1.3, 8.1.5, 8.1.11, 9.4, 10.2, 10.3.1, 10.3.2, 10.4.2, 10.4.4, 10.9, 10.10, 10.14, 11.12.6, and 12.2. Revised Tables 1.2a, and 1.10, Deleted Sections 2.22 and 7.12. Added Attachment III.

Pace Analytical®
National Center for Testing & Innovation

STANDARD OPERATING PROCEDURE

TITLE: Determination Metals and Trace Elements in Various Matrices by ICP-AES (EPA Methods 6010B, 6010C, 6010D [ICP-OES], and 200.7) Including Hardness (EPA Methods 200.7 and 6010B/C/D and SM 2340B)

ISSUER: Pace National – Mt. Juliet, Tennessee

Version	Date	Description of Revisions
18	10/28/2016	Technical and quality review and update. Update per South Carolina DHEC correspondence of 6/24/16. Header and signature block re-formatting. Revised SOP title. Revised Sections 1.1, Table 1.2a, 1.3, 1.6, 1.10, 2.12, 2.15, 5.6, 7.5.2, 7.10.1, 9.1, 10.2, 10.3.1, 10.4.2, 10.4.3, 10.4.5, 10.5, 10.6.1, 10.6.2, 10.7.1, 10.8.1.2, 10.8.2.2, 10.10.1, 10.16, and Attachment III Table 2. Deleted Table 1.2b. Deleted Sections 2.2, 2.12, 2.13, 2.14, 2.15, 2.17, 2.20, through 2.33, 3.5, 4.8, 7.14, 7.16, 8.1.3, 9.7 through 9.10, 9.13, 10.4.5.1, 10.4.5.2, 10.4.5.3, 10.10.3, 13.5, 14.7, 14.8, 14.9, and 14.10. Added Sections 2.14, 7.10.3 and all subsections, 9.11, 9.12, 10.1.1, 10.6.4, 10.6.5, 10.8.1.2, 10.9.1 and all subsections, 10.11.1 and all subsections, and 11.6.1.
19	11/30/2017	Update in response to A2LA audit finding CAR2872. Changed ESC logo. Updated Sections 1.5, 3.1, 7.9, 10.11, 14.1, 14.2, 14.3, 14.4, 14.5, 14.6, 14.7, 14.8, 14.9, and Attachment III Table 5.
20	7/27/18	Update in response to WI and AZ audit findings. Changed logo and references to “ESC” to “Pace National”. Revised Section 7.14, 10.4.4, and 10.7.3. Added State Note to Sections 10.8.1.2 and 10.11. Also added 6010C note to Section 10.13
21	9/10/18	Update in response AZ audit finding CAR3296. Revised Post-Spike acceptance criteria to ±20% in Section 10.9.1.2

Superseded Versions (Pace National):

Date	Description of Revisions
1/27/2019	Technical and quality review and update. Deleted header, footer and signature. Revised sections 1.0, 1.3, 1.5, 1.6, 1.7, 1.13, 1.13.1, 2.8.1(2.7.1), 2.8.2(2.7.1), 4.2, 4.4, 4.7, 6.2, 6.3, 6.4, 6.5, 6.6, 7.1, 7.2, 7.3, 7.4, 7.5.1, 7.5.3, 7.6, 7.7, 7.8, 7.9, 7.10, 7.10.1, 7.10.2, 7.13, 9.5, 9.6, 9.9, 10.1, 10.2, 10.3, 10.4, 10.4.3, 10.4.5, 10.5, 10.6.3, 10.6.4, 10.6.5, 10.7.3, 10.8, 10.8.1, 10.8.1.2, 10.8.2, 10.8.3, 10.10.1, 10.11, 10.11.1.2, 10.12, 10.13, 10.16, 11.1, 11.2, 11.12.6, 12.1, 12.2, 14.2, 14.6, 14.7 and 14.8. Revised Table 1.10. Added section 7.5 and renumbered as necessary. Deleted sections 7.10.3, 7.10.3.1, 7.10.3.2, 7.11, 7.12, 7.14, 10.9.1, 10.9.1.1 and 10.9.1.2. Revised Attachment I. Revised Attachment II sections 4.10, 4.17 and 4.18.
5/7/2019	Technical and quality review and update. Revised sections 1.1, 1.2, 2.14, 8.1.1.2, 10.3.2, 10.9, 10.11, 10.11.1, 10.11.1.1, 10.11.1.2, 11.8, 11.12.6 and 12.1. Correctly numbered section 7.6.1.1.
6/19/2019	Added corporate header and footer. Revised based on LA DW Auditor’s comments. Revised Sections 8.1.6, 10.3.2, and Attachment I.
10/16/2019	Revised section 1.2 and 10.7.1. Added section 11.12.7.
2/4/2020	West Virginia audit response. Revised Section 10.8.2.2.
4/8/2020	Revision in response to West Virginia audit deficiency. Revised Sections 10.8.1.2 and 11.6.1.
8/13/2020	Technical and quality review and update. Revised header. Revised sections 2.7, 7.5, 7.11, 8.1.7, 10.3.2, 10.11, 11.8 and 11.10. Added sections 2.7.3, 2.7.4, 2.7.5, 7.11.3, 7.11.4, 7.11.5 and 7.12 and renumbered as necessary. Revised all subsections of 3.0.

Attachment II: Potential ICP interferences arising from analytes present in field samples at concentrations of 100mg/L

Analyte	Wavelength (nm)	Interferant[a,b] Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Ti	V
Aluminum	308.215	--	--	--	--	--	--	0.21	--	--	1.4
Antimony	206.833	0.47	--	2.9	--	0.08	--	--	--	0.25	0.45
Arsenic	193.696	1.3	--	0.44	--	--	--	--	--	--	1.1
Barium	455.403	--	--	--	--	--	--	--	--	--	--
Beryllium	313.042	--	--	--	--	--	--	--	--	0.04	0.05
Cadmium	226.502	--	--	--	--	0.03	--	--	0.02	--	--
Calcium	317.933	--	--	0.08	--	0.01	0.01	0.04	--	0.03	0.03
Chromium	267.716	--	--	--	--	0.003	--	0.04	--	--	0.04
Cobalt	228.616	--	--	0.03	--	0.005	--	--	0.03	0.15	--
Copper	324.754	--	--	--	--	0.003	--	--	--	0.05	0.02
Iron	259.940	--	--	--	--	--	--	0.12	--	--	--
Lead	220.353	0.17	--	--	--	--	--	--	--	--	--
Magnesium	279.079	--	0.02	0.11	--	0.13	--	0.25	--	0.07	0.12
Manganese	257.610	0.005	--	0.01	--	0.002	0.002	--	--	--	--
Molybdenum	202.030	0.05	--	--	--	0.03	--	--	--	--	--
Nickel	231.604	--	--	--	--	--	--	--	--	--	--
Selenium	196.026	0.23	--	--	--	0.09	--	--	--	--	--
Sodium	588.995	--	--	--	--	--	--	--	--	0.08	--
Thallium	190.864	0.30	--	--	--	--	--	--	--	--	--
Vanadium	292.402	--	--	0.05	--	0.005	--	--	--	0.02	--
Zinc	213.856	--	--	--	0.14	--	--	--	0.29	--	--

[a] Dashes indicate that no interference was observed even when interferents were introduced at the following levels:

Al -	1000 mg/L	Mg -	1000 mg/L
Ca -	1000 mg/L	Mn -	200 mg/L
Cr -	200 mg/L	Tl -	200 mg/L
Cu -	200 mg/L	V -	200 mg/L
Fe -	1000 mg/L		

[b] The figures recorded as analyte concentrations are not the actual observed concentrations; to obtain those figures, add the listed concentration to the interferant figure.
[c] Interferences will be affected by background choice and other interferences may be present.

NOTE: Using the above table, if analyzing for Lead in a sample containing 1000mg/L Aluminum, the lead results could demonstrate a high bias of 0.17mg/L. (If the sample contained 10000mg/L of Al, the bias in lead could be 1.7mg/L), if background corrections are not accurately applied by the instrument.

Attachment III: DoD Requirements

1.0 Equipment/Instrument Maintenance

Instrument maintenance must be performed routinely to optimize instrument performance and improve chromatography. Commonly performed maintenance includes changing pump tubing, replacing the torch, cleaning the nebulizer, etc. A new calibration curve must be analyzed following any major maintenance performed on the analytical system.

2.0 Computer Hardware and Software

QTegra, Version 2.4

3.0 Troubleshooting

Table 1. GC Troubleshooting Guide

Problem	Cause	Treatment
Poor Precision	Nebulizer Pressure	Pressure should be about 0.15 mPa for aqueous solutions. If pressure is substantially higher, clean the nebulizer orifice or replace it entirely.
	Pooling in Spray Chamber	Usually caused by an oily film in the spray chamber. Aspirate 0.1% HF solution for about 20 seconds or 0.01% Triton X-100 solution.
	Center Tube	Replace the tube.
	Capillary Tubing	Air bubble migration through tubing should be smooth and consistent. Replace kinked/ pinched tubing.
	Peristaltic Pump	Adjust platen pressure. Check for leaks. Replace damaged pump.
Poor Accuracy	Pump Rate	Ensure the flush pump rate is the same as the analysis pump rate.
	Flush Time	Ensure proper time set for adequate rinse (typically 30 seconds).
Poor Detection Limits	Dirty Window or Mirror	Clean or replace dirty components.

4.0 Other Requirements

4.1 All hardcopy laboratory notebooks must be reviewed by the Supervisor, or their designee, on a monthly basis.

4.2 If not self-explanatory (e.g., a typo or transposed number), corrections to technical and quality records shall also include a justification for the change.

4.3 A person performing a manual integration must sign and date each manually integrated chromatogram and record the rationale for performing manual integration. Electronic signatures are acceptable.

4.4 The results of calibration and verification of support equipment must be within the specifications required of the application for which this equipment is used or the equipment must be removed from service until repaired. Calibration and verification records, including those of established correction

factors, must be maintained. In the absence of method-specific requirements, the minimum requirements are as follows:

Table 2. Support Equipment Checks

Performance Check	Frequency	Acceptance Criteria
Balance calibration check [Using two standard weights that bracket the expected mass]	Daily prior to use	Top-loading balance: ±2% or ±0.02g, whichever is greater Analytical balance: ±0.1% or ±0.5 mg, whichever is greater
Verification of standard mass [Using weights traceable to the International System of Units (SI) through a NMI	Every 5 years	Certificate of Calibration from ISO/IEC 17025 accredited calibration laboratory
Monitoring of refrigerator/freezer temperatures	Daily (i.e. 7 days per week) [use MIN/MAX thermometers or data loggers equipped with notification of out of control event capabilities if personnel not available to record daily]	Refrigerators: 0˚C to 6˚C Freezers: ≤-10˚C
Thermometer verification check [Using a thermometer traceable to the SI through an NMI] [Performed at two temperatures that bracket the target temperature(s). Assume linearity between the two bracketing temperatures.] [If only a single temperature is used, at the temperature of use]	Liquid in glass: Before first use and annually Electronic: Before first use and quarterly	Apply correction factors or replace thermometer
Volumetric labware	Class B: By lot before first use Class A and B: Upon evidence of deterioration	Bias: Mean within ±2% of nominal volume Precision: RSD ≤1% of nominal volume (based on 10 replicate measurements)
Non-volumetric labware [Applicable only when used for measuring initial sample volume and final extract/ digestates volume]	By lot before first use or upon evidence of deterioration	Bias: Mean within ±3% of nominal volume Precision: RSD ≤3% of nominal volume (based on 10 replicate measurements)
Mechanical volumetric pipette	Quarterly	Bias: Mean within ±2% of nominal volume Precision: RSD ≤1% of nominal volume (based on minimum of 3 replicate measurements) [Note: for variable volume pipettes, the nominal volume is the volume of use]
Glass microliter syringe	Upon receipt and upon evidence of deterioration	General Certificate of Bias & Precision upon receipt Replace if deterioration is evident
Drying oven temperature check	Daily prior to and after use	Within ±5% of set temperature

Table 2. Support Equipment Checks		
Performance Check	**Frequency**	**Acceptance Criteria**
Water purification system	Daily prior to use	See method blank criteria given in Section 4.20 of this addendum

4.5 The expiration date of the prepared standard shall not exceed the expiration date of the primary standard. All containers must bear a preparation date.

4.6 To avoid preparing non-representative samples, the laboratory shall not "target" within a relatively small mass range (e.g., 1.00 ± 0.01g) because such targeting will produce non-representative subsamples if the sample has high heterogeneity. The laboratory shall not manipulate the sample material so the sample aliquot weighs exactly 1.00g ± 0.01g, as an example.

4.7 In the absence of project-specific requirements, the minimum standard data qualifiers to be used are:

U Analyte was not detected and is reported as less than the LOD or as defined by the customer. The LOD has been adjusted for any dilution or concentration of the sample.

J The reported result is an estimated value (e.g., matrix interference was observed or the analyte was detected at a concentration outside the quantitation range).

B Blank contamination. The recorded result is associated with a contaminated blank.

N Non-target analyte. The analyte is a tentatively identified compound using mass spectrometry or any non-customer requested compounds that are tentatively identified.

Q One or more quality control criteria failed (e.g., LCS recovery, surrogate spike recovery, or CCV recovery).

Additional data qualifiers may be used, or different letters or symbols to denote the qualifiers listed above, as long as they are appropriately defined and their use is consistent with project-specific requirements (e.g., QSM 5.0, the contract, and project-planning documents).

4.8 If the time of the sample collection is not provided, assume the most conservative time of day. For the purpose of batch processing, the start and stop dates and times of the batch preparation shall be recorded.

4.9 Each preparation method listed on the scope of accreditation must have quarterly LOD/LOQ verifications. However, not all possible combinations of preparation and cleanup techniques are required to have LOD/LOQ verifications. If LOD/LOQ verifications are not performed on all combinations, the laboratory must base the LOD/LOQ verifications on the worst case basis (preparation method with all applicable cleanup steps).

4.10 After each MDL determination, the laboratory must establish the LOD by spiking a quality system matrix at a concentration of at least 2 times but no greater than four times the MDL. This spike concentration establishes the LOD and the concentration at which the LOD shall be verified. It is specific to each suite of analyte, matrix, and method (including sample preparation). The following requirements apply to the initial LOD establishment and to the LOD verifications:

- The apparent signal to noise (S/N) ratio at the LOD must be at least three and the results must meet all method requirements for analyte identification (e.g., ion abundance, second column confirmation, or pattern recognition). For data systems that do not provide a

measure of noise, the signal produced by the verification sample must produce a result that is at least three standard deviations greater than the mean method blank concentration. This is initially estimated based on a minimum of four method blank analyses and later established with a minimum of 20 method blank results.

- If the LOD verification fails, then the laboratory must repeat the MDL determination and LOD verification or perform and pass two consecutive LOD verifications at a higher spike concentration and set the LOD at the higher concentration.
- The laboratory shall maintain documentation for all MDL determinations and LOD verifications.
- The DL and LOD must be reported for all analyte-matrix-methods suites, unless it is not applicable to the test or specifically excluded by project requirements.

4.11 The LOD shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOD verifications on a one per batch basis. All verification data will be in compliance, reported, and available for review.

4.12 For DoD, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.

4.13 All initial instrument calibrations must be verified with a standard obtained from a second manufacturer prior to analyzing any samples. The use of a standard from a second lot obtained from the same manufacturer (independently prepared from different source materials) is acceptable for use as a second source standard. The concentration of the second source standard shall be at or near the midpoint of the calibration range. The acceptance criteria for the initial calibration verification must be at least as stringent as those for the continuing calibration verification.

4.14 Exclusion of calibration points without documented scientifically valid technical justification is not permitted.

4.15 The concentration of the CCV standard shall be greater than the low calibration standard and less than or equal to the midpoint of the calibration range.

4.16 All CCVs analyzed must be evaluated and reported. If a CCV fails, reanalysis or corrective actions must be taken.

- If a CCV fails, the laboratory can immediately analyze two additional consecutive CCVs (immediately is defined as starting a consecutive pair within one hour; no samples can be run between the failed CCV and the two additional CCVs). This approach allows for spurious failures of analytes to be reported without reanalysis of samples. Any corrective actions that change the dynamics of the system (e.g., clip column, clean injection port, run blanks) requires that all samples since the last acceptable CCV be reanalyzed.
- Both of these CCVs must meet acceptance criteria in order for the samples to be reported without reanalysis.
- If either of these two CCVs fail or if the laboratory cannot immediately analyze two CCVs, the associated samples cannot be reported and must be reanalyzed.
- Corrective action(s) and recalibration must occur if the above scenario fails. All affected samples since the last acceptable CCV must be reanalyzed.

- Flagging of data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory must notify the client prior to reporting data associated with a failed CCV.

4.17 The results of all MS/MSDs must be evaluated using the same acceptance criteria used for the DoD LCS limits (see Addendum Tables 3 and 4) or project limits, if specified. If the specific analyte(s) are not available in the Addendum Tables 3 and 4, the laboratory shall use their LCS in-house limits (see the LIMS) as a means of evaluating MS/MSDs. The MS and MSD must be spiked with all reported analytes.

4.18 Surrogate spike results shall be compared with DoD LCS limits (see Addendum Tables 3 and 4) or acceptance criteria specified by the client. If these criteria are not available, the laboratory shall compare the results with its in-house statistically established LCS criteria (see the LIMS).

4.19 The method blank shall be considered to be contaminated if:

- The concentration of any target analyte (chemical of concern) in the blank exceeds 1/2 the LOQ and is greater than 1/10th the amount measured in any associated sample, or 1/0th the regulatory limit, whichever is greater;
- The concentration of any common laboratory contaminant in the blank exceeds the LOQ;
- If a method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the LOD. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers.

4.20 Sporadic Marginal Exceedances are not allowed for target analytes (chemicals of concern as identified by a project) without project-specific approval. Target analytes are considered those few analytes that are critical for the success of a project (such as risk drivers) where sporadic marginal exceedances cannot be allowed. Laboratories should consult with clients whenever long lists of analytes are requested for analysis to determine if marginal exceedances will not be allowed.

4.21 DoD considers the same analyte exceeding the LCS control limit two (2) out of three (3) consecutive LCS to be indicative of non-random behavior, which requires corrective action and reanalysis of the LCS.

Table 3. LCS Control Limits – Method 6010 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
7429-90-5	Aluminum	6258	96.7	7.5	74	119
7440-36-0	Antimony	5997	96.4	5.7	79	114
7440-38-2	Arsenic	9530	96.2	4.9	82	111
7440-39-3	Barium	9236	98.3	5	83	113
7440-41-7	Beryllium	6799	97.8	5.1	83	113
7440-42-8	Boron	2312	93	7.1	72	114
7440-43-9	Cadmium	9466	97.5	5.3	82	113
7440-70-2	Calcium	6347	98.1	5.8	81	116
7440-47-3	Chromium	9598	98.9	4.6	85	113
7440-48-4	Cobalt	6725	98.7	4.5	85	112
7440-50-8	Copper	7839	99.1	6	81	117
7439-89-6	Iron	5746	99.7	6.1	81	118
7439-92-1	Lead	10160	96.8	5.1	81	112
7439-93-2	Lithium	551	98.8	4.5	85	112
7439-95-4	Magnesium	6283	96.1	6.1	78	115
7439-96-5	Manganese	6732	99.1	4.9	84	114
7439-98-7	Molybdenum	4424	98.7	5.7	82	116
7440-02-0	Nickel	7412	98.1	4.9	83	113
7723-14-0	Phosphorus	189	103.1	3.8	92	114
7440-09-7	Potassium	6574	98.3	5.8	81	116
7782-49-2	Selenium	8862	94.5	5.6	78	111
7440-22-4	Silver	9105	97.3	5	82	112
7440-23-5	Sodium	5825	100.1	5.8	83	118
7440-24-6	Strontium	2573	98.5	5	83	114
7440-28-0	Thallium	6416	96.8	4.6	83	111
7440-31-5	Tin	2780	100.1	6.6	80	120
7440-32-6	Titanium	2107	98.2	5.2	83	114
7440-61-1	Uranium	109	97.4	5.2	82	113
7440-62-2	Vanadium	6934	98.3	5.4	82	114
7440-66-6	Zinc	7882	97.4	5	82	113

Table 4. LCS Control Limits – Method 6010 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
7429-90-5	Aluminum	11532	100	4.8	86	115
7440-36-0	Antimony	10737	100.2	4.2	88	113
7440-38-2	Arsenic	14123	99.9	4.3	87	113
7440-39-3	Barium	14476	100.3	4.1	88	113
7440-41-7	Beryllium	11552	100.4	4	89	112
7440-69-9	Bismuth	147	95.8	3.2	86	105
7440-42-8	Boron	3871	98.8	4.8	85	113
7440-43-9	Cadmium	13922	100.8	4.1	88	113
7440-70-2	Calcium	11382	100	4.2	87	113
7440-47-3	Chromium	15027	101.1	3.9	90	113
7440-48-4	Cobalt	11824	101.2	4.2	89	114
7440-50-8	Copper	12910	100.2	4.6	86	114
7439-89-6	Iron	13797	100.7	4.7	87	115
7439-92-1	Lead	14391	99.3	4.4	86	113
7439-93-2	Lithium	938	100.7	5.3	85	117
7439-95-4	Magnesium	11423	98.8	4.8	85	113
7439-96-5	Manganese	12767	101.9	4.1	90	114
7439-98-7	Molybdenum	8251	101.1	4	89	113
7440-02-0	Nickel	12699	100.5	4.1	88	113
7440-05-3	Palladium	492	99.8	4	88	112
7723-14-0	Phosphorus	203	100.5	4.2	88	113
7440-09-7	Potassium	11006	99.9	4.7	86	114
7782-49-2	Selenium	13264	98.5	5.2	83	114
7440-21-3	Silicon	1525	100.6	6.1	82	119
7440-22-4	Silver	13770	99.1	5.1	84	115
7440-23-5	Sodium	10893	100.9	4.7	87	115
7440-24-6	Strontium	3782	101.3	3.8	90	113
7704-34-9	Sulfur	145	100.7	3.9	89	112
7440-28-0	Thallium	10063	99.5	4.7	85	114
7440-31-5	Tin	4502	101.3	4.4	88	115
7440-32-6	Titanium	5625	101.1	3.4	91	111
7440-61-1	Uranium	223	101.3	5.8	84	119
7440-62-2	Vanadium	12032	100.2	3.6	90	111
7440-66-6	Zinc	13549	100.6	4.6	87	115

Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Linear Dynamic Range (LDR) or high-level check standard	At initial set up and checked every 6 months with a high standard at the upper limit of the range.	Within ± 10% of true value.	Dilute samples within the calibration range, or re-establish/ verify the LDR.	Flagging is not appropriate.	Data cannot be reported above the high calibration range without an established/passing high-level check standard.
Initial Calibration (ICAL) for all analytes	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, r2 ≥ 0.99.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	Minimum one high standard and a calibration blank. No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 10% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	After every 10 field samples, and at the end of the analysis sequence.	All reported analytes within ± 10% of the true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since the last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Low-Level Calibration Check Standard (LLCCV)	Daily	All reported analytes within ± 20% of true value.	Correct problem and repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed without a valid low-level calibration check standard (LLCCV). Low-level calibration check standard should be less than or equal to the LOQ. If the concentration of the lowest calibration standard is less than or equal to the LOQ, the lowest standard may be re-quantified against the calibration curve as a LLCCV. Otherwise, a separate standard must be analyzed as LLCCV prior to the analysis of any samples.

Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch.	The absolute values of all analytes must be < ½ LOQ or < 1/10th the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all QC and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Non-detects associated with positive blank infractions may be reported. Sample results >10X the LOQ associated with negative blanks may be reported. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Initial and Continuing Calibration Blank (ICB/CCB)	Immediately after the ICV and immediately after every CCV.	The absolute values of all analytes must be < ½ LOQ or < 1/10th the amount measured in any sample.	ICB: Correct problem and repeat ICV/ICB analysis. If that fails, rerun ICAL. All samples following the last acceptable Calibration Blank must be reanalyzed. CCBs may not be reanalyzed without reanalysis of the associated samples and CCV(s).	Flagging is not appropriate.	Results may not be reported without a valid calibration blank. Non-detects associated with positive blank infractions may be reported. Sample results >10X the LOQ associated with negative blanks may be reported. For CCB, failures due to carryover may not require an ICAL.

Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Interference Check Solutions (ICS) (also called Spectral Interference Checks)	After ICAL and prior to sample analysis.	ICS-A: Absolute value of concentration for all non-spiked project analytes <1/2 LOQ (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within ± 20% of true value.	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the failed ICS.	All analytes must be within the LDR. ICS-AB is not needed if instrument can read negative responses.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all reported analytes. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J flag if acceptance criteria are not met and explain in the case narrative.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to the source(s) of difference (i.e., matrix effect or analytical error).
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.

Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Dilution Test	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within ± 10% of the original measurement.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J flag if acceptance criteria are not met and explain in the case narrative.	Only applicable for samples with concentrations > 50 x LOQ (prior to dilution). Use along with MS/MSD and PDS data to confirm matrix effects.
Post-Digestion Spike (PDS) Addition (ICP only)	Perform if MS/MSD fails. One per preparatory batch (using the same sample as used for the MS/MSD if possible).	Recovery within 80-120%.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J flag if acceptance criteria are not met and explain in the case narrative.	Criteria applies for samples with concentrations <50 X LOQ prior to dilution.
Method of Standard Additions (MSA)	When dilution test or post digestion spike fails and if required by project.	NA	NA	NA	Document use of MSA in the case narrative.

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Daniel J. Wright
General Manager 1

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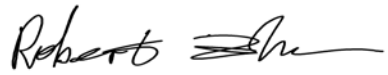
Kelly M. Nance
Quality Manager



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Robert Zhu
Technical Specialist

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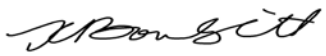
Bradley E. Belding
Operations Manager



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Kristina P. Bouknight
Environmental Health and Radiation Safety Officer

PROCESS STANDARD OPERATING PROCEDURE
TITLE: Hazardous and Non-Hazardous Laboratory Waste Management Plan
ISSUER: Pace ENV - Local Quality - WCOL

Table of Contents

1.0 Purpose

This standard operating procedure (SOP) describes the systems, processes and procedures that this location uses to manage generated wastes.

2.0 Scope and Application

The policies and procedures contained in this SOP are applicable to all personnel responsible for all aspects of waste handling and management.

This SOP is applicable to all processes that involve generated waste, and is designed to assist its operations in adhering to regulations set forth in the following federal statutes: Resource Conservation and Recovery Act (RCRA), Clean Water Act (CWA), Toxic Substances Control Act (TSCA), and DOT Title 49, and Transportation (parts 100-199). Particular attention is given to local pretreatment standards covering discharges to publicly owned treatment works (POTW) when performing elementary neutralization on acidic and basic waste. The local standards are based in part upon provisions in the National Pretreatment Standards and Prohibited Discharge Standards.

The degree to which RCRA regulations apply to Pace facilities is dependent upon the generator status of the operation. Under the federal rules (state requirements may be more stringent or give the classes a slightly different name) there are three different classes of hazardous waste generators based upon the amount of waste generated in a month to month time frame.

3.0 Summary

Pace Analytical Services (Pace) acknowledges its obligation to the responsible management of the environment and its resources. Pace senior management is committed to operating in such a way that meets or exceeds the state and federal laws governing waste management and encourages the use of best practices to reduce, reuse and recycle waste material where possible.

It is Pace's policy to minimize the amount of hazardous waste it produces and to reduce the hazardous properties of those wastes whenever practical within regulatory compliance. This can be achieved by periodic auditing of all processes producing hazardous waste; reduction of sample volume delivered by the client; return of excess sample material to clients whenever practical and economical; investigation of new technologies that might require smaller volumes of sample, or produce fewer or less hazardous by-products; implementation of lab cleaning procedures that reduce the volume of cleaning residue; recycling of hazardous materials; and investigation of new treatment technologies that are comprehensively destructive or are effective in reducing the volume or hazardous qualities of the wastes produced.

Pace facilities that generate waste must initially contact the EPA to obtain an ID number. Each unique type of generated waste is classified and characterized into waste streams according to procedures in 40 CFR 261. The amount of waste the facility generates determines the Generator Status of a lab, which in turn determines how long and how much waste can accumulate. Pace is ultimately responsible for the waste it generates, and is required to obey any and all regulations during the process of creating, accumulating, disposing, and releasing waste to a TSDF for final disposal. Documentation is kept to prove all regulations have been obeyed.

4.0 Definitions

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

Acutely Hazardous Waste - A waste which is hazardous as identified with an (H) Hazard Code in the lists of Hazardous Waste in 40 CFR Part 261, Subpart D, Sections 261.30, 261.31 and 261.33.

Animal and Plant Health Inspection Service (APHIS) - an agency of the USDA responsible for protecting animal health, animal welfare, and plant health. APHIS is the lead agency for collaboration with other agencies to protect U.S. agriculture from invasive pests and diseases.

Clean Air Act - The Federal Clean Air Act, 42 U.S.C. 7401, and amendments thereto amending 42 U.S.C. 1857 et.seq.

Confined Space – A space that is large enough and so configured that an employee can bodily enter and perform assigned work; and has limited or restricted means for entry or exit (for example, tanks, vessels, silos, storage bins, hoppers, vaults, and pits are spaces that may have limited means of entry); and is not designed for continuous employee occupancy.

Container - Any device material is stored, transported, treated, disposed of, or otherwise handled.

Contingency Plan - A document setting out an organized, planned, and coordinated course of action to be followed in case of fire, explosion, or release of hazardous waste or hazardous waste constituents which could threaten human health or the environment.

Designated Hazardous Waste Storage Area / Central Accumulation Area [CAA]- Area used to hold hazardous waste for a temporary period, at the end of which the hazardous waste is treated, disposed of, or stored elsewhere. This is the storage area into which hazardous waste from the laboratory (e.g., satellite waste) is moved.

DOT - The United States Department of Transportation.

DTSC – Department of Toxic Substances Control.

Elementary Neutralization Unit – A device which: (1) is used for neutralizing wastes which are hazardous only because they exhibit the corrosivity characteristic defined in 40 CFR 261.22 or are listed in Subpart D of Part 261; and (2) meets the definition of tank, container, transport vehicle, or vessel in 40 CFR 260.10.

EPA - The United States Environmental Protection Agency.

EPA Hazardous Waste Number - The EPA number assigned to each EPA hazardous waste identified in 40 CFR Part 260, Subpart D - Lists of Hazardous Wastes.

EPA Identification Number - The site-specific number assigned to each generator, transporter, and TSDF upon approval of a notification form.

Federal Clean Water Act - 33 U.S.C. 1251, et. Seq.

Foreseeable Emergency - Any fire, explosion, or sudden or non-sudden release of hazardous waste or hazardous waste constituents to the air, soil, or surface water, which could threaten human health or the environment.

Generator - Any person, by site who owns or operates a facility where hazardous waste is generated, i.e. Pace.

Hazardous Waste Coordinator - The Pace employee responsible for creating, guiding, and implementing all hazardous waste management operations.

Hazardous Waste - As defined in 40 CFR Part 261, Subparts B and C, a solid, semi-solid, liquid or contained gaseous waste, or any combination of these wastes.

Which, because of either quantity, concentration, physical, chemical, or infectious characteristics may:

- Cause or contribute to an increase in mortality or an increase in irreversible or incapacitating reversible illness; or
- Pose a substantial present or potential hazard to human health or the environment when improperly treated, stored, transported, disposed of or otherwise mismanaged.
- Or which has been identified as having a characteristic of hazardous waste by the EPA using the criteria established under 40 CFR Part 261, Subpart C, or as listed under Sections 261.31, 261.32, 261.33, and 261.34. Such wastes include, but are not limited to, those which are reactive, toxic, corrosive, ignitable, irritants, strong sensitizers or which generate pressure through decomposition, heat or other means. Such wastes do not include radioactive substances that are regulated by the Atomic Energy Act of 1954, as amended. A waste is considered hazardous if it is listed or it fits into one of four categories. These categories are as follows:
  - Ignitable – (40 CFR 261.21, Waste Code D001) A flash point of less than 60°C/140°F.
  - Corrosive – (40 CFR 261.22, D002) A pH of less than or equal to 2.0 or greater than or equal to 12.5.
  - Reactive – (40 CFR 261.23, Waste Code D003) Reactive wastes exhibit one or more of the following characteristics:
    - It is unstable and can undergo a violent change without detonating.
    - It can react violently with water.
    - When mixed with water it can generate toxic gases, vapors, or fumes in a quantity sufficient to present a danger to human health or the environment.
    - It is cyanide or sulfide bearing waste that, when exposed to pH conditions between 2.0 and 12.5, can generate gases, vapors, or fumes that can present a danger to human health or the environment.
    - It is capable of detonation or explosive reaction if it is subjected to a strong initiating source or if heated under confinement.
    - It is readily capable of detonation or explosive decomposition or reaction at standard temperature and pressure.
    - It is a forbidden explosive as defined in 49 CFR 173.51, or a Class A explosive as defined in 49 CFR 173.53, or a Class B explosive as defined in 49 CFR 173.88.
  - Toxic – (40 CFR 261.24, Waste Codes D004-D043) A solid waste that contains a toxic concentration of a contaminant listed in 40 CFR 261.24, Table 1. A toxic waste is given any and all D-codes that apply to the particular material.

Hazardous Waste Constituent - A substance, compound, or element listed as hazardous waste in EPA 40 CFR 261.

Lab Pack Material - A hazardous waste that does not match a listed Pace waste stream category.

Large Quantity Generator (LQG) - Any generator who generates at a rate greater than 1000 kilograms of hazardous waste per month.

Listed Wastes – Wastes that the EPA has determined to be hazardous. Wastes are categorized, based on their source and/or type, into 4 lists located in SC HWMR.61-79.261.31, 261.32, and 261.33. Each hazardous waste listed is assigned an EPA hazardous waste number, which is specific to the waste type, source, or constituents. For example, spent halogenated solvents such as methylene chloride (dichloromethane), tetrachloroethylene, trichloroethylene, 1,1,1-trichloethane, and chlorobenzene are found on the F list. Any spent halogenated solvent wastes containing these constituents would be considered a hazardous waste based on this listing and would subsequently carry the hazards waste code F002, as identified by the EPA.

Manifest - As defined in 40 CFR Part 262, Subpart B, namely “the form used for identifying the origin, quantity composition, routing and destination of hazardous waste”.

Plant Protection and Quarantine (PPQ) – A program within APHIS which attempts to safeguard agriculture and natural resources in the U.S. against the entry, establishment, and spread of animal and plant pests and noxious weeds.

Profiles – Profiles are formal declarations describing a waste stream. Profiles are used by waste treatment facilities to characterize hazardous and non-hazardous waste for destruction or disposal. Profiles must be accurate and up to date, as the waste characteristics define how a waste can be legally transported, categorized, and destroyed. Generally, profiles are given number identifications that are used to label the waste. A list of PAS-WCOL’s approved waste profiles is posted in each laboratory area.

Regulated Soil – Soil from foreign countries, U.S. territories and areas within states that are under Federal quarantine that can be moved into or through continental U.S. only if conditions and safeguards prescribed by the USDA and APHIS are met.

Sample - Any solid material/waste, water, soil, or air that is collected for the sole purpose of being tested to determine its characteristics or composition.

Samples are not subject to any requirements of 40 CFR Part 261.5 or Parts 262 through 267 or Part 270 or Part 124 or to the notification requirements of Section 3010 of RCRA, when:

- The sample is being transported to a laboratory for the purpose of testing; or
- The sample is being transported back to the sample collector after testing; or
- The sample is being stored by the sample collector before transport to a laboratory for testing; or
- The sample is being stored in a laboratory before testing; or
- The sample is being stored in a laboratory after testing but before it is returned to the sample collector; or

- The sample is being stored temporarily in the laboratory after testing for a specific purpose (for example, until conclusion of a court case or enforcement action where further testing of the sample may be necessary).

In order to qualify for the exemption above, a sample collector shipping samples to a laboratory and a laboratory returning samples to a sample collector must:

- Comply with U.S. Department of Transportation (DOT), U.S. Postal Service (USPS), or any other applicable shipping requirements; or
- Comply with the following requirements if the sample collector determines that DOT, USPS, or other shipping requirements do not apply to the shipment of the sample:
    - Assure that the following information accompanies the sample:
        - The sample collector's name, mailing address, and phone number;
        - The laboratory's name, mailing address, and phone number;
        - The quantity of the sample;
        - The date of shipment; and
        - A description of the sample.
    - Package the sample so that it does not leak, spill, or vaporize from its packaging.
    - This exemption does not apply if the laboratory determines that the waste is hazardous but the laboratory is no longer meeting any of the conditions stated

Satellite Waste or Laboratory Satellite Waste or Satellite Accumulation Area [SAA] - Hazardous waste generated by Pace that is at or near any point of generation and under the control of the operator. Satellite accumulation provisions allow generators to accumulate up to 55 gallons of hazardous waste (or 1 quart of acute hazardous waste) in containers without starting the storage clock as described in Section 3.4.

Satellite Waste Container - Any portable device used to accumulate laboratory generated waste prior to transfer to the hazardous waste storage area.

Small Quantity Generator (SQG) - A generator who produces no more than 1000 kilograms of hazardous waste (or a total of 1000 kilograms of any residue or contaminated soil, waste or other debris resulting from the cleanup of a spill, into or on any land or water, or any acute hazardous waste) in a calendar month. The total amount of hazardous waste which may be accumulated on-site is 6000 kilograms.

TSDF - A Treatment/Storage/Disposal Facility.

Universal Waste – any hazardous wastes that are subject to universal waste requirements. The federal universal wastes include Batteries as described in §273.2; Pesticides as described in §273.3; Mercury-containing equipment as described in §273.4; Lamps as described in §273.5; and Aerosol cans as described in §273.6. State or local regulations may allow for additional universal waste designations.

Very Small Quantity Generator - A generator who produces no more than 100 kilograms of hazardous waste or one kilogram of acutely hazardous waste (or a total of 100 kilograms of any residue or contaminated soil, waste or other debris resulting from the cleanup of a spill, into or on any land or water, or any acute hazardous waste) in a calendar month. The total amount of hazardous waste which may be accumulated on-site is 1000 kilograms.

Waste Stream - The generic profile of chemical and physical properties that satellite wastes exhibit.

5.0 Procedure

5.1. Generator's US EPA Identification Number

All Pace facilities that generate hazardous waste must have a Generator's US EPA Identification Number. The ID number is obtained through the applicable state or EPA region's office by completing EPA form 8700-12, and must be completed before generating any hazardous waste. A new ID number is necessary when changing locations as the number is tied to the facility address. Pace only utilizes transporters and treatment, storage, or disposal facilities (TSDFs) that have EPA identification numbers for hazardous waste handling and meet the TSDF transfer requirements.

This facility's US EPA Identification Number is SCR000075879.

5.2. Generator Status

Hazardous Waste Generator Class	Quantity of Hazardous Waste Generated per Month	Generated Monthly Acute Hazardous Waste	Maximum Allowable Hazardous Waste Quantity on-site	Maximum Permitted Waste Accumulation Time
Very Small Quantity	<100kg	<1 kg	<1000kg	Unlimited
Small Quantity	100-1000kg	<1 kg	<6000kg	180 days (270 days if the waste must be sent >200 miles to TSDF)
Large Quantity	>1000kg	>1kg	Unlimited	90 days

Table 5.1 - Waste Generator Class Limits (federal categories, some operations may have different Hazardous Waste Generator Class names):

Based on Table 5.1, this facility is classified as a Large Quantity Generator.

5.3. Hazardous Waste Characterization and Classification

Hazardous waste classification is the most critical step in establishing an effective, compliant waste-handling program. Laboratory wastes are classified using the criteria set forth under RCRA for ascertaining non-hazardous versus hazardous status, and this criterion is listed in the definition of hazardous waste in Section 4.0.

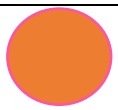
The laboratory generates wastes originating from several source types: materials and chemicals used to prepare and analyze samples (e.g., solvents, acids), unconsumed liquid and solid samples, certain types of batteries, automobile waste, and mercury from lamps and broken thermometers.

Unconsumed samples may include laboratory-contaminated sample residue (both liquid and solid) generated as part of digestion, extraction, etc., procedures used to prepare samples for analysis.

The following are the waste streams resulting from materials and chemicals used in the laboratory operation. Applicable information for each is given pertaining to packing, labeling, or listing on a manifest. A description of how the wastes are created, and the preferred method of final disposal for each, is included. The overriding principle in hazardous waste classification is application of a conservative formula based on all known or suspected hazards related to a waste material.

5.3.1.Corrosive Waste

Corrosive waste is generated in the majority of the departments in the laboratory. This waste stream consists primarily of spent or excess aqueous reagent solutions generated from preservatives, acid digestions of metals, impinger solutions or other corrosive solutions generated in the course of analysis. The predominant corrosives include hydrochloric acid, nitric acid and sulfuric acid, but corrosives also include bases. Varying concentrations of metals may be present dependent upon the composition of the reagents added. This waste stream only has the hazardous quality of being corrosive; therefore, if a waste has any additional hazardous waste quality (e.g., Toxic or Ignitable) it cannot be mixed with this stream. This stream is treated onsite in the elementary neutralization unit [ENU].

Corrosive Waste	
Profile Number	Elementary Neutralization Unit [ENU]
DOT Shipping Name	RQ Waste, Corrosive Liquid, N.O.S (i.e. corrosive material)
EPA Hazard Codes	D002
Container	55 Gallon Poly Closed Top
Average pH	≤2.0, ≥12.5
Disposal Method	Treatment by Neutralization
Hazard Label	Corrosive 
Core ID	1 

5.3.2.Chlorinated Solvent Waste / Solvent Waste (Mixed Solvents)

The Solvent Waste (Mixed Solvent) [Profile Shealy 10] waste stream consists primarily of methylene chloride with a very small amount of other organic solvents derived from extraction procedures performed on samples and from rinsing glassware. As a best practice, effort is made to have this waste stream as pure as possible in order to offer for recycling. This waste stream is rarely used. Waste may be stored at the point of generation in satellite accumulation containers. Satellite containers must be taken to the waste accumulation building and bulked into the drum labeled “Mixed Solvent Waste” [Profile Shealy 10].

Solvent Waste (Mixed Solvents)	
Profile Number	Shealy10
DOT Shipping Name	UN1992, Waste Flammable Liquids, Toxic, N.O.S. (Xylene, Chloroform), 3, (6.1), PG II
EPA Hazard Codes	D001, D002, F002, F003
Container	55G Poly closed top drum with vent cap
Average pH	4.1 – 10.0 SU
Disposal Method	Disposal to Treatment, Storage and/or Disposal Facility (TSDF)
Hazard Label	Flammable, Toxic 
Core ID	6 

The primary solvent waste (mixed solvents) waste stream is profile number CH162857 and consists of the following spent solvents: hexane, ether, methylene chloride, ethyl acetate, chloroform, and xylene. This waste stream is generated during samples extraction and rinsing procedures during several processes such as CTAS, MBAS, Flashpoint, and organic extraction procedures. Waste may be stored at the point of generation in satellite accumulation containers. Satellite containers must be taken to the waste accumulation building and bulked into the drum labeled “Mixed Solvent Waste” [Profile CH162857].

Solvent Waste (Mixed Solvents) / Mixed Flam & Halogenated Solvent	
Profile Number	CH162857
DOT Shipping Name	UN1992, Waste Flammable Liquids, Toxic, n.o.s. (Xylene, Chloroform), 3, 6.1, PG II
EPA Hazard Codes	D001, D022, F002, F003
Container	55G Poly Closed Top Drum
Average pH	4.1 to 10.0
Disposal Method	Disposal to Treatment, Storage and/or Disposal Facility (TSDF)
Hazard Label	Flammable, Toxic, Irritant 
Core ID	5 

5.3.3.COD Waste

COD Waste is specific waste that results from COD analysis. This waste comes from used and expired COD vials of samples and reagent. This stream has sulfuric acid, mercuric sulfate, potassium dichromate, and silver sulfate.

Spent vials generated during Chemical Oxygen Demand (COD) analysis may be stored at the point of generation in satellite containers. Satellite containers must be taken to the waste accumulation building and bulked into the drum labeled “COD Vials with Mercury” [Profile CH162853].

COD Waste [COD Vials (Containing Mercury)]	
Profile Number	CH162853
DOT Shipping Name	Corrosive Liquids, Toxic, N.O.S. 8, (6.1), II
EPA Hazard Codes	D002, D007, D009, D011
Container	30 Gallon poly open top drum
Average pH	≤ 2.0 SU
Disposal Method	Disposal to Treatment, Storage and/or Disposal Facility (TSDF)
Hazard Label	Corrosive, Toxic 
Core ID	13 

5.3.4.Hazardous Non-Aqueous Waste

Non-Aqueous liquid samples are treated as hazardous waste. There is a drum labeled “Hazardous Non-Aqueous Waste” [Profile 259531] for disposal of these samples. Non-Aqueous samples are removed from sample containers and emptied per the requirements set forth in section 5.8.2.

Hazardous Non-Aqueous Waste	
Profile Number	259531
DOT Shipping Name	Hazardous waste, liquid, n.o.s. (Lead, Chromium), 9, III
EPA Hazard Codes	D001, D002, D004, D007, D008, D011
Container	5 gallon poly jerrican or 15 gallon poly closed top drum
Average pH	≤ 2.0 SU
Disposal Method	Disposal to Treatment, Storage and/or Disposal Facility (TSDF)
Hazard Label	Miscellaneous 
Core ID	17 

5.3.5.Hazardous Aqueous Waste (Organics with pH > 2SU)

Hazardous Aqueous Waste consists of aqueous samples found to be flammable during the course of analysis are labeled with a flammability pictogram. Any sample bearing this pictogram must be segregated and disposed of as hazardous waste. Flammable hazardous aqueous samples are disposed of in the drum labeled “Hazardous Aqueous (Organics with pH > 2 SU) [Profile 259534].

Organic extraction process wastes generated during samples extraction may be stored at the point of generation in satellite accumulation containers. Satellite accumulation containers must be taken to the waste accumulation building and bulked in the drum labeled “Hazardous Aqueous Waste (Organics with pH > 2 SU) [Profile 259534]. The organic extraction processes that may produce this waste stream are EDB, DBCP, Herbicide, Continuous Liquid-Liquid Extraction and Oil and Grease.

Hazardous Aqueous (Organics with pH > 2 SU)	
Profile Number	259534
DOT Shipping Name	UN3286 Waste Flammable Liquids, Toxic, Corrosive, n.o.s. (Lead, Hydrochloric Acid), 3, (6.1, 8), PG II, RQ (1 lb)
EPA Hazard Codes	D001, D002, D004, D006, D007, D008, D011, F002
Container	55 gallon poly closed top drum
Average pH	> 2 SU
Disposal Method	Disposal to Treatment, Storage and/or Disposal Facility (TSDF)
Hazard Label	Corrosive, Toxic, Flammable 
Core ID	11 

5.3.6.Soil Samples in Methanol Vials

Soil samples in Methanol vials are excess samples from Volatile Organic Analysis Department (VOA) that are disposed of in the 55-gallon steel drum labeled “Soil in Methanol Vials” [Profile 270048]. Samples are not removed from their sample containers.

Soil Samples in Methanol Vials	
Profile Number	270048
DOT Shipping Name	UN1993, Flammable liquids, n.o.s. (Methanol), 3, PG II, RQ (D001)
EPA Hazard Codes	D001, F003
Container	55 Gallon Metal Drum
Average pH	4.1 – 10.0 SU
Disposal Method	Disposal to Treatment, Storage and/or Disposal Facility (TSDF)
Hazard Label	Flammable 
Core ID	7 

5.3.7.Lab Waste in Vials (Organics)

Lab waste in vials (Organics) waste stream consists of standards dissolved in solvents, acid clean-up wastes, sample extracts, and un-used sample collection bottles with solvent.

Standards Dissolved in Methanol, Acetone, Hexane, or Methylene Chloride - Standards dissolved in methanol, acetone, hexane, or methylene chloride (ampules) may be stored at the point of generation in satellite containers. Satellite containers must be taken to the waste accumulation area and bulked in the drum labeled "Lab Waste in Vials (Organics)" [Profile 275280].

Acid Clean-up Wastes - Spent sulfuric acid generated during acid clean-up procedures may be stored at the point of generation in satellite accumulation containers labeled "Lab Waste in Vials (Organics)" [Profile 275280]. Satellite waste containers must be transferred to the waste accumulation building whenever they become full.

Sample Extracts - Vials containing sample extracts may be stored at the point of generation in satellite containers. Satellite waste containers must be taken to the waste accumulation building and bulked into the 55-gallon drum labeled "Lab Waste in Vials (Organics)" [Profile 275280].

Un-Used Sample Collection Bottles – When sample bottles containing preservation are returned without being used, the preservation must be emptied from the bottle prior to bottle disposal. Acid and base preservations are emptied into satellite accumulation containers labeled "Elementary Neutralization Satellite." Satellites are bulked into an elementary neutralization unit and neutralized following the procedures outlined in section 5.8. Solvent preservations are bulked, without being poured off, into a 55-gallon drum labeled "Lab Waste in Vials (Organics)" [Profile 275280].

Lab Waste in Vials (Organics)	
Profile Number	275280
DOT Shipping Name	UN3286, Waste Flammable Liquids, Toxic, Corrosive, n.o.s. (Methylene chloride, Hexane), 3, (6.1, 8), PG I
EPA Hazard Codes	D001, D002, F002, F003
Container	55 Gallon Poly Open Top Drum
Average pH	4.1 – 10.0 SU
Disposal Method	Disposal to Treatment, Storage and/or Disposal Facility (TSDF)
Hazard Label	Flammable, Toxic, Corrosive 
Core ID	5 

5.3.8.Lab Waste in Vials (Acidic)

Lab waste in vials (acidic) profile consists of excess samples, samples screening vials, and auto-injector vials. The waste may be stored at the point of generation in satellite containers. Satellite containers must be taken to the waste accumulation area and bulked in the drum labeled "Laboratory Waste in Vials (Acidic)" [Profile 283339].

Lab Waste in Vials (Acidic)	
Profile Number	283339
DOT Shipping Name	UN1760, Corrosive Liquids, n.o.s. (Hydrochloric acid, water), 8, PG II, RQ (D002)
EPA Hazard Codes	D002
Container	55 Gallon Poly Open Top Drum
Average pH	≤ 2 SU
Disposal Method	Disposal to Treatment, Storage and/or Disposal Facility (TSDF)
Hazard Label	Corrosive 
Core ID	1 

5.3.9.Spent Synthetic Hydrocarbons

Spent vacuum pump oil may be stored at the point of generation in satellite containers. Satellite containers must be taken to the waste accumulation area and bulked in the container labeled “Spent Synthetic Hydrocarbons” [Profile 311190].

Spent Synthetic Hydrocarbons	
Profile Number	311190
DOT Shipping Name	Non-regulated material
EPA Hazard Codes	None
Container	5 Gallon Metal
Average pH	4.1 – 10.0 SU
Disposal Method	Disposal to Treatment, Storage and/or Disposal Facility (TSDF)
Hazard Label	None
Core ID	23 

5.3.10. Soil Samples in Glass Vials

Soils samples in glass vials waste stream consists of excess soil samples in glass vials and standard dissolved in water. Satellite containers must be taken to the waste accumulation area and bulked in the drum labeled “Soil Samples in Glass Vials” [Profile 318384]. Samples are not removed from their sample containers.

Soil Samples in Glass Vials	
Profile Number	318384
DOT Shipping Name	Non-regulated material (Glass vials, soils)
EPA Hazard Codes	None
Container	55 Gallon Metal Drum
Average pH	4.1 -10.0
Disposal Method	Disposal to Treatment, Storage and/or Disposal Facility (TSDF)
Hazard Label	None
Core ID	24 

5.3.11. Limestone

Limestone waste profile consists of used limestone from the lime-pit located in the elementary neutralization unit.

Limestone	
Profile Number	PACE-WCOL12
DOT Shipping Name	Non-regulated material
EPA Hazard Codes	None
Container	55 Gallon Metal or Poly Drum
Average pH	4.1 – 10.0 SU
Disposal Method	Disposal to Treatment, Storage and/or Disposal Facility (TSDF)
Hazard Label	None
Core ID	24 

5.3.12. Foreign Soil and/or Regulated Domestics

Foreign soil and/or regulated domestic soil (when applicable) is treated as hazardous waste and must be segregated from soil samples not from foreign or regulated domestic sites due to possible pest contamination from foreign sources. To mitigate this risk, foreign soil is treated as infectious waste. Due to the additional regulations applicable to all aspects of foreign soil analysis, characterization, and disposal, a separate policy has been written. For details on how to handle foreign soil, refer to Foreign Soil and/or Regulated Domestic Soil Samples SOP [HS SOP ME001J9].

Foreign Soil and/or Regulated Domestics	
Profile Number	323354
DOT Shipping Name	UN3077, Environmentally Hazardous Substances, Solid, N.O.S. (Soil), 9, PG I
EPA Hazard Codes	None
Container	55 Gallon Metal Drum
Average pH	4.1 – 10.0 SU
Disposal Method	Disposal to Treatment, Storage and/or Disposal Facility (TSDF)
Hazard Label	Miscellaneous
Core ID	24 

5.3.13. Fluorescent Bulbs

Universal waste lamps must be managed in a manner which prevents releases to the environment. Lamps must be accumulated in appropriate 4' or 8' boxes provided by PACE-WCOLs waste broker. These boxes must remain closed at all times unless lamps are actively being added to the box. The box must be labeled with a "Universal Waste Lamp" sticker and an accumulation start date indicating the date that the first lamp was added to the box.

Fluorescent Bulbs	
Profile Number	328285
DOT Shipping Name	UN3077, Environmentally Hazardous Substances, Solid, N.O.S. (Mercury, Fluorescent Lamps), 9, PG III
EPA Hazard Codes	None
Container	4' Bulb Box
Average pH	4.1 – 10.0 SU
Disposal Method	Disposal to Treatment, Storage and/or Disposal Facility (TSDF)
Hazard Label	Miscellaneous
Core ID	23 

5.3.14. Methanol and Water Waste

Methanol and water waste stream consist of water-soluble solvents, standards dissolved in methanol or acetone, HPLC and LC/MS/MS instrument waste, and spent methanol waste.

Water Soluble Solvents - Spent acetone, methanol, and acetonitrile may be stored at the point of generation in satellite accumulation containers. Satellite containers must be taken to the waste

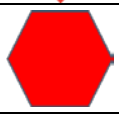
accumulation building and bulked in the drum labeled “Methanol and Water Waste” [Profile 335071].

Standards Dissolved in Methanol or Acetone - Standards dissolved in methanol or acetone may poured off into satellite containers and stored at the point of generation. Satellite containers must be taken to the waste accumulation area and bulked in the drum labeled “Methanol and Water Waste” [Profile 335071].

HPLC Instrument Waste - HPLC acetonitrile, methanol and water waste may be stored at the point of generation in satellite accumulation containers. Satellite waste containers must be taken to the waste accumulation building and bulked in the drum labeled “Methanol and Water Waste” [Profile 335071].

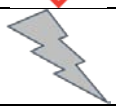
LC/MS/MS Instrument Waste – LC/MS/MS methanol and water waste may be stored at the point of generation in satellite accumulation containers. Satellite waste containers must be taken to the waste accumulation building and bulked in the drum labeled “Methanol and Water Waste” [Profile 335071].

Spent Methanol - Spent methanol generated during rinsing procedures may be stored at the point of generation in satellite containers. Satellite containers must be taken to the waste accumulation area and bulked in the container labeled “Methanol and Water Waste” [Profile 335071].

Methanol and Water Waste	
Profile Number	335071
DOT Shipping Name	UN1993, Waste Flammable Liquids, n.o.s. (Methanol, Acetone), 3, PG II, RQ (D001)
EPA Hazard Codes	D001, F003
Container	55 Gallon Metal Drum
Average pH	4.1 – 10.0 SU
Disposal Method	Disposal to Treatment, Storage and/or Disposal Facility (TSDF)
Hazard Label	Flammable 
Core ID	3 

5.3.15. Soils for Incineration

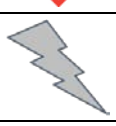
Soil samples received under the EPAs Contract Laboratory Program (CLP) must be incinerated. Samples are bulked in drum labeled “Soils for Incineration” [Profile 1000058495B]. Solids are removed from sample containers and emptied per the requirements set forth in section 5.8.2.

Soils for Incineration	
Profile Number	1000058495B
DOT Shipping Name	UN3077, Waste Environmentally Hazardous Substances, Solid, n.o.s. 9, PG III
EPA Hazard Codes	D006, D008
Container	55 Gallon Open Top Metal Drum
Average pH	4.1 – 10.0 SU
Disposal Method	Disposal to Treatment, Storage and/or Disposal Facility (TSDF)
Hazard Label	Miscellaneous 
Core ID	21 

5.3.16. Hazardous Soil Samples

Hazardous soils samples waste profile consists of extracted soils, spend sodium sulfate, and excess soil samples. Extracted soil residue and spent sodium sulfate used for sample drying are stored at the point of generation in a 55-gallon steel satellite drum labeled “Hazardous Soil Samples” [Profile CH162852]. The satellite drum must be transferred to the waste accumulation area whenever it becomes full.

Solid samples (soils/sludges) are disposed of as hazardous waste. Solid samples are disposed of in the drum labeled “Hazardous Soil Samples” [Profile CH162852]. Solids are removed from sample containers and emptied per the requirements set forth in section 5.8.2.

Hazardous Soil Samples	
Profile Number	CH162852
DOT Shipping Name	NA3077, Hazardous Waste, Solid, n.o.s. (Soil, Glass), 9, PG III
EPA Hazard Codes	D006, D008
Container	55G Open Top Metal Drum
Average pH	4.1 – 10.0 SU
Disposal Method	Disposal to Treatment, Storage and/or Disposal Facility (TSDF)
Hazard Label	Miscellaneous 
Core ID	21 

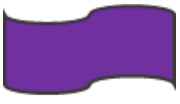
5.3.17. Waste Pyridine

Pyridine waste generated during cyanide by flow injection analysis may be accumulated at the point of generation in satellite containers labeled “Waste Pyridine” [Profile CH162856]. Satellite waste containers must be taken to the waste accumulation building whenever they become full.

Waste Pyridine	
Profile Number	CH162856
DOT Shipping Name	UN1282, Pyridine, 3, PG II
EPA Hazard Codes	D001, D002, D038, F002, F005
Container	5 Gallon Jerrican
Average pH	4.1 – 10.0 SU
Disposal Method	Disposal to Treatment, Storage and/or Disposal Facility (TSDF)
Hazard Label	Flammable 
Core ID	10 

5.3.18. Foreign Soil and/or Regulated Domestics in Methanol Vials

Foreign soil and/or regulated domestic soil in methanol vials (when applicable) is treated as hazardous waste and must be segregated from soil samples not from foreign or regulated domestic sites due to possible pest contamination from foreign sources. To mitigate this risk, foreign soil is treated as infectious waste. Due to the additional regulations applicable to all aspects of foreign soil analysis, characterization, and disposal, a separate policy has been written. For details on how to handle foreign soil, refer to Foreign Soil and/or Regulated Domestic Soil Samples SOP [HS SOP ME001J9].

Foreign Soil and/or Regulated Domestics in Methanol Vials	
Profile Number	Shealy07
DOT Shipping Name	UN3077, Waste Environmentally Hazardous substances, Solid, n.o.s. (Soil, methanol), 9, PG III, RQ (D001)
EPA Hazard Codes	D001, F003
Container	Poly close top drum
Average pH	4.1 – 10.0 SU
Disposal Method	Disposal to Treatment, Storage and/or Disposal Facility (TSDF)
Hazard Label	Miscellaneous 
Core ID	7 

5.3.19. Hazardous Aqueous (No Organics, low pH)

Hazardous aqueous (No Organics, low pH) waste stream consists of excess non-flammable hazardous aquesous samples, TCLP leachates, and nitrate-nitrite process wastes.

Non-Flammable Hazardous Aqueous Samples – Aqueous samples found to be hazardous during the course of analysis must be segregated and disposed of as a hazardous waste. If an aqueous sample is found to contain hazardous constituents at or above the limits listed in Table 1, during the course of analysis, the sample is flagged in the LIMS system as hazardous. When flagged hazardous samples are scanned out for disposal using the ICOC module in LIMS, the system will alert the user to dispose of the flagged samples as hazardous waste. Samples flagged as hazardous must be segregated and disposed of in the drum labeled "Hazardous Aqueous (No Organics, Low pH) [Profile Shealy 11]. Aqueous samples are removed from sample containers and emptied per the requirements set forth in section 5.8.2.

TCLP Leachates - Aqueous leachates generated during the Toxicity Characteristic Leaching Procedure (TCLP) may be stored at the point of generation in satellite accumulation containers. Satellite containers must be taken to the waste accumulation building and bulked in the drum labeled "Aqueous Hazardous Waste (No Organics, Low pH)" [Profile PACE-WCOL11].

Nitrate-Nitrite Process Wastes - Cadmium containing waste generated during nitrate-nitrite analysis may be stored at the point of generation in satellite containers. Satellite waste containers must be taken to the waste accumulation building and bulked into the drum labeled "Aqueous Hazardous Waste (No Organics, Low pH)" [PACE-WCOL11].

Hazardous Aqueous (No Organics, low pH)	
Profile Number	Shealy11
DOT Shipping Name	UN2922 Corrosive liquids, toxic, n.o.s. (Lead, sulfuric acid), 8, (6.1), PG II
EPA Hazard Codes	D002, D004, D006, D007, D008, D011
Container	Poly close top drum
Average pH	< 2 SU
Disposal Method	Disposal to Treatment, Storage and/or Disposal Facility (TSDF)
Hazard Label	Corrosive, Toxic 
Core ID	16 

5.3.20. Mercury Thermometers - Broken

Mercury thermometers – broken waste stream consists of broken mercury thermometers where the mercury droplets were treated with mercury tamer. Satellite waste containers must be taken to the waste accumulation building and bulked into the drum labeled "Mercury Thermometers – Broken" [Profile Shealy 13].

Mercury Thermometers – Broken	
Profile Number	Shealy13
DOT Shipping Name	UN2809 Waste Mercury, 8, (6.1), PG III
EPA Hazard Codes	D009
Container	5 Gallon Poly bucket
Average pH	4.1 – 10.0 SU
Disposal Method	Disposal to Treatment, Storage and/or Disposal Facility (TSDF)
Hazard Label	Corrosive, Toxic 
Core ID	None

5.3.21. Samples Contaminated with PCBs

Samples analyzed for PCBs where levels meet or exceed 50 ppm should be returned to the client for disposal. When a sample is analyzed for PCBs where levels exceed 50 ppm, the sample is flagged in the LIMS system and a PCB Hit Notification is sent to the waste manager and project manager. Additionally, when flagged samples are scanned out for disposal using the ICOC module in LIMS, the LIMS system will alert the user to return the sample to the client for disposal. The project manager will schedule the sample return with the client and notify sample receiving personnel regarding the return details.

All containers (sample containers and extracts) associated with the sample must be located and returned to the client following appropriate chain of custody procedures.

Under certain circumstances, the client may not be able to accept a returned sample. Samples that cannot be returned to the client must be scanned out to a specific drum using the LIMS ICOC. Samples are disposed of in the drum labeled “PCB Contaminated Samples” [Profile PTA826167 (PCB<500 ppm)] or [Profile PTA208995 (PCB>500ppm)]. Samples are not removed from their sample container. All samples analyzed for PCBs where levels exceed 10,000 ppm must be returned to the client for disposal.

Sample containers must be placed in a designated temporary sample storage area while awaiting return to the client or transfer to accumulation drum. The temporary sample storage area must be segregated from other laboratory wastes and samples. Samples may not be stored in the temporary storage area for more than 30 days. Each sample container must be labeled with a PCB label prior to placement into the temporary storage area.

PCBs > 500 ppm	
Profile Number	PTA208995
DOT Shipping Name	UN1993, Waste Flammable Liquids, n.o.s., 3, II (Hexane, Polychlorinated Biphenyls > 500 ppm) ERG 128
EPA Hazard Codes	D001
Container	Drum
Average pH	5 – 9 SU
Disposal Method	Disposal to Treatment, Storage and/or Disposal Facility (TSDF)
Hazard Label	Flammable 
Core ID	None

PCBs < 500 ppm	
Profile Number	PTA826167
DOT Shipping Name	UN1993, Waste Flammable Liquids, n.o.s., 3, II (Hexane, Polychlorinated Biphenyls > 500 ppm) ERG 128
EPA Hazard Codes	D001
Container	Drum
Average pH	5 – 9 SU
Disposal Method	Disposal to Treatment, Storage and/or Disposal Facility (TSDF)
Hazard Label	Flammable 
Core ID	None

5.3.22. Lab-pack

At times there are items that do not fit into the hazardous waste streams. These items are collected in lab-pack bins and are sent off-site with the waste hauler periodically. Items that could be considered lab-pack items: pure discarded chemicals, empty RSK cylinders, and empty propane cylinders. The waste manger, or designee, will collect lab-pack bins periodically to perform inventory and provide a list to the waste hauler. Empty gas cylinders are not refillable.

5.3.23. Samples Inoculated with Growth Media

Microbiology samples inoculated in media and all debris produced during analysis are stored bio-hazardous waste containers located in the Inorganic Non-Metals/Wet Chem. Department. Wastes must be placed in bio-hazard autoclave bags prior to storage in waste containers. Each week or when containers are full, whichever comes first; the materials must be autoclaved for 30 minutes at 121˚C / 17 psi. After materials are autoclaved, they may be disposed of as non-hazardous waste.

5.4. Declared Hazardous Wastes

Some waste can become complicated when attempting to classify as non-hazardous or hazardous due to the list of hazardous constituents contained in sections 40 CFR 261.30-261.35 including a majority of analytes of interest routinely analyzed in Pace laboratories. Definitions have been established for each of the F, K, P, and U lists covering hazardous waste originating from non-specific sources, specific sources and discarded commercial chemical products, off-specification species, container residues, and spill residues. The application of listed hazardous wastes and substances is intended for manufacturing processes involving pure products, by-products, wastes generated as part of the production process and cleanup of materials contaminated from a spill of the listed commercial chemical product or manufacturing chemical intermediate. See Attachment I for common F-listed wastes.

Hazardous waste classification of unconsumed samples by listed hazardous waste criteria is not commonly applied in laboratory operations. Examples of sample types which would be identified as hazardous wastes include the following:

- Samples containing 5% or more (by volume) of halogenated and non-halogenated “spent solvents:” (e.g., drum sample with > 10% TCE);
- Pure product and two phase solution samples containing a listed chemical product or manufacturing intermediate (e.g., drum sample) or an unknown sample that appears to be oil or oily material;
- Samples representing any residue or contaminated soil, water or other debris resulting from the cleanup of a spill into or on any land or water of any commercial chemical product or manufacturing chemical intermediate having a generic name listed in section 261.33, or any residue or contaminated soil, water or other debris resulting from the cleanup of a spill, into or on any land or water, of any off-specification chemical product and manufacturing chemical intermediate which, if it met specifications, would have the generic name listed in section 261.33.

5.5. Sample Disposal

For the samples listed in 5.4, disposal can be achieved by individually lab packing them or combining with other compatible hazardous wastes. However, it is important to note that in order for the laboratory to ascertain that the samples were derived from a specific listed source or from a spill of a listed chemical, they must be so informed by the industrial concern or lead agency (e.g., EPA, state regulators) submitting the sample for analysis. If a water or soil sample contains a listed hazardous waste substance whose origin is unknown or uncertain to the lead agency, then that sample is not classified as a listed hazardous waste. Rather in this case, determination of a hazardous waste classification can only be obtained by the waste exhibiting a characteristic of hazardous waste (e.g., ignitability, corrosivity, reactivity).

Due to the fact that the majority of samples analyzed by Pace do not meet the well-defined criteria for identifying “listed” hazardous waste, disposal classification of unconsumed samples will be based upon characteristics of hazardous waste:

- Non-Hazardous - Analysis results indicate an absence of contaminants; unless contaminants listed under the hazardous disposal categories are parts of the requested sample analysis.

- Hazardous – Analysis results indicate presence of contaminants (Attachment III) or sample analysis requires hazardous materials and contaminants. Samples in this category are segregated from others and disposed of as hazardous according to laboratory procedures.

- ***PCB Waste*** - Generated exclusively by samples contaminated with greater than trace levels of polychlorinated biphenyls (> 50ppm). Samples containing 50ppm (total) or higher of PCBs must be segregated and disposed of as PCB waste.

- ***Waste Oil/Paint*** - Samples which are predominantly of an oil matrix (e.g., highly viscous organic liquid) or paint (solvent and pigment blend) are segregated and disposed in a separate container. Though these samples are defined as nonhazardous, oil samples are a special case and are never disposed as nonhazardous. Note: Bottle caps and liners do not typically contain sample residuals and can be disposed of directly through the non-hazardous building refuse.

USDA-APHIS-PPQ Regulated Soils (Regulated Soils) are a special case of sample strictly controlled under quarantine regulations 7 CFR 330, 331, and 318-330 because they can readily provide a pathway for a variety of dangerous organisms throughout the United States. The movement of soil into the United States from foreign sources and from certain regulated areas within the continental U.S. is restricted unless permitted by APHIS under specific conditions and safeguards.

Any laboratory that handles Regulated Soils must have an approved Compliance Agreement from USDA-APHIS-PPQ, and labs that handle foreign soils must have an approved Permit to Receive Soil. See the local version of Regulated Soil Handling COR-HSE-0002 for all information regarding the handling of these materials. The local document is Foreign Soil and Regulated Domestic Soil: Sample Receiving, Laboratory Handling, Disposal, and Documentation [HS SOP ME001J9].

5.6. Consolidation of Wastes

Consolidation of wastes from the laboratory proceeds via two distinct routes covering either laboratory-generated hazardous wastes or excess unconsumed samples.

5.6.1. Laboratory Accumulation and Satellite Accumulation Areas/Containers

Waste materials from routine lab procedures are collected in containers of appropriate construction, placed in convenient locations at the point of generation. Under RCRA guidelines, these are defined as satellite containers.

The amount of hazardous waste stored in the laboratory at the individual satellite areas cannot exceed 55 gallons (liquid) or 550 lbs. (solid) per waste stream, for non-acute hazardous waste.

Satellite waste containers must be labeled in accordance with all regulations, including:

- Designation of the contents to be hazardous waste with the words “Hazardous Waste” clearly legible.

- The waste stream description (e.g., acid waste).

- A hazard communication label (e.g., corrosive).

- A reference to profile number (e.g. 259534).

The satellite containers must be maintained such that evolution of chemical vapors is precluded. This requires that the container be closed at all times, except when adding or emptying hazardous waste to and from the container.

The most critical point in the waste handling system is when a person (e.g., analyst, technician) places a waste material into a satellite container. Here, the characteristics or listing of the waste and the waste stream must both be known to match. For this reason, only material from approved procedures should be placed in the compatible satellite containers. All materials from experimental procedures, unknown or out of the ordinary sources, or from spill cleanups must be characterized and described to the Hazardous Waste Coordinator, who determines the proper method of disposal.

Full satellite containers must be transferred to the proper accumulation drum within 3 calendar days. Lab collection containers must not be filled to the top of the opening. Space must be left to prevent splashing of hazardous material when containers are emptied and to allow for expansion and contraction within the drum during transport.

Satellite containers for liquid hazardous waste must have secondary containment made of material that could successfully contain the entire satellite container's contents.

5.6.2.Unconsumed Sample Disposal

Client samples are stored on-site for a defined period of time after the final analytical report is generated and prior to sample disposal. The purpose of sample storage is to provide the client time to review the analytical report and determine if the samples require additional testing or need to be returned to the client. Samples are not considered a waste during this time according to 40 CFR 261.4(d)(1)(vi).

The default sample storage time is at a minimum of four weeks from receipt, unless otherwise specified by the client. The determination of what samples are ready to be disposed of is based on a color code system. Using a color code chart, the custodian removes and disposes of the samples received approximately during the sixth week prior to the current week (soils are dumped during the sixth week or afterwards). Exceptions to this include samples whose holding times are short and those that are consumed during analysis.

During sample storage, the process and sample status must be obvious to employees, customers and auditors. This transparency is imperative to ensure samples are considered active test specimens to be retained until they are categorized as a waste for disposal.

Samples which cannot be returned to the client for disposal are characterized according to section 5.5. Samples are characterized by one of three methods:

- Analytical results are evaluated against characterization criteria established for the sample waste stream. The samples which exhibit waste characteristics as previously outlined are segregated and denoted per laboratory/facility policies. The completed form is then forwarded to the Hazardous Waste Coordinator, who in turn uses the information to coordinate removal of unconsumed samples from active sample storage by the log-in staff. OR:

- Samples are scanned out of LIMS 5 as RCRA nonhazardous to be disposed as the waste stream is normally handled unless the sample tested over RCRA limits, in which the LIMS will prompt the employee that the sample is destined for Hazardous disposal, and is segregated from the nonhazardous samples. OR:

- Samples of a certain type are all “declared” to be hazardous, and all are placed into an accumulation drum with all required RCRA labeling for that waste stream.

- All samples containers that contain client information, including labels placed on bottles by Sample Receiving containing sample identification numbers and client identification numbers, and must be blacked out in a permanent, immutable manner before being disposed in any waste stream. These procedures are outlined in the Destruction and Disposal of Empty Sample Containers SOP [HS SOP ME001H7].

Samples are pulled from storage and disposed according to Sample Receiving SOP [AD SOP ME0013H].

5.7. Unconsumed Soil Samples

Unconsumed soil samples are kept in storage until the samples are ready to be disposed. Depending on the sample type, and test results will depend on which waste stream the sample is scanned to in the ICOC system in LIMS 5. Refer to the Sample Receiving SOP [AD SOP ME0013H] for specifics.

Unconsumed soil samples can be disposed in one of the following profile types:

- Soils Samples in Methanol Vials [Profile 270048]

- Soil Samples in Water Vials [Profile 318384]

- Soil Samples [Profile CH162852]

- EPA CLP Soils [Profile 1000058495B]

- Flammable Soils [Profile 311180]

- Foreign and/or Domestic Regulated Soils [Profile 323354 or Shealy07]

- Samples Contaminated with PCBs [Profile PTA826167 (PCB < 500 ppm) or PTA208995 (PCB > 500 ppm)]

Refer to section 5.3 for specific profile information.

5.8. Elementary Neutralization

Dilute corrosive solutions (e.g., preserved metals samples) which do not exhibit any hazardous characteristics other than being corrosive, may be neutralized. Elementary neutralization is exempt

from RCRA permitting requirements for on-site hazardous waste treatment. While exempt under RCRA guidelines, before utilizing this practice to reduce off-site treatment or disposal of wastes, local pretreatment and discharge standards must be met for publicly owned treatment works (POTW).

The discharges listed below are prohibited under the National Pretreatment Standards and Prohibited Discharge Standards:

- Pollutants causing fire or explosion (waste with a flashpoint < 60 °C);
- Corrosive wastes with pH less than or equal to 2 or greater than or equal to 12.5;
- Solid or viscous pollutants that could potentially block the system;
- Oxygen-demanding pollutants;
- Wastes which generate toxic gases.

Non-hazardous wastes and wastes hazardous based only on the characteristic of corrosivity (D002) are bulked into elementary neutralization units located in CAA2. Wastes are neutralized by the addition of either 10N sodium hydroxide or a diluted sulfuric acid solution (1:1) prior to discharge to the City of Cayce WWTP (POTW).

Wastes must be neutralized to a pH between 6.0 SU and 9.0 SU prior to discharge to the POTW. The initial pH before adjustment and the final pH after adjustment must be recorded in the Elementary Neutralization Log [HS Form ME0014I], which is accessible electronically via the computer station located in the ENU area. pH determinations must be made using a calibrated pH meter.

Once the waste has been treated for corrosivity, it loses its hazardous characteristic (becomes deactivated), and can be disposed of as non-hazardous. Wastes are pumped into the lime-pit via a manual siphon or motorized pump for conveyance to the WWTP.

NOTE: Elementary neutralization can only be performed without obtaining a permit if it is performed in a designated elementary neutralization unit and only if the waste has been determined to only be hazardous because it exhibits the characteristic of corrosivity, D002. All other wastes that have been determined to be hazardous based on other hazardous characteristics or because it is listed on one of the EPA tables, must be disposed of into the appropriate hazardous waste stream.

NOTE: If an analyst is ever unsure of the proper disposal techniques for a specific waste, the waste manager must be notified immediately. The waste manager will make a hazardous waste determination and dispose of the waste accordingly.

5.8.1. Discharge to POTW (Wastewater)

Wastewaters introduced into the sewer system in laboratory areas are initially sent (via sewer pipes) to a lime-pit located on-site prior to final disposition to the POTW.

The lime-pit is a leak proof reservoir filled with limestone pebbles. The influent pipe extends down into the limestone to ensure incoming wastes make intimate contact with the limestone. Additionally, the effluent flow from the lime-pit to the WWTP is very slow, conducive to intimate contact. Slightly acidic water will be neutralized by the calcium carbonate; however, it is important to note that pH adjustment is a slow process. Large amounts of highly acidic waste may not be neutralized before being permanently discharged. In addition, the limestone can only neutralize acidic water. Caustic wastewaters with high pH will not be neutralized by the limestone.

To ensure the limestone pit has not reached a level below 5 SU or above 10 SU, a pH test is performed regularly by field technicians using a pH meter. Pit pH values are documented in The Lime Pit pH Log [HS Form ME0016A], which is accessible electronically via the computer station located in the ENU area. If the reading is out of range, the CEO must be notified immediately.

The daily pH testing results are used to determine when the lime-pit must be cleaned and the limestone replaced. If pH result trending suggests that the limestone is becoming ineffective (i.e. several readings near the lower limit), the CEO will arrange for limestone replacement. All limestone, and sludge accumulated at the base of the tank from possible heavy metal precipitation, must be disposed of as a hazardous waste unless determined to be non-hazardous based on analysis.

Based on PACE-WCOL's wastewater composition and the volume of wastewater being discharged into the head-works of the WWTP (less than 1%), PACE-WCOL is not subject to categorical pretreatment standards; however, PACE-WCOL must comply with the general prohibitions listed in the City of Cayce Sewer Ordinances; no wastes shall be introduced to the sewer system that:

- Cause a pass through or interference
- Any pollutant or wastewater to which water is added for the purpose of diluting wastes that would otherwise exceed applicable maximum concentration limitations for any wastewater constituents
- Any substance which will cause the POTW to violate its NPDES permit or the receiving water quality standards
- Pollutants which create a fire hazard for explosive hazard
- Wastewater having a pH less than 5.0 SU or more than 10.0 SU
- Solid or viscous substances which will cause obstruction of the flow into the POTW
- Pollutants released in a discharge at a flow rate which will cause interference with the POTW

5.8.2.Empty Container Disposal

Sample and reagent containers may be discarded into a non-hazardous waste stream provided they are empty, which is specifically defined by SC HWMR.61-79.CFR 261.7. To be considered empty by RCRA standards the following conditions must be met:

Personnel must remove as much material from the container as possible using normal methods such as pouring, pumping or scraping, AND There must be no more than 1 inch of residue remaining on the bottom of the container,

OR

There is no more than 0.3% by weight of the total capacity remaining in a container less than 110 gallons.

There are two major exceptions to the rules listed above:

Aerosol bottles are considered empty when the contents reach atmospheric pressure and the contents can longer be expelled from the can.

Acutely hazardous waste containers, such as P listed waste, must be triple rinsed with an adequate rinsate or solvent known to dissolve the contaminant before it can be discarded (SC HWMR.61-79.261.7(b)(3)).

In addition to the RCRA standards for emptying containers, all labels on sample containers, that contain client information, including labels placed on bottles by Sample Receiving containing sample identification numbers and client identification numbers, and must be blacked out in a permanent, immutable manner before being disposed in any waste stream. These procedures are outlined in the Destruction and Disposal of Empty Sample Containers SOP [HS SOP ME001H7].

5.9. Transferring Satellite Waste to the Waste Storage/Accumulation Area

All transfers of satellite waste to waste drums must be made by the Hazardous Waste Coordinator or designated, trained personnel. When a satellite waste container is full, the Hazardous Waste Coordinator, or designee must be notified. Regular disposal events may be scheduled to dispose satellite waste on a continuous basis. When transferring hazardous waste:

- Find the correct accumulation container by referring to the Hazardous Waste placard, hazard label, and internal symbol/color combination. If an employee is not certain the waste profile from the satellite and accumulation container are the same, they must stop and ask the Hazardous Waste Coordinator for more information. Mixing solvents that are not compatible may result in a reaction or a more hazardous waste mixture.
- Ensure there is enough capacity in the drum to hold all the content that will be dispensed.
- Check to make sure there is a ground connection before opening a solvent waste drum.
- Open and slowly pour the contents of the satellite container into the proper waste drum using an appropriate solvent resistant funnel.
- Close the container, by securing the lid on the funnel or drum, or replace the cap on the bunghole and carefully screw the cap on but do not tighten the cap.

5.10. Waste Storage Container Requirements

Drums in the hazardous waste storage area are labeled consistent with both DOT and EPA regulations concerning hazardous materials and wastes (see Attachment IV for example of label).

Closure instructions must be available for all containers used to transport hazardous materials. If a container in the accumulation area is the same one the waste will be shipped away in, the Waste Coordinator must obtain the closure instructions from the provider of the containers.

Labels must be easily visible and legible (e.g., a drum must not be labeled and then placed in such a way that the label cannot be seen).

The Accumulation Start Date must be recorded on the drum. The date should reflect the first time waste was added to the drum and not the date when the waste was generated in the laboratory.

- Once a waste is removed from the point of generation to a hazardous waste staging area, the clock is started for storage time prior to disposal.

- Drums must be picked up by TSDF for disposal before accumulation time exceeds RCRA requirement for lab's generator status (see Table 5.1).

The hazardous waste staging room must be arranged in such a fashion to assure direct access pathways in the event of foreseeable emergency and for safe waste transfer. A minimum aisle space of three feet must be maintained at all times to access hazardous waste containers.

All hazardous waste drums and containers must be securely closed when not in use. All volatile and flammable hazardous waste liquid containers must be securely grounded at all times. Drums containing these liquids should also be manipulated with non-sparking tools and fitted with a drum venting bung, to assure that excess pressure build-ups are safely released.

All liquid waste stream containers must be provided with secondary containment devices. Such containment devices must be made of materials compatible with each waste, and they must be free of leaks.

- The waste storage room may act as secondary containment as long as the room has been constructed to safely and effectively contain a hazardous waste spill.

- Secondary containers must exceed the total volume of the largest container stored in each containment device for indoor storage.

Compatibility of wastes must be considered in arranging storage areas. For example, acid waste should never be stored adjacent to basic waste, particularly cyanide wastes. Further examples are outlined in 40 CFR 264, Appendix V.

The hazardous waste staging area is controlled so unauthorized personnel are not able to access the room or contents.

The maximum volume of acutely hazardous waste (e.g., P-listed wastes) that can be accumulated in the laboratory is one quart. The volumetric measurement of one quart is based upon container size in which the waste is stored and not the actual amount (volume) of waste present. An example of how this one quart limit can inadvertently be exceeded involves the disposal of a neat standard of 2,4-dinitrophenol into a one gallon bottle. While the neat standard itself may only constitute 1-2mL, the volume as defined under RCRA would be one gallon, thus the laboratory would be out of compliance.

5.11. Waste Inspections

The central accumulation staging room must have a documented inspection weekly. Satellite Accumulation Areas must have documented monthly. The inspections should ensure all regulations are obeyed; see sections 5.10 and 5.6.1 for accumulation storage and satellite rules.

A record of the inspections must be kept for at least 3 years in an inspection log or summary

Records must be maintained for at least three years from the date of inspection. At a minimum, the records must indicate:

- The date of the inspection;

- The name and signature of the inspector

- A notation of the observations made (can be in a check-off format, e.g., fire extinguisher: charged X_ requires recharging __);

- The date and nature of any repairs or other remedial actions.

5.12. Waste Documentation and Reporting

The following hazardous waste records must be maintained a minimum of five years and should be retained indefinitely:

- Sample Reports;
- Sample disposal information and waste records;
- Analytical records relating to sample waste stream profiling and characterization;
- Labpack inventory logs;
- Biennial Reports, Exception Reports, or other reports filed for compliance reasons;
- Records related to unresolved enforcement action must be retained indefinitely until such a time that the matter is resolved;
- Facility Certificates of Destruction or Recycling.

All drums containing hazardous waste are recorded in a LIMS 5. The information contained in this log is useful when filling out EPA biennial reports and for retaining an accurate description of how much waste has been accumulated. The following information is entered into the database;

- Drum number or ICOC number;
- Waste profile description;
- Waste profile number;
- Date filling the drum was started;
- Select if the drum contains foreign soil
- Date the drum was sent off-site;

A Waste Manifest is the documentation form that must accompany all shipments of hazardous waste while in transit.

A Hazardous Waste Manifest Cover Sheet [HS Form ME0043N] will be utilized to ensure waste transfer from generator to TSDF fulfills all legal requirements.

The manifest is be signed and dated by a DOT trained Pace employee responsible for the shipment and the transporter. The transporter will leave 2-3 of these “two-signature page” copies of the manifest.

Within 35 days you will receive a three-signature page (generator, transporter, facility) showing the waste reached its intended destination. The waste coordinator is responsible to ensure it is filed in purge documents.

If you do not receive the three signature page within 35 days of shipment, call the facility to find out why you have not received it. If you do not receive the three-signature page within 45 days you must file an exception report with South Carolina Bureau of Land and Waste Management.

All manifests must be kept for a minimum of three years.

A copy of land disposal notification form will also be signed and left by the transporter. File all purge documents in corresponding folder. Once invoice is received with certificate of waste management, must be kept on file with manifest documents and/or electronically.

Annual Generation Reports are required to be filed with Agency that requires Annual Report:

Large Quantity Generators must file a biennial report with the local state or EPA Region. SC DHEC Bureau of Land and Waste Management requires a quarterly report to submitted by the end of the following month. The report is submitted on SC DHEC Form D-1962 and D-1963 as well as form D-2701 when needed. The report must include the following items:

- Laboratory name, address, and EPA identification number;
- Quarter and year;
- State assigned waste index number;
- The name, address and EPA identification number for each TSDF used during the calendar year;
- A description, EPA hazardous waste number (e.g., F002) and quantity of each waste shipped off-site for treatment and disposal. This must be listed by the TSDF used;
- The signed certification statement on D-1962 Form of authorized representative.

5.13. Equipment and Supplies

The following equipment is mandatory under RCRA guidelines unless otherwise denoted. Periodic review (not to exceed monthly) of availability of equipment and supplies below should be conducted to maintain an adequate and viable supply.

***Chemical Spill Control Neutralizers*:** The waste room stores three types of bulk dry spill neutralizers: solvent, acid and base. They may be utilized by placing the dry neutralizer onto a liquid chemical spill. Neutralization is indicated by a prevalent color change.

Communication Device: Required for emergency notification of spill, fire, etc.

Drums: Common types of waste drums used for storing and shipping hazardous wastes are polyethylene, steel-polyethylene lined, and steel. Sizes are typically 5 gal, 15 gal, 30 gal, and 55 gal. Drums used for liquids typically are closed top with an opening to pour the solvent through a funnel, while drums used for solids or lab packs are open-top. The UN rating for all containers must be suitable if the waste is to be transported under DOT regulations.

Emergency Drench Shower: Shower should deliver water approximately twenty gallons per minute with a non-interruptible flow. It may be turned on by pulling the shower handle down. It may be turned off by pushing the handle back to the 'off' position.

Emergency Lighting (as needed): The waste room is outfitted with emergency lighting that goes on if power fails.

Exit Signs (as needed): Exit signs are provided on all waste room doors. These signs are self-illuminating.

Fire Alarm Pull Station/Evacuation Alarm: A fire alarm pull station must be in close proximity to the hazardous waste room. The alarm may be activated by pulling the switch. Other alarm systems may be utilized as long as all personnel are trained on the procedures and the process can effectively notify facility employees of an emergency.

Fire Extinguisher: An extinguisher with a rating appropriate to the waste being stored in the area must be in close proximity to the hazardous waste room.

Labels: A multitude of labels are provided to ensure compliant labeling. They may be purchased or prepared manually.

Liquid Chemical Neutralizers: Liquid chemical neutralizers (base and acid) may be used to neutralize a contained hazardous liquid. This may be done by slowly adding the neutralizer to the liquid starting at the outside of the spill and circling inward.

Spill Control Pads: Spill pads are used to soak up hazardous liquids. They do not neutralize spills. They are especially effective for cleaning up oily materials. Various pads are available for aqueous and petroleum based liquids.

Spill Control Pillows: Spill pillows may be used to soak up large amounts of liquid chemical spills. No neutralization occurs.

Spill Dikes: vary depending on the size and type of room: Their purpose is to encircle a spill, barring the spread of a hazardous chemical. They will also absorb liquids, but do not neutralize spills.

6.0 Responsibilities

Pace employees that perform any part of this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

7.0 Attachments

Attachment I: RCRA Requirements for Labs as a Function of Generator Status.

Attachment II: Hazard Codes for Common F-List Wastes (solvents).

Attachment III: TCLP Contaminant List with Concentration Limits.

Attachment IV: Hazardous Waste Label for Accumulation Drum (example).

Attachment V: Satellite Container Inspection Form (example).

Attachment VI: Weekly Waste Inspection Form (example).

Attachment VII: Hazardous Waste Manifest Cover Sheet.

8.0 References

Pace Chemical Hygiene/Safety Manual-most current version.

Pace Quality Assurance Manual- most current version.

National Environmental Laboratory Accreditation Conference (NELAC) Standard- most current version.

The NELAC Institute (TNI) Standard- most current version applicable to each lab.

Department of Defense (DoD) Quality Systems Manual- most current version.

9.0 Revision history

Revision #	Revision Date	Section Modified	Modification	Reason for Modification
-09	**04/29/2021**	All	Update to Pace Format	Acquisition
		Title Page	Update Personnel for QM and EHS to Kelly Nance and Kristina Bouknight	Personnel change

Attachment I: RCRA Requirements for Labs as a Function of Generator Status

Requirement (40CFR)	CESQG	SQG	LQG
Waste Determination (262.11)	Applicable	Applicable	Applicable
Generation Rate Limits (261.5 and 262.34)	<100 kg/mo	100-1,000 kg/mo	1,000 kg/mo or greater
Accumulation Quantity Limit w/o Permit (261.5 and 262.34)	Not to exceed 1,000 kg at any time. Not to exceed 1 kg acute at any time	not to exceed 6,000 kg at any time	No limit
Accumulation Time (261.5 and 262.34)	No limit	180 days or 270 if waste is to be transported over 200 miles.	90 days
EPA ID Number (262.12)	Not required***; possible state requirement	Required	Required
Mark Containers with Start Date (262.34)	Not applicable	Applicable	Applicable
Mark Containers “Hazardous Waste” (262.34(a))	Not applicable	Applicable	Applicable
Air Emission Standards 40 CFR 265 Subpart CC	Not applicable	Not applicable	Applicable
Satellite Accumulation (262.34(c))	Not applicable	Applicable	Applicable
Use Manifests (262, Subpart B)	Not required; possible state requirement	Required	Required
Exception Reporting (262.42)	Not required	Required after 60 days. No TSDF notification requirement.	Required after 45 days. Notification of TSDF within 35 days.
Biennial Report (262.41)	Not required	Not required; possible state requirement	Required
Contingency Plan (265, Subpart D)	Not required, but OSHA (29 CFR 1910.38) requires emergency planning	Basic planning required in accordance with the standards in 262.34(d)(4) and (5) and 265, Subpart C as well as OSHA regulations	Full written plan in accordance with 265 Subpart D, is required by 262.34(a)(4) and OSHA regulations
RCRA Personnel Training (262.34 and 265.16)	Not required, but recommended	Basic training required by 262.34(d)(5)(iii)	Full compliance with the training requirements in 265.16 is required by 262.34(a)(4)
Storage Requirements (without permit) (262.34 and 265)	None, but OSHA regulations under 29 CFR 1910, Subparts H and N, apply, particularly 29 CFR 1910.106	Compliance with technical standards in Part 265, Subparts I and J; for containers and tanks is required by 262.34(d)(2) and (3) and OSHA regulations	Compliance with technical standards in Part 265, Subparts I, J, W, and DD, is required by 262.34(a)(1) and OSHA regulations

Requirement (40CFR)	CESQG	SQG	LQG
Recordkeeping Requirements (262.40)	Waste determinations and generation log required (notification of regulated waste activity, training records, manifests, and land disposal restriction notifications recommended)	Notification of regulated waste activity, waste determinations, generation log, manifests, land disposal restriction notifications, exception reports, and correspondence with local emergency responders (written contingency plan, weekly container inspection & periodic equipment maintenance logs, and RCRA training records recommended)	Notification of regulated waste activity, waste determinations, generation log, manifests, land disposal restriction notifications, exception reports, biennial reports, correspondence with local emergency responders, RCRA training records, and written contingency plan required (weekly container inspection is required & periodic equipment maintenance logs is recommended)
Waste “Designated Facility”	State-approved or RCRA permitted facility or legitimate recycler	RCRA-permitted facility or legitimate recycler	RCRA-permitted facility or legitimate recycler
Land Disposal Restrictions (268.7)	Possible state requirement	Applicable	Applicable

Attachment II: Common F-Listed Solvents

Waste Name	Hazardous Waste Code(s)		Waste Name	Hazardous Waste Code(s)
Acetone	F003		Methylene Chloride	F002
Benzene	F005		Methyl ethyl ketone (MEK)	F005
iso-Butanol	F005		Methyl isobutyl ketone	F003
n-Butyl alcohol	F003		Nitrobenzene	F004
Carbon Disulfide	F005		2-Nitropropane	F005
Carbon Tetrachloride	F001		Orthodichlorobenzene	F002
Chlorobenzene	F002		Pyridine	F005
Chlorinated fluorocarbons (CFC)s	F001		Tetrachloroethylene	F002
Cresols	F004		Toluene	F005
Cresylic acid	F004		1,1,1-Trichloroethane	F002
Cyclohexanone	F003		1,1,2-Trichloeoethane	F002
2-Ethoxyethanol	F005		1,1,2-Trichloro-1,2,2-trifluoroethane	F002
Ethyl acetate	F003		Trichloroethylene	F002
Ethyl benzene	F003		Trichloroflourormethane	F002
Ethyl ether	F003		Xylene	F003
Methanol	F003			

Attachment III: TCLP Contaminant List

Waste Code	Contaminant	Conc (mg/L)
D004	Arsenic	5.0
D005	Barium	100.0
D006	Cadmium	1.0
D007	Chromium	5.0
D008	Lead	5.0
D009	Mercury	0.2
D010	Selenium	1.0
D011	Silver	5.0
D012	Endrin	0.02
D013	Lindane	0.4
D014	Methoxychlor	10.0
D015	Toxaphene	0.5
D016	2,4-D	10.0
D017	2,4,5-TP Silvex	1.0
D018	Benzene	0.5
D019	Carbon Tetrachloride	0.5
D020	Chlordane	0.03
D021	Chlorobenzene	100.0
D022	Chloroform	6.0
D023	o-Cresol	200.0
D024	m-Cresol	200.0
D025	p-Cresol	200.0
D026	Cresol	200.0
D027	1,4-Dichlorobenzene	7.5
D028	1,2-Dichloroethane	0.5
D029	1,1-Dichloroethylene	0.7
D030	2,4-Dinitrotoluene	0.13
D031	Heptachlor	0.008
D032	Hexachlorobenzene	0.13
D033	Hexachlorobutadiene	0.5
D034	Hexachloroethane	3.0
D035	Methyl ethyl ketone	200.0
D036	Nitrobenzene	2.0
D037	Pentachlorophenol	100.0
D038	Pyridine	5.0
D039	Tetrachloroethylene	0.7
D040	Trichlorethylene	0.5
D041	2,4,5-Trichlorophenol	400.0
D042	2,4,6-Trichlorophenol	2.0
D043	Vinyl Chloride	0.2

PROCESS STANDARD OPERATING PROCEDURE
TITLE: Hazardous and Non-Hazardous Laboratory Waste Management Plan
ISSUER: Pace ENV - Local Quality - WCOL

Attachment IV: Hazardous Waste Label for Accumulation Drum (Example)

PROCESS STANDARD OPERATING PROCEDURE
TITLE: Hazardous and Non-Hazardous Laboratory Waste Management Plan
ISSUER: Pace ENV - Local Quality - WCOL

Attachment V: Satellite Container Inspection Form (Example)

Document Name: Monthly Satellite Accumulation Inspection
Document No.: ME003LL-01
Document Revised: February 14, 2020
Page 1 of 3
Issuing Authority: Pace WCOL Quality

Monthly Satellite Accumulation Inspection

Department	Container ID	1* Clearly Labeled?	2* Secondary Containment?	3* Closed When Not In Use?
		If any of the below fields are "NO", use the comments section to denote how container brought back into compliance.		
Volatiles	VOA1	✓ YES / NO	✓ YES / NO	✓ YES / NO
Volatiles	VOA2	✓ YES / NO	✓ YES / NO	✓ YES / NO
Volatiles	VOA3	✓ YES / NO	✓ YES / NO	✓ YES / NO
Volatiles	VOA4	✓ YES / NO	✓ YES / NO	✓ YES / NO
Volatiles	VOA5	✓ YES / NO	✓ YES / NO	✓ YES / NO
Volatiles	VOA6	✓ YES / NO	✓ YES / NO	✓ YES / NO
Volatiles	VOA7	✓ YES / NO	✓ YES / NO	✓ YES / NO
Volatiles	VOA8	✓ YES / NO	✓ YES / NO	✓ YES / NO
Volatiles	VOA9	✓ YES / NO	✓ YES / NO	✓ YES / NO
Volatiles	VOA10	✓ YES / NO	✓ YES / NO	✓ YES / NO
Volatiles	VOA11	✓ YES / NO	✓ YES / NO	✓ YES / NO
Volatiles	VOA12	✓ YES / NO	✓ YES / NO	✓ YES / NO
Volatiles	VOA13	✓ YES / NO	✓ YES / NO	✓ YES / NO
PFAS	PFAS1	✓ YES / NO	✓ YES / NO	✓ YES / NO
PFAS	PFAS2	✓ YES / NO	✓ YES / NO	✓ YES / NO
PFAS	PFAS3	✓ YES / NO	✓ YES / NO	✓ YES / NO
PFAS	PFAS4	✓ YES / NO	✓ YES / NO	✓ YES / NO
PFAS	PFAS5	✓ YES / NO	✓ YES / NO	✓ YES / NO
PFAS	PFAS6	✓ YES / NO	✓ YES / NO	✓ YES / NO
PFAS	PFAS7	✓ YES / NO	✓ YES / NO	✓ YES / NO
PFAS	PFAS8	✓ YES / NO	✓ YES / NO	✓ YES / NO
Sample Receiving	SR1	✓ YES / NO	✓ YES / NO	✓ YES / NO
Sample Receiving	SR2	✓ YES / NO	✓ YES / NO	✓ YES / NO
Sample Receiving	SR3	✓ YES / NO	✓ YES / NO	✓ YES / NO
Semivolatiles	SVOA1	✓ YES / NO	✓ YES / NO	✓ YES / NO
Semivolatiles	SVOA2	✓ YES / NO	✓ YES / NO	✓ YES / NO
Metals	IM1	✓ YES / NO	✓ YES / NO	✓ YES / NO
Metals	IM2	✓ YES / NO	✓ YES / NO	✓ YES / NO
Metals	IM3	✓ YES / NO	✓ YES / NO	✓ YES / NO
Metals	IM4	✓ YES / NO	✓ YES / NO	✓ YES / NO
Metals	IM5	✓ YES / NO	✓ YES / NO	✓ YES / NO
Metals	IM6	✓ YES / NO	✓ YES / NO	✓ YES / NO
Metals	IM7	✓ YES / NO	✓ YES / NO	✓ YES / NO
Metals	IM8	✓ YES / NO	✓ YES / NO	✓ YES / NO
Metals	IM9	✓ YES / NO	✓ YES / NO	✓ YES / NO
Metals	IM10	✓ YES / NO	✓ YES / NO	✓ YES / NO
Metals	IM11	✓ YES / NO	✓ YES / NO	✓ YES / NO
Metals	IM12	✓ YES / NO	✓ YES / NO	✓ YES / NO
Metals	IM13	✓ YES / NO	✓ YES / NO	✓ YES / NO
Metals	IM14	✓ YES / NO	✓ YES / NO	✓ YES / NO
Metals	IM15	✓ YES / NO	✓ YES / NO	✓ YES / NO
Wet Chem.	INM1	✓ YES / NO	✓ YES / NO	✓ YES / NO
Wet Chem.	INM2	✓ YES / NO	✓ YES / NO	✓ YES / NO

Attachment VI: Weekly Waste Inspection Form (Example)

Weekly Waste Management Inspection Log (ME001B9-03)
Issuing Authority: Pace ENV - WCOL

Revised:10/8/2020
Page 1 of 1

Weekly Waste Management Inspection Log

Inspection Date: _____ **Inspection Time:** _____ **Inspector Initials:** _____

Area Inspected	Yes/No/NA	Comments/Corrective Actions
Are the doors secured in a manner to prevent unauthorized entry?	Yes	
Are the windows secured in a manner to prevent unauthorized entry?	Yes	
Are the concrete floors free from residue?	Yes	
Is aisle space adequate and free from obstruction?	Yes	
Are all containers closed, in good condition, and properly labeled?	Yes	
Are all wastes accumulating in the proper containers?	Yes	
Are all containers, accumulating wastes containing free liquds, placed in secondary containment?	Yes	
Are all universal wastes accumulating in the proper containers?	Yes	
Are all containers, accumulating wastes, properly segregated to prevent instances of incompatability?	Yes	
Are "no smoking" signs in place where flammables/ignitables are accumulating?	Yes	
Are all metal drums accumulating flammables/ignitables properly grounded?	Yes	
Is the fire extinguisher charged, easily accessible, and tagged?	Yes	
Is the water hose functional and accessible?	Yes	
Is a copy of the the Integrated Contingency Plan accessible and current?	Yes	
Is the public address system/phone system accessible and functional?	Yes	
Are all emergency/spill response materials fully stocked and easily accessible?	Yes	
Is the first-aid kit and safety eyewash/shower station easily accessible and functioning?	Yes	
Are any sample containers disposed of without client labels destroyed?	No	

Attachment VII: Hazardous Waste Manifest Cover Sheet (Example)

Manifest Coversheet (ME0043N-01)
Issuing Authority: Pace ENV - WCOL

Revised:5/3/2021
Page 1 of 1

HAZARDOUS WASTE MANIFEST COVER SHEET

Hazardous Waste Pickup Date: 5/3/2021

Manifest Number(s):
3-Sig Manifest Return Date:

2-Signature Form/Pick-up Checklist

- Drums Requested for Pickup/Present Matches Manifest
- Hazard Codes are Correct for Each Waste Stream
- Pace Representative and Transporter Signature Present

35 Days From Pickup Date: 6/7/2021 45 Days From Pickup Date: 6/17/2021

If the three signature page has not been received within 35 days, contact the disposal facility to determine where the shipment is and request a copy of the three signature page.

If the three signature page has not been received within 45 days, you are required to file an exception report with the local regulating authority.

3 - Signature Manifest(s) Checklist:

- File Cover Sheet
- 3-Sig Forms
- 2-Sig Forms
- Retain for at least 3 years.

Waste Coordinator:
Date Completed:

Attachment VIII: Sample Storage Signs (Example)

	Document Name: Sample Storage - Standard Hold-Time for Client Review	Document Revised: 5/3/2021 Page 1 of 1
	Document No.: ME0043M-01	Issuing Authority: Pace ENV – Local Quality - WCOL

Sample Storage
40 CFR 261.4 (d)(1)(vi)
Standard Hold-Time for Client Review

	Document Name: Sample Storage – Client Requested Long Term Storage	Document Revised: 5/3/2021 Page 1 of 1
	Document No.: ME0043L-01	Issuing Authority: Pace ENV – Local Quality - WCOL

Sample Storage
40 CFR 261.4 (d)(1)(vi)
Client-Requested Long Term Storage

Document Information

Document Number: ENV-MAN-WCOL-0001 **Revision:** 00

Document Title: Laboratory Quality Manual

Department(s): Quality

Date Information

Effective Date: 01 Feb 2021

Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

Signature Manifest

Document Number: ENV-MAN-WCOL-0001 **Revision:** 00

Title: Laboratory Quality Manual

All dates and times are in Central Time Zone.

ENV-MAN-WCOL-0001

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Kelly Nance (090523)	Manager - Quality	22 Jan 2021, 10:22:54 AM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Kelly Nance (090523)	Manager - Quality	22 Jan 2021, 10:23:08 AM	Approved
Xiang Zhu (090550)	Technical Specialist	22 Jan 2021, 10:36:38 AM	Approved
Jeffrey Graham (005586)	Regional Vice President - Oper	22 Jan 2021, 10:54:26 AM	Approved
Kristina Bouknight (090465)	Quality Analyst 1	22 Jan 2021, 11:15:36 AM	Approved
Daniel Wright (090548)	General Manager 1	25 Jan 2021, 08:20:51 AM	Approved
Bradley Belding (090462)	Manager - Operations	25 Jan 2021, 10:57:44 AM	Approved
Cheryl Melton (090516)	Software Architect	25 Jan 2021, 01:35:22 PM	Approved
Lucas Odom (090526)	Project Manager 2	28 Jan 2021, 01:11:29 PM	Approved
Stephanie Atkins (090460)	Manager - Quality Program	29 Jan 2021, 12:18:50 PM	Approved
Michael Kilpatrick (090505)	Manager	01 Feb 2021, 03:04:56 PM	Approved

TITLE PAGE

LABORATORY QUALITY MANUAL

Prepared for:

Pace Analytical Services, LLC
106 Vantage Point Drive
West Columbia, SC 29172
Phone: 803.791.9700

Pace Analytical Services, LLC
228 Westinghouse Boulevard
Charlotte, NC 28273
Phone: 704.589.9802

Pace Analytical Services, LLC
1 Chick Springs Road, Suite 207B
Greenville, SC 29609
Phone: 803.206.9537

Pace Analytical Services, LLC
1436A North Point Lane
Mount Pleasant, SC 29464
Phone: 843.412.7200

Quality Manual Approval Signatories

Approval of this quality manual by managerial personnel is recorded on the Signature Manifest located before the Title Page of this manual.

The individuals listed below represent the management team that was in place on the effective date of this version of the manual for the following location:

Pace Analytical Services, LLC [Analytical Laboratory]
106 Vantage Point Drive
West Columbia, SC 29172
Phone: 803.791.9700

Pace Analytical Services, LLC [Charlotte Service Center]
228 Westinghouse Boulevard
Charlotte, NC 28273
Phone: 704.589.9802

Pace Analytical Services, LLC [Greenville Service Center]
1 Chick Springs Road, Suite 207B
Greenville, SC 29609
Phone: 803.206.9537

Pace Analytical Services, LLC [Charleston Service Center]
1436A North Point Lane
Mount Pleasant, SC 29464
Phone: 843.412.7200

Each of the following individuals is a signatory for the quality manual for the location listed above. The application of their signature to the manual signifies their commitment to communicate, implement, and uphold the requirements, policies and procedures specified in this manual and their commitment to continuously improve the effectiveness of the quality management system based on customer feedback and internal assessment.

Name[1]	Title	Address[2]	Phone[2]
Stephanie Atkins	Corporate Quality Program Manager	Home Office: Located in West Columbia, SC	612-723-8459
Jeffrey Graham	Regional Vice-President - Operations	8 Tower Cir E, Ormond Beach, FL 32174	704-977-0954
Daniel Wright	General Manager	All locations listed above	Above
Kelly Nance	Quality Manager	All locations listed above	Above
Kristina Bouknight	Health & Safety, however named.	All locations listed above	Above
Cheryl Melton	IT Manager	All locations listed above	Above
Robert Zhu	Technical Specialist[3]	Analytical Laboratory – West Columbia, SC	Above
Bradley Belding	Operations Manager	Analytical Laboratory – West Columbia, SC	Above
Michael Kilpatrick	Client Services Manager – Acting Director for Charlotte Service Center	Charlotte Service Center – Charlotte, NC	Above
Lucas Odom	Project Manager – Acting Director for Greenville Service Center	Greenville Service Center – Greenville, SC	Above

[1] Members of the local management team are subject to change during the lifecycle of this document version.
[2] Include if different from the physical address and phone number of the facility.
[3] This individual serves as an Acting Technical Manager for TNI for one or more fields of accreditation.

TABLE OF CONTENTS

1.0 PURPOSE AND SCOPE

1.1 Purpose

This quality manual (manual) outlines the quality management system (QMS) and management structure of the laboratories and service centers affiliated with the environmental sciences (ENV) division of Pace Analytical Services, LLC (PAS). A laboratory is defined by ENV as any facility, however named, that provides testing, sampling, or field measurement services. When the term 'laboratory" is used in this manual, the term refers to all locations listed on the Title Page of this manual and in Section 4.1.3 unless otherwise specified.

The ENV quality management system is also referred to as the quality program throughout this document. In this context, the phrase "quality management system" and "quality program" are synonymous and may be referred to by the acronym QMS.

The quality management system is the collection of policies and processes established by ENV management to consistently meet customer requirements and expectations, and to achieve the goals of providing PAS customers with high quality, cost-effective, analytical measurements and services.

The quality management system is also intended to establish conformance[1] and compliance with the current versions of the following international and national quality system standards:

- ISO/IEC 17025: *General requirements for the competence of testing and calibration laboratories*
- NELAC/TNI Standard Volume 1: *Management and Technical Requirements for Laboratories Performing Environmental Analysis*

[1]The statement of conformity to these Standards pertains only to testing and sampling activities carried out by the laboratory at its physical address, in temporary or mobile facilities, in-network, or by laboratory personnel at a customer's facility.

In addition to the international and national standards, the quality management system is designed to achieve regulatory compliance with the various federal and state programs for which the laboratory provides compliance testing and/or holds certification or accreditation. When federal or state requirements do not apply to all ENV locations, the requirements for compliance to those specifications are provided in addendum to this manual or in other documents that supplement the manual. Customer-specific project and program requirements are not included in the manual in order to maintain client confidentiality.

- A list of accreditation and certifications held by each laboratory associated with this manual is provided in Appendix A.
- A list of analytical testing capabilities offered by each laboratory associated with this manual is provided in Appendix B.

1.2 Scope and Application

This manual applies to each of the PAS locations listed on the Title Page.

The manual was prepared from the quality manual template (template) created by ENV corporate quality personnel. The template outlines the minimum requirements ENV management considers necessary for every ENV location, regardless of scope of services or number of personnel, to establish in order to maintain a quality management system that achieves the objectives of the Quality Policy (See 4.2.2). In this regard, the template is the mechanism used by the corporate officers (a.k.a. 'top

management') to communicate their expectations and commitment for the quality program to ENV personnel.

Each location also has the responsibility to comply with federal and state regulatory and program requirements for which it provides analytical services and holds certification or accreditation. When those requirements are more stringent than the template, the requirements for compliance are provided in addendum to this manual or in other documents that supplement the manual. This document structure maintains consistency in the presentation of the quality management system across the network while providing the location a mechanism to describe and achieve compliance requirements on a program basis.

1.2.1 Quality Manual Template

The quality manual template is developed by the Corporate Quality Director with contribution and input from corporate quality personnel and the corporate officers. Approval of the template by the corporate officers (aka "top management") confirms their commitment to develop and maintain a quality management system appropriate for the analytical services offered by the organization and to communicate their expectations of the quality program to all personnel.

The template and instructions for use of the template are released by corporate quality personnel to the quality assurance manager responsible for each location (Local QM). The local QM uses the template to prepare the laboratory's manual by following the instructions provided. Since the template provides the minimum requirements by which ENV locations must abide, the laboratory may not alter the font, structure or content of the template except where specified by instruction to do so. As previously stated, program specific requirements are provided in addendum or in documents that supplement this manual.

The template is reviewed by corporate quality personnel annually and updated if needed. More frequent review and revision may be necessary to manage change, to maintain conformance and compliance to relevant standards, or to meet customer expectations.

See standard operating procedure (SOP) ENV-SOP-CORQ-00015 *Document Management and Control* for more information.

1.2.2 Laboratory Quality Manual

The manual is approved and released to personnel under the authority of local management whose signatures are identified on the Manual Signatory Page of this manual. The manual is reviewed annually, and location specific information is updated, if needed. More frequent review and revision may be necessary when there are significant changes to the organizational structure, capabilities, and resources of the laboratory. Review and revision of the manual is managed by the local QM. If review indicates changes to the main body of the manual are necessary to maintain conformance and compliance to relevant standards, or to meet customer expectations, the local QM will notify corporate quality personnel to initiate review and/or revision of the template.

See SOP ENV-SOP-CORQ-00015 *Document Management and Control* for more information.

1.2.3 References to Supporting Documents

The template and the manual include references to other laboratory documents that support the quality management system such as policies and standard operating procedures (SOPs).

These references include the document's document control number and may include the document title.

This information is subject to change. For example, an SOP may be converted to a policy or the document's title may change. For these types of administrative changes, the manual and template are updated to reflect the editorial change during the manual's next scheduled review/revision cycle or the next time a new version of the manual is released, whichever is sooner.

The local QM maintains a current list of controlled documents used at each location that support the quality management system. This list, known as the "Master List", lists each document used by document control number, title, version, effective date, and reference to any document(s) that the current version supersedes. When there is a difference between the manual and the Master List, the document information in the Master List takes precedence. The current Master List is readily available to personnel for their use and cross-reference. Parties external to the laboratory should contact the laboratory for the most current version.

2.0 REFERENCES

References used to prepare this manual include:

- "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act." Federal Register, 40 CFR Part 136, most current version.
- "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods." SW-846.
- "Methods for Chemical Analysis of Water and Wastes", EPA 600-4-79-020, 1979 Revised 1983, U.S. EPA.
- U.S. EPA Contract Laboratory Program Statement of Work for Organic Analysis, current version.
- U.S. EPA Contract Laboratory Program Statement of Work for Inorganic Analysis, current version.
- U.S. EPA Contract Laboratory Program Statement of Work for Superfund Analytical Methods, current version.
- "Standard Methods for the Examination of Water and Wastewater." Current Edition APHA-AWWA-WPCF.
- "Annual Book of ASTM Standards", Section 4: Construction, Volume 04.04: Soil and Rock; Building Stones, American Society of Testing and Materials.
- "Annual Book of ASTM Standards", Section 11: Water and Environmental Technology, American Society of Testing and Materials.
- "NIOSH Manual of Analytical Methods", U.S. Department of Health and Human Services, National Institute for Occupational Safety and Health, most current version.
- "Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water", U.S. EPA, Environmental Monitoring and Support Laboratory – Cincinnati (Sep 1986).
- Quality Assurance of Chemical Measurements, Taylor, John K.; Lewis Publishers, Inc. 1987.
- Methods for Non-conventional Pesticides Chemicals Analysis of Industrial and Municipal Wastewater, Test Methods, EPA-440/1-83/079C.

- Environmental Measurements Laboratory (EML) Procedures Manual, HASL-300, US DOE, February 1992.
- Requirements for Quality Control of Analytical Data, HAZWRAP, DOE/HWP-65/R1, July 1990.
- Quality Assurance Manual for Industrial Hygiene Chemistry, AIHA, most current version.
- National Environmental Laboratory Accreditation Conference (NELAC) Standard- most current version.
- ISO/IEC 17025, General requirements for the competence of testing and calibration laboratories, 2nd Edition 2005-05-15; 3rd Edition 2017-11

  The following are implemented by normative reference to ISO/IEC 17025:

  - ISO/IEC Guide 99, *International vocabulary of metrology –Basic and general concepts and associated terms*
  - ISO/IEC 17000, *Conformity assessment – Vocabulary and general principles*
- Department of Defense Quality Systems Manual (QSM), most current version.
- TNI (The NELAC Institute) Standard, 2009 and 2016 versions.
- UCMR Laboratory Approval Requirements and Information Document, most current version.
- US EPA Drinking Water Manual, most current version.

3.0 TERMS AND DEFINITIONS

Refer to Appendix C for terms, acronyms, and definitions used in this manual and in other documents used by the laboratory to support the quality management system.

4.0 MANAGEMENT REQUIREMENTS

4.1 Organization

4.1.1 Legal Identity

Pace Analytical Services, LLC is authorized under the State of Minnesota to do business as a limited liability company.

4.1.1.1 Change of Ownership

If there is a change of ownership, if a location goes out of business, or if the entire organization ceases to exist, Pace Analytical Services, LLC ensures that regulatory authorities are notified of the change within the time-frame required by each state agency for which the location is certified or accredited.

Requirements for records and other business information are addressed in the ownership transfer agreement or in accordance with appropriate regulatory requirements, whichever takes precedence.

4.1.2 Compliance Responsibility

Laboratory management has the responsibility and authority to establish and implement procedures and to maintain sufficient resources necessary to assure its activities are carried out in such a way to meet the compliance requirements of the quality management system.

4.1.3 Scope of the Quality Management System

The quality management system applies to work carried out at each location covered by this manual including permanent facilities, at sites away from its permanent facilities, or in associated temporary or mobile facilities.

The permanent and mobile facilities to which this manual applies are listed on the Title Page of this manual.

4.1.4 Organization History and Information

Founded in 1978, Pace Analytical Services, LLC (PAS) is a privately held scientific services firm operating one of the largest full-service contract laboratory and service center networks in the United States. The company's network offers inorganic, organic and radiochemistry testing capabilities; specializing in the analysis of trace level contamination in air, drinking water, groundwater, wastewater, soil, biota, and waste.

With over 90 laboratories and services centers in the contiguous US and in Puerto Rico, the network provides project support for thousands of industry, consulting, engineering and government professionals.

PAS delivers the highest standard of testing and scientific services in the market. We offer the most advanced solutions in the industry, backed by truly transparent data, a highly trained team, and the service and support that comes from four decades of experience.

4.1.4.1 Organization Structure

Each location maintains a local management structure under the oversight and guidance of corporate personnel. Local management is responsible for making day-to-day decisions regarding the operations of the facility, implementing the quality management system, upholding the requirements of the quality program, and for supervision of personnel.

Local management is provided by the Regional Vice-President - Operations (RVPO), Corporate Quality Program Manager (QPM), General Manager (GM), Quality Manager (QM), and department specific management and supervisory personnel.

The GM reports to a Vice-President of Operations (RVPO), who is responsible for the management of multiple laboratories and service centers across the division. The RVPO reports directly to the Chief Operating Officer (COO).

The QM reports to a Quality Program Manager (QPM), who is responsible for managing quality personnel for multiple locations across the division. The QPM reports directly to the Corporate Quality Director (CQD). The QM also maintains an indirect reporting relationship to the GM of each location that the QM manages.

Technical oversight for each location is provided by corporate personnel, RVPO, QPM, GM, QM, and department-specific management.

Refer to the organization charts provided in Appendix D to view the management structure, reporting relationships, and the interrelationships between positions.

4.1.5 Management Requirements

4.1.5.1 Personnel

The laboratory is staffed with administrative and technical personnel who perform and verify work under the supervision of managerial personnel.

- Technical personnel include analysts and technicians that generate or contribute to the generation of analytical data and managerial personnel that oversee day to day supervision of laboratory operations. Including the reporting of analytical data and results, monitoring QA/QC performance, and monitoring the validity of analysis to maintain data integrity and reliability.
- Administrative personnel support the day-to-day activities of the laboratory.
- IT personnel maintain the information technology systems and software used at the laboratory.
- Client services personnel include project managers and support staff that manage projects.
- Managerial personnel make day-to-day and long-term decisions regarding the operations of the facility, supervise personnel, implement the quality management system and uphold the requirements of the quality program.

All personnel regardless of responsibilities are expected to carry out their duties in accordance with the policies and processes outlined in this manual and in accordance with standard operating procedures (SOPs) and other quality system documents. The laboratory's policies and procedures are designed for impartiality and integrity. When these procedures are fully implemented, personnel remain free from undue pressure and other influences that adversely impact the quality of their work or data.

4.1.5.1.1 Key Personnel

Key personnel include the management positions that have the authority and responsibility to plan, direct, and control, activities of the division (corporate) or the laboratory.

The following tables list key personnel positions by PAS job title and the position's primary deputy:

Key Personnel: Corporate

Key Personnel	Primary Deputy
Chief Executive Officer	Chief Operating Officer
Chief Operating Officer	Chief Executive Officer
Chief Compliance Officer	Quality Director
Corporate Quality Director	Chief Compliance Officer
Health and Safety Director	Chief Compliance Officer
IT Director	LIMS Administrator, however named.

Key Personnel: Laboratory

Key Personnel	Primary Deputy

Regional Vice President - Operations	Chief Operating Officer or as designated.
Quality Program Manager	A different QPM or Corporate Quality Director
General Manager	Regional Vice President of Operations
Quality Manager	Quality Program Manager
Manager – Client Services	General Manager or as designated.
Local IT	Corporate IT Director or as designated.
Department Manager	General Manager
Technical Director[1]/Manager[1] Acting Technical Manager TNI	Another qualified employee
Operations Manager[1]	General Manager

[1] *Position may not be staffed at each location.*

Some state certification programs require the agency to be notified when there has been a change in key personnel. Program-specific requirements and timeframes for notification by agency, are tracked and upheld by the local QM, when these requirements apply.

4.1.5.2 Roles and Responsibilities

The qualifications, duties, and responsibilities for each position are detailed in job descriptions maintained by PAS's corporate Human Resource's Department (HR).

The following summaries briefly identify the responsibility of key personnel positions in relation to the ENV quality management system.

Chief Executive Officer (CEO): The CEO has overall responsibility for performance of the organization and endorses the quality program. Working with corporate and laboratory management, the CEO provides the leadership and resources necessary for ENV locations to achieve the goals and objectives of the quality management system and quality policy statement.

Chief Operating Officer (COO): The COO oversees all aspects of operations management including, strategic planning, budget, capital expenditure, and management of senior management personnel for ENV In this capacity, the COO provides leadership and resources necessary to help top management at each ENV location achieve the goals and objectives of the quality management system and quality policy statement.

Chief Compliance Officer (CCO): The CCO oversees the quality assurance and environmental health and safety programs (EHS) for each business unit. The CCO is responsible for planning and policy development for these groups to ensure regulatory compliance and to manage risk. The position provides leadership and guidance necessary for all PAS locations to achieve the goals and objectives of the quality and EHS programs.

The CCO also serves as the Ethics Officer (ECO). The ECO develops the Ethics and Data Integrity Policy and Training Program and provides oversight for reporting and investigation of ethical misconduct to maintain employee confidentiality during the process. The ECO provides guidance and instruction for follow-up actions necessary to remedy the situation and deter future recurrence.

Corporate Director of Quality (CQD): The Corporate Director of Quality is responsible for developing and maintaining the ENV quality program under guidance and assistance from the CEO, COO, and CCO. This position develops corporate quality policy and procedure and analyzes metric data and other performance indicators to assess and communicate the effectiveness of the quality program to top management. The position provides leadership and guidance for implementation of the quality program across all ENV locations.

Corporate Quality Program Manager (QPM): The Quality Program Manager is responsible for managing the implementation of the ENV quality program for one or more locations in the network. Working with the CQD and local laboratory management to which they are assigned, the QPM provides leadership, guidance and resources, including allocation of personnel, necessary to achieve the goals of ENV quality program.

Corporate Director of Information Technology: The Corporate Director of IT oversees the systems and processes of information technology used to support the quality program. These systems include Laboratory Information Management Systems (LIMS); data acquisition, reduction, and reporting software; virus-protection, communication tools, and ensuring the integrity and security of electronic data.

Regional Vice-President of Operations): The RVPO has full responsibility for administrative and operations management and performance of a group of ENV laboratories and service centers. Working with the COO and local laboratory management, the RVPO provides leadership, guidance and resources, including allocation of personnel, necessary to achieve the goals of ENV quality program.

General Manager (GM): The GM is responsible for the overall performance and administrative and operations management of an ENV location and associated service center(s). This position is responsible to provide leadership and resources, including allocation and supervision of personnel, necessary for the location to implement and achieve the goals of the PAS quality program. In this capacity, the position assures laboratory personnel are trained on and understand the structure and components of the quality program defined in this manual as well as the policies and procedures in place to implement the quality management system.

The GM of NELAC/TNI Accredited laboratories is also responsible for the designation of technical personnel to serve as acting technical managers for TNI for the fields of accreditation held by the laboratory (See Section 4.1.5.2.1) and for notifying the accreditation body (AB) of any extended absence or reassignment of these designations.

Quality Manager (QM): The QM oversees and monitors implementation of the quality management system for each ENV location assigned and communicates deviations to laboratory management. The QM is independent of the operation activities for which they provide oversight and has the authority to carry out the roles and responsibilities of their position without outside influence.

Additionally, in accordance with the TNI Standard, the QM:

- serves as the focal point for QA/QC and oversees review of QC data for trend analysis;
- evaluates data objectively and perform assessments without outside influence;
- has documented training and experience in QA/QC procedures and the laboratory's quality system;
- has a general knowledge of the analytical methods offered by the laboratory;
- coordinates and conducts internal systems and technical audits;
- notifies laboratory management of deficiencies in the quality system;
- monitors corrective actions;
- provides support to technical personnel and may serve as the primary deputy for the acting TNI Technical Manager(s).

Manager-Client Services (CSM): This position is responsible for training and management of client facing staff that serve as the liaison between PAS and the customer to ensure that projects are successfully managed to meet the expectations and needs of PAS customers. This position is also responsible for sharing positive and negative customer feedback with laboratory management so that this information may be used to improve the quality program.

Local IT Manager, however named: Local IT managers are responsible for maintaining the IT systems used to support the quality program. These systems include Laboratory Information Management Systems (LIMS); data acquisition, reduction, and reporting software; virus-protection, communication tools, and ensuring the integrity and security of electronic data.

Department Manager (DM): The DM is responsible for administrative and operations management and implementation of the quality management system in the work area he/she oversees. These responsibilities include but are not limited to: training and supervision of personnel, monitoring work activity to maintain compliance with this manual, SOPs, policies and other instructional documents that support the quality management system; method development, validation and the establishment and implementation of SOPs to assure regulatory compliance and suitability for intended purpose; monitoring QA/QC performance, proper handling and reporting of nonconforming work, purchasing of supplies and equipment adequate for use, maintaining instrumentation and equipment in proper working order and calibration, and general maintenance of administrative and technical processes and procedures established by the laboratory.

Operations Manager (OM): The OM is responsible for management of production and/or other duties assigned by the GM.

4.1.5.2.1 Acting Technical Manager (TNI Accreditation):

For ENV locations that are NELAC/TNI accredited:

The TNI Standard specifies requirements for the qualification and duties of technical personnel with managerial responsibility. These requirements are associated in the Standard to the designation 'technical manager(s), however named'. These responsibilities may be assigned to multiple individuals and are not associated with any specific job title.

The TNI requirements for personnel that provide technical oversight correlate with ENV job descriptions for Department Manager or Supervisor. However, the duties may be assigned to any PAS employee that meets the TNI specified qualifications.

Personnel assigned this designation retain their assigned job title. The job title may be appended with *"acting as technical manager for TNI"* and the technology or field of accreditation for which the employee is approved, if necessary.

When TNI Accreditation Bodies (AB) refer to these employees as 'technical manager' or 'technical director' on the official certificate or the scope of accreditation, this reference is referring to their approval to carry out duties of the 'technical manager, however named' as specified in the TNI Standard.

In accordance with the TNI Standard, the acting Technical Manager(s) for TNI are responsible for monitoring the performance of QC/QA in the work areas they oversee.

If the absence of any employee that is approved as acting technical manager for TNI exceeds 15 calendar days, the duties and responsibilities specified in the TNI Standard are temporarily reassigned to another employee that meets the qualifications for the technology or field of accreditation. If the employee's absence exceeds 35 calendar days, the QM will formally notify the TNI primary AB of the absence and the details of reassignment of duties in writing.

Refer to the applicable TNI Standard for requirements for technical oversight and required qualifications of the acting Technical Manager(s) for each discipline (chemical, limited inorganic chemistry, and microbiology).

4.1.5.3 Conflict of Interest

A conflict of interest is a situation where a person has competing interests. Laboratory management looks for potential conflict of interest and undue pressures that might arise in work activities and then includes countermeasures in policies and procedures to mitigate or eliminate the conflict.

See policy COR-POL-0004 *Ethics Policy* for more information.

4.1.5.4 Confidentiality

Laboratory management is committed to preserving the confidentiality of PAS customers and confidentiality of business information.

Procedures used by the laboratory to maintain confidentiality include:

- A Confidentiality Agreement which all employees are required to sign at the time of employment and abide by the conditions of throughout employment;
- Record retention and disposal procedures that assure confidentiality is maintained;
- Physical access controls and encryption of electronic data; and
- Protocol for handling Confidential Business Information (CBI).

Client information obtained or created during work activities is considered confidential and is protected from intentional release to any person or entity other than the client or the client's authorized representative, except when the laboratory is required by law to release confidential information to another party, such as a regulatory agency or for litigation purposes. In which case, the laboratory will notify the client of the release of information and the information provided.

The terms of client confidentiality are included in ENV Standard Terms and Conditions (T&C). With the acceptance of ENV Terms and Conditions and/or the implicit contract for analytical services that occurs when the client sends samples to the laboratory for testing, the client authorizes PAS to release confidential information when required.

See policy COR-POL-0004 *Ethics Policy* for more information.

4.1.5.5 Communication

Communication is defined as the imparting or exchanging of news and information. Effective (good) communication occurs when the person(s) you are exchanging information with actively gets the point and understands it.

4.1.5.5.1 Workplace Communication

Good communication in the workplace is necessary to assure work is done correctly, efficiently, and in accordance with client expectations.

Instructions for how to carry out work activities are communicated to personnel via written policy, standard operating procedures, and standard work instructions.

Information about laboratory performance (positive and negative) and ideas for improvement are communicated using various communication channels such as face to face meetings, video conferencing, conference calls, email, memoranda, written reports, and posters.

4.1.5.5.2 External Communication

Communication with external parties such as customers, vendors, business partners, and regulatory agencies takes place every day.

Laboratory management ensure personnel learn to communicate in professional and respectful ways in order to build strong relationships and learn to communicate effectively to avoid misunderstanding.

4.2 Quality Management System

4.2.1 Quality Management System Objectives

The objectives of the laboratory's quality management system are to provide clients with consistent, exemplary professional service, and objective work product that is of known and documented quality that meets their requirements for data usability and regulatory compliance.

Objective work product is analytical services, data, test results, and information that is not influenced by personal feeling or opinions. The quality of being objective is also known as 'impartiality'.

4.2.1.1 Impartiality

The laboratory achieves and maintains impartiality by implementing and adhering to the policies and processes of the quality management system, which are based on industry accepted standards and methodologies.

The laboratory's procedures for handling nonconforming work (See 4.9), corrective and preventive actions (See 4.11, 4.12) and management review (See 4.15) are the primary mechanisms used to identify risk to impartiality and to prompt actions necessary to eliminate or reduce the threat when risk to impartiality is suspected or confirmed.

4.2.1.2 Risk and Opportunity Assessment

Risks are variables that make achieving the goals and objectives of the quality management system uncertain. An opportunity is something that has potential positive consequences for the laboratory.

Laboratory personnel manage risks and opportunities on a daily basis by carrying out the processes that make up the quality management system. Some of the ways in which the quality management system is designed to identify, minimize, or eliminate risk on a daily basis include but are not limited to:

- Capability and capacity reviews of each analytical service request to assure the laboratory can meet the customer's requirements;
- Maintenance of accreditation and certification for test methods in multiple states and programs to cover a broad range of jurisdiction for regulatory compliance;
- SOPs and other controlled instructional documents are provided to personnel to eliminate variability in process. These documents include actions to counter risk

factors inherent in the process and are reviewed on a regular basis for on-going suitability and relevancy;

- Participation in proficiency testing programs and auditing activities to verify on-going competency and comparability in performance;
- Provision of on-the-job training and established protocol for quality control (QC) corrective action for nonconforming events;
- An established program for ethics, and data integrity;
- Tiered data review process;
- Culture of continuous improvement;
- Monitoring activities to assess daily and long-term performance; and
- Annual critical review of the effectiveness of the quality management system.

ENV also promotes a continuous improvement culture based on the principles of lean manufacturing. These principles include 3P (Process, Productivity, Performance) and Kaizen. 3P is a platform used by Pace to share best practices and standardization across the network to achieve operational excellence. Kaizen is a team-based process used to implement tools and philosophies of lean to reduce waste and achieve flow with the purpose of improving both external and internal customer satisfaction. ENV's lean programs and activities help to mitigate risk because they generate a collective understanding of vulnerabilities and utilize group-effort to develop and implement solutions at all levels.

Risk and opportunities may also be formally identified using specific risk and opportunity assessment methods such as SWOT Analysis (Strength, Weakness, Opportunity, Threats) and 3-Stage Impact/Probability Grids.

4.2.1.3 Communication of the Quality Management System

This manual is the primary mechanism used by laboratory management to communicate the quality management system to laboratory personnel.

To assure personnel understand and implement the quality program outlined in the manual:

- All laboratory personnel are required to sign a Read and Acknowledgement Statement to confirm the employee has: 1) been informed of the manual by laboratory management, 2) has access to the manual, 3) has read the manual 4) understands the content of the manual, and 5) agrees to abide by the requirements, policies and procedures therein.
- Personnel are informed that the manual provides the "what" of the quality management system. The "how to" implementation of the quality management system is provided in policy, SOPs, standard work instructions, and other controlled instructional documents.

4.2.2 Quality Policy Statement

The quality policy of the laboratory is to provide customers with data of known and documented quality fit for their intended purpose. The laboratory achieves this policy by implementing the quality management system defined in this manual, by following industry accepted protocol for analytical testing and quality assurance and quality control (QA/QC) activities, by conformance with published and industry accepted testing methodologies, and by compliance with international and national standards for the competency and/or accreditation of testing laboratories.

Intrinsic to this policy statement is each of the following principles:

- The laboratory will provide customers with reliable, consistent, and professional service. This is accomplished by making sure the laboratory has the resources necessary to maintain capability and capacity; that staff are trained and competent to perform the tasks they are assigned; that client-facing staff are trained and prepared to find solutions to problems and to assist customers with their needs for analytical services. Customer feedback, both positive and negative, is shared with personnel and used to identify opportunities for improvement.
- The laboratory maintains a quality program that complies with applicable state, federal, and industry standards for analytical testing and competency.

  ISO/IEC 17025 and the TNI (The NELAC Institute) Standard is used by ENV to establish the minimum requirements of the ENV quality program.

  ISO/IEC 17025 is a competency standard that outlines the general requirements for the management system for calibration and testing laboratories. It is the primary quality system standard from which other quality system standards, such as the TNI Standard, are based. The TNI Standards are consensus standards that provides management and technical requirements for laboratories performing environmental analysis.
- Laboratory management provides training to personnel so that all personnel are familiar with the quality management system outlined in this manual and that they understand that implementation of the quality management system is achieved by adherence to the organization's policies and procedures.
- Laboratory management continuously evaluates and improves the effectiveness of the quality management system by responding to customer feedback, and other measures of performance, such as but not limited to: the results of internal/external audits, proficiency testing, metrics, trend reports, and annual and periodic management reviews.

4.2.2.1 Ethics Policy / Data Integrity Program

PAS has established a comprehensive ethics and data integrity program that is communicated to all PAS employees in order that they understand what is expected of them. The program is designed to promote a mindset of ethical behavior and professional conduct that is applied to all work activities.

The key elements of the PAS Ethics / Data Integrity Program include:

- Ethics Policy (COR-POL-0004);
- Ethics Officer;
- Standardized data integrity training course taken by all new employees on hire and a yearly refresher data integrity training course for all existing employees;
- Policy Acknowledgement Statements that all PAS personnel, including contract and temporary, are required to sign at the time of employment and again during annual refresher training to document the employee's commitment and obligation to abide by the company's standards for ethics, data integrity and confidentiality;
- SOPs that provide instructions for how to carry out a test method or process to assure tasks are done correctly and consistently by each employee;
- On the Job Training;
- Data integrity monitoring activities which include, but are not limited to, primary, secondary and completeness data reviews, internal technical and system audits, data audits, data surveillance, and proficiency testing; and
- Confidential reporting process for alleged ethics and data integrity issues.

All laboratory managers are expected to provide a work environment where personnel feel safe and can report unethical or improper behavior in complete confidence without fear of retaliation. Retaliation against any employee that reports a concern is not tolerated.

PAS has engaged Lighthouse Services, Inc. to provide personnel with an anonymous reporting process available to them 24 hours a day/7 days per week. The alert line may be used by any employee to report possible violations of the company's ethics and data integrity program. When using the reporting process, the employee does need to specify the location of concern and when reporting by email, also include the company name. Messages are collected, documented, reviewed, and will be followed up on by the Ethics Compliance Officer to resolve the matter. Investigations concerning data integrity are kept confidential.

Lighthouse Compliance Alert Lines:

English Speaking US & Canada	(844) 940-0003
Spanish Speaking North America	(800) 216-1288
Internet	www/lighthouse-services.com/pacelabs
Email	reports@lighthouse-services.com

4.2.3 Management Commitment: Quality Management System

Evidence of management's commitment for the development, maintenance, and on-going improvement of the quality management system is provided by the application of their signature of approval to this manual. Their signature confirms they understand their responsibility to implement the quality management system outlined in this manual, to

communicate the quality program to personnel, and to uphold requirements of the program during work activities.

4.2.4 Management Commitment: Customer Service

Management communicates the importance of meeting customer and regulatory requirements to personnel by training personnel on the quality management system outlined in this manual, implementing the quality management system outlined in this manual, and upholding these requirements for all work activities.

4.2.5 Supporting Procedures

Documents that support this manual and quality management system are referenced throughout this manual. The structure of the document management system is outlined in SOP ENV-SOP-CORQ-0015 *Document Management and Control* and summarized in the following subsections.

4.2.5.1 Quality Management System Document Structure

Documents associated with the quality management system are classified into document types that identify the purpose of the document and establish how the document is managed and /or controlled.

Document types are ranked to establish which documents takes precedence when there is an actual or perceived conflict between documents and to establish the hierarchal relationships between documents. The ranking system also provides information to document writers and reviewers to assure downline documents are in agreement with documents of higher rank. Project specific documents are not ranked because client specific requirements are not incorporated into general use documents in order to maintain client confidentiality.

Examples: ENV QMS Documents: Internal

Document Type	Purpose
Quality Manual	Outlines the laboratory's quality management system and structure and how it works for a system including policy, goals, objectives and detailed explanation of the system and the requirements for implementation of system. Includes roles and responsibilities, relationships, procedures, systems and other information necessary to meet the objectives of the system described.
Policy	Provide requirements and rules for a PAS process and is used to set course of actions and to guide and influence decisions. Policy describes the "what", not the "how".
Standard Operating Procedure	Provide written and consistent set of instructions or steps for execution of a routine process, method, or set of tasks performed by PAS. Includes both fundamental and operational elements for implementation of the systems described in PAS manual(s). Assures that activities are performed properly in accordance with applicable requirements. Designed to ensure consistency, protect EHS of employees and environment, prevent failure in the process and ensure compliance with company and regulatory requirements. SOPs describes the "how" based on policy.
Standard Work Instruction	Provide step by step visual and/or written instruction to carry out a specific task to improve competency, minimize variability, reduce work injury and strain, or to boost efficiency and quality of work (performance). SWI are associated with an SOP unless the task described is unrelated to generation of or contribution to environmental data or analytical results.
Template	Pre-formatted document that serves as a starting point for a new document.

Guide	Provide assistance to carry out a task.
Form	Used for a variety of purposes such as to provide a standardized format to record observations, to provide information to supplement an SOP.
Guidance	Non-binding advice used to explain internal policies, procedures, or practices.

Example: ENV QMS Documents: External

Certificate	Lists parameters, methods, and matrices for which the laboratory is certified/accredited to perform within the jurisdiction of the issuing regulatory agency or accreditation body.
Reference Document	Provide information, protocol, instructions, and/or requirements. Issued by the specifier. Examples include quality system standards such as ISO/IEC, TNI, DoD and published referenced methods such as Standard Methods, ASTM, SW846, EPA, and federal and state regulatory bodies.
Project Document	Provides requirements necessary to meet individual client expectations for intended use of data. Examples include project quality assurance plans (QAPP), client-program technical specifications, contracts, and other agreements.

Document Hierarchy

Rank	Document
1	Reference Documents
2	Corporate Manual
3	Corporate Policy
4	Corporate SOP
5	Corporate SWI, Templates, Guides, Forms, Guidance
6	Laboratory Manual
7	Laboratory SOP
8	Laboratory SWI, Templates, Guide, Forms, Guidance
NA	Project Documents

4.2.6 Roles and Responsibilities

The roles and responsibilities for technical management and the quality manager is provided in section 4.1.5.2.

4.2.7 Change Management

When significant changes to the ENV quality management system are planned, these changes are managed by corporate quality personnel to assure that the integrity of the quality management system is maintained.

4.3 Document Control

4.3.1 General

The laboratory's procedures for document control are provided in SOP ENV-SOP-CORQ-0015 *Document Management and Control.*

The laboratory uses electronic document management software (eDMS) to carry out the document control procedures of the SOP. eDMS automates the process for unique document identification, version control, approval, access, and archival. The eDMS software used by ENV restricts access to archived documents except to authorized users to prevent the use of obsolete documents.

The local QM maintains a master list of controlled documents used at the laboratory. The master list includes the document control number, document title, and current revision status and is made available to personnel for their reference.

See SOP ENV-SOP-CORQ-0015 *Document Management and Control* for more information.

4.3.2 Document Approval and Issue

Documents that support the quality management system are reviewed by qualified personnel and approved by laboratory management prior to release for general use.

Only the approved versions of documents are available to personnel for use unless use of a draft document is authorized by management.

See SOP ENV-SOP-CORQ-0015 *Document Management and Control* for more information.

4.3.3 Document Review and Change

Unless a more frequent review is required by regulatory, certification or accreditation program the laboratory formally reviews documents at least every two years to ensure the document remains current, appropriate, and relevant.

Documents are also informally reviewed every time the document is used. Personnel are expected to refer to and follow instructions in controlled documents when they carry out their work activities. Consequently, any concerns or problems with the document should be caught and brought to the attention of laboratory management on an on-going basis.

Documents are revised whenever necessary to ensure the document remains usable and correct. Older document versions and documents no longer needed are made obsolete and archived for historical purposes.

ENV does not allow hand-edits to documents. If an interim change is needed pending re-issue of the document, the interim change is communicated to those that use the document using a formal communication channel, such as SOP Change in Progress form, email, or memorandum.

The document review, revision, and archival process is managed by quality personnel at the location from which the document was released using the procedures established in SOP ENV-SOP-CORQ-0015 *Document Management and Control.*

4.4 Analytical Service Request, Tender, and Contract Review

The laboratory's management and/or client service personnel perform thorough reviews of requests and contracts for analytical services to verify the laboratory has the capability, capacity, and resources necessary to successfully meet the customer's needs. These review procedures are described in laboratory SOP *Project Management* [AD SOP ME001HD].

The procedures in this SOP(s) are established to ensure that:

- The laboratory understands the purpose of data collection in order to ensure the test methods requested are appropriate for the intended use of the data and capable of meeting the client's data quality objectives;

- The laboratory and any subcontractor has the capability, capacity, and resources to meet the project requirements and expectations within the requested time frame for delivery of work product;
- Any concerns that arise from review are discussed and resolved with the client; and
- The results of review and any correspondence with the client related to this process and/or any changes made to the contract are recorded and retained for historical purposes.

Capability review confirms that the in-network laboratories and any potential subcontractors hold required certification/accreditation for the test method, matrix, and analyte and verifies the laboratory can achieve the client's target compound list and data quality objectives (DQOs) for analytical sensitivity and reporting limits, QA/QC protocol, and hardcopy test report and electronic data deliverable (EDD) formats.

Capacity review verifies that the in-network laboratories and any potential subcontractors are able to handle the sample load and deliver work production within the delivery timeframe requested.

Resource review verifies that the laboratory and any potential subcontractors have adequate qualified personnel with the skills and competency to perform the test methods and services requested and sufficient and proper equipment and instrumentation needed to perform the services requested.

4.5 Subcontracting and In-Network Work Transfer

The terms 'subcontract' and "subcontracting" refers to work sent to a business external to Pace Analytical Services, LLC (PAS) and the term 'subcontractor' refers to these external businesses, which are also called vendors.

Work transferred within the ENV network is referred to as interregional work orders (IRWO) and network laboratories are referred to as IRWO, IR, or a network laboratory.

The network of ENV laboratories offers comprehensive analytical capability and capacity to ensure PAS can meet a diverse range of client needs for any type of project. If the laboratory receives a request for analytical services and it cannot fulfill the project specifications, the laboratory's client services team will work with the client to place the work within the ENV network. When it is not possible to place the work within network, the laboratory will, with documented client approval, subcontract the work to a subcontractor that has the capabilities to meet the project specifications and can meet the same commitment agreed on between the laboratory and the client. Some client programs require client consent even for in-network work transfer, and when this applies, the client services team obtains consent as required. The laboratory retains the record of client notification and their consent in the project record for historical purposes.

Whenever work is transferred to a subcontractor or an in-network laboratory, the laboratory responsible for management of the project verifies each of these qualifications:

- The subcontractor or in network laboratory has the proper accreditation/certifications required for the project and these are current; and
- The use of the subcontractor or in network laboratory is approved by the client and/or regulatory agency, when approval is required. Record of approval is retained in the project record.

All subcontractor laboratories must maintain a quality management system like ENV and that complies with ISO/IEC 17025 and the TNI Standard(s).

ENV also evaluates and pre-qualifies subcontractors as part of the company's vendor qualification program. The complete list of approved vendors is maintained by the corporate procurement department and is made available to all ENV locations. Pre-qualification of a subcontractor does not negate the requirement for the placing laboratory to verify the capability, capacity, and resources of any selected subcontractor on a project-specific basis to confirm the subcontractor can meet the client's needs.

For both subcontracting and in-network work transfer, the project specifications are always communicated to the subcontractor or the in-network laboratory by the project manager so that the laboratory performing the work is aware of and understands these requirements.

The procedures for subcontracting are outlined in laboratory SOP *Sample Receiving* [AD SOP ME0013H].

4.6 Purchasing Services and Supplies

Vendors that provide services and supplies to the laboratory are prequalified to verify the vendor's capability to meet the needs of PAS. These needs include but are not limited to competitive pricing, capacity to fill purchase orders, quality of product, customer service, and business reputation and stability. The records of vendor evaluation and the list of approved vendors is maintained by the corporate procurement department.

The procedures for vendor qualification are specified in the corporate process for vendor qualification, however named.

The laboratory may purchase goods and services from any supplier on the approved vendor list.

The specifications (type, class, grade, tolerance, purity, etc.) of supplies, equipment, reagents, standard reference materials and other consumables used in the testing process are specified in SOPs. The SOP specifications are based on the governing requirements of the approved reference methods and any additional program driven regulatory specification, such as drinking water compliance. All requisitions for materials and consumables are approved by the department supervisor to confirm the purchase conforms with specified requirements. After approval the requisition is handled by the laboratory's designated purchasing agent. On receipt, the product is inspected and verified before use, when applicable.

The laboratory's procedure for the purchase of services and supplies is specified in laboratory SOP *Procurement of Laboratory Supplies, Services, and Equipment* [AD SOP ME0015U].

4.7 Customer Service

Project details and management is handled by the laboratory's customer service team. Each customer is assigned a Project Manager (PM) that is responsible for review of contract requirements and handling laboratory to customer communication about the project status.

4.7.1 Commitment to Meet Customer Expectations

The laboratory cooperates and works closely with our customers to ensure their needs are met and to establish their confidence in the laboratory's capability to meet their needs for analytical services and expectations for service.

The PM is the customer's primary point of contact for each analytical service request. The PM gathers information from the customer to ensure the details of their request are understood. After samples are received, the PM monitors the progress of the project and alerts

the customer of any delays or excursions that may adversely impact data usability. Laboratory supervisors are expected to keep the PM informed of project status and any delays or major issues, so that the PM can keep the client informed.

The laboratory encourages customers to visit the laboratory to learn more about the laboratory's capabilities, observe performance and to meet laboratory personnel.

ENV customers expect confidentiality. Laboratory personnel will not divulge or release information to a third party without proper authorization unless the information is required for litigation purposes. See Section 4.1.5.4 of this manual and policy COR-POL-0004 *Ethics Policy* for more information on the laboratory's policy for client confidentiality.

4.7.2 Customer Feedback

The laboratory actively seeks positive and negative feedback from customers through surveys and direct communication. Information from the client about their experience working with the laboratory and their satisfaction with work product is used to enhance processes and practices and to improve decision making. Customer feedback is communicated to laboratory management and corporate personnel in management reports and analyzed yearly during management review (See 4.15) to identify risk and opportunity. Corrective, preventive, or continuous improvement actions are taken based on nature of and/or feedback trends.

Also see sections 4.9, 4.10, 4.11, 4.12, 4.14, and 4.15 for more information about how customer feedback is managed by the laboratory and used to enhance the quality management system.

4.8 Complaints

Complaints provide opportunities to improve processes and build stronger working relationships with our clients.

The laboratory's complaint resolution process includes three steps. First, handle and resolve the complaint to mutual satisfaction. Second, perform corrective action to prevent recurrence (See 4.11). Third, record and track the complaint and use these records for risk and opportunity assessment and preventive action (See 4.12).

4.9 Nonconforming Work

4.9.1 Definition of Nonconforming Work

Nonconforming work is work that does not conform to customer requirements, standard specifications, laboratory policies and procedures, or that does not meet acceptance criteria.

The discovery of non-conforming work comes from various sources which include, but are not limited to:

- results of quality control samples and instrument calibrations;
- quality checks on consumables and materials;
- general observations of laboratory personnel;
- data review;
- proficiency testing;
- internal and external audits;

- complaints and feedback;
- management review and reports; and
- regulatory and certification and accreditation actions.

The way in which the laboratory handles nonconforming work depends on the significance and impact (risk) of the issue. Some issues may simply require correction, others may require investigation, corrective action (See 4.11) and/or data recall (See 4.16). When the laboratory releases data and test results associated with nonconforming QC and acceptance criteria, test results are qualified, or non-conformances are noted in the final analytical report to apprise the data user of the situation. (See 5.10)

Nonconforming work also includes unauthorized departure from laboratory policies, procedures and test methods. Authorized departures are explained in the following subsections. Situations that do not conform to these conditions are considered unauthorized departure(s).

4.9.1.1 Authorized Departure from SOP

An authorized departure from a test method SOP is one that has been reviewed and approved by the Department Manager, designated Acting Technical Manager for TNI for the discipline the SOP pertains to (Chemistry, Inorganic Chemistry, Microbiology), Quality Manager, or the General Manager. Management review is conducted to confirm the departure does not conflict with regulatory compliance requirements for which the data will be used or does not adversely affect data integrity. The departure may originate from client request or may be necessary to overcome a problem.

An authorized departure from administrative or process-oriented SOP is typically necessary to correct an error in the SOP. These departure requests are reviewed and pre-approved by the QA Manager.

Documentation of SOP departures and approval decisions are retained by the laboratory as evidence that the departure was authorized. When necessary, approved departures from test method SOPs are noted in the final test report to advise the data user of any ramification to data quality.

4.9.1.2 Authorized Departure from Test Methods (Method Modifications)

When test results are associated to a published reference test method, the laboratory's test method SOP must be consistent with the test method. If the test method is mandated for use by a specific regulatory program such as drinking water or wastewater or a certification or accreditation program, such as TNI/NELAC, the SOP must also comply with or include these requirements. If the procedures in the SOP are modified from the test method, these modifications must be clearly identified in the SOP. The conditions under which the laboratory may establish an SOP that is modified from these reference documents, and what is considered a modification are specified in ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification*.

Modifications that do not meet the requirements of this SOP (ENV-SOP-CORQ-0011) are unauthorized. Client requests to deviate from the test method are handled

as client requests to depart from the test method SOP since it is the SOP that the laboratory follows when performing work.

4.9.1.3 Stop Work Authority

Stop Work Authority provides laboratory personnel with the responsibility and obligation to stop work when there is a perceived unsafe condition or behavior that may result in an unwanted event.

All laboratory and corporate personnel have the authority to stop work when needed to preserve data integrity or safety of workers.

Once a stop work order has been initiated and the reason for doing so is confirmed valid; laboratory management is responsible for immediate correction and corrective action (see section 4.11) before resumption of work.

4.10 Continuous Improvement

The laboratory's quality management system is designed to achieve continuous improvement through the implementation of the quality policy and objectives outlined in this manual. Information about the laboratory's activities and performance is gained from many sources such as customer feedback, audits, QC, trend analysis, business analytics, management reports, proficiency testing, and management systems review. This information is subsequently used during the laboratory's corrective action (see section 4.11) and preventive action (see section 4.12) processes and during annual review of the management system (see section 4.15) to establish goals and objectives for improvement.

ENV also promotes a continuous improvement culture based on the principles of lean manufacturing. These principles include 3P (Process, Productivity, Performance) and Kaizen. 3P is a platform used by Pace to share best practices and standardization across the network to achieve operational excellence. Kaizen is a team-based process used to implement tools and philosophies of lean to reduce waste and achieve flow with the purpose of improving both external and internal customer satisfaction.

4.11 Corrective Action

Corrective action is a process used to eliminate the cause of a detected nonconformity. It is not the same as a correction. A correction is an action taken to fix an immediate problem. The goal of the corrective action process is to find the underlying cause(s) of the problem and to put in place fixes to prevent the problem from happening again. The corrective action process, referred to as CAPA by ENV, is one of the most effective tools used by the laboratory to prevent nonconforming work, identify risk and opportunity, and improve service to our customers.

The laboratory has two general processes for corrective action:

The process used for actions taken in response to day to day quality control (QC) and acceptance criteria exceptions (nonconformance) that occur during the day to day testing process are called corrections. These events do not usually include formal methods for cause analysis; instead the reason for the failure is investigated through troubleshooting or other measures. Required actions for correction of routine nonconformance is specified in laboratory SOPs. When corrective action is not taken, cannot be taken, or is not successful, test results associated with the nonconforming work are qualified in the final test report. Documentation of the nonconformance and corrective action taken is documented in the analytical record.

A 7-stage corrective action process is used when there is a problem or departure from the quality management system, technical activities, or when the extent of a single problem has significant impact on data, regulatory compliance or customer needs. These problems are identified through various activities such as but not limited to: quality control trends, internal and external audits, management review, customer feedback, and general observation.

The laboratory's 7 Stage CAPA Process includes:

1) Identification and Containment
2) Evaluation
3) Investigation
4) Cause Analysis
5) Action Plan
6) Implementation
7) Follow Up and Effectiveness Review

The 7 stage CAPA process may be initiated by any employee. Once the process is initiated it is overseen and coordinated by laboratory management. The CAPA process is documented using a software-based workflow process called Qualtrax. The Qualtrax CAPA record includes tracking information, dates, individuals involved, those responsible for action plan implementation and follow-up, and timelines and due dates.

ENV's procedures for corrective action, are specified in corporate SOP ENV-SOP-CORQ-0018, *Procedure for Corrective and Preventive Action.* Additional explanation about certain aspects of the laboratory's corrective action process are outlined in the next three subsections.

4.11.1 Cause Analysis

Cause analysis is the process of investigation used by the laboratory to identify the underlying cause(s) of the problem. Once causal factors are identified, ways to mitigate the causal factors are reviewed and corrective action(s) most likely to eliminate the problem are selected.

The laboratory uses different methods to conduct this analysis. The most common approach is 5-Why, but fishbone diagrams, or even brainstorming may be appropriate depending on the situation. The method used is documented in the CAPA record.

4.11.2 Effectiveness Review

Monitoring corrective actions for effectiveness is an activity shared by laboratory supervisors and quality assurance personnel. Effectiveness means the actions taken were sustainable and appropriate. Sustainable means the change is still in place. Appropriate means the action(s) taken prevented recurrence of the problem since the time corrective action was taken.

The timeframe in which effectiveness review takes place depends on the event and is recorded in the CAPA record with any addition actions that need to be taken.

Corrective action trends are also monitored by laboratory management and used to identify opportunities for preventive action or to gain lessons learned when actions taken were not adequate to solve the problem. See Section 4.12 (Preventive Action) and 4.15 (Management Review) for more information.

4.11.3 Additional Audits

When non-conformities or other problems cast doubt on compliance with the laboratory's policies, procedures, or compliance to regulatory requirements; the quality manager schedules a special audit of the area of activity in accordance with Section 4.14.1 as soon as possible. These special audits are used to determine the scope of the problem and to provide information for the CAPA process. Additional full-scale audits are done when a serious issue or risk to the laboratory's business is identified.

4.12 Preventive Action

Preventive action is an action taken to eliminate the cause of a potential nonconformity and to achieve improvement. Preventive action is a forward-thinking process designed to prevent problems opposed to reacting to them (corrective action).

Some examples of preventative action include, but are not limited to:

- Scheduled instrument maintenance (Preventative maintenance)
- Addition of Staff and Equipment
- Professional Development Activities
- Implementation of New Technology

The laboratory looks for opportunities for preventive action from a variety of sources including but not limited to: employee idea's, customer feedback, business partners input, trend analysis, business analytics, management reviews, proficiency testing results, lean management events, and risk-benefit analysis.

Laboratory management evaluates the success of preventive actions taken in any given year during annual management review. See Section 4.15 for more information.

4.12.1 Change Management

Preventive actions may sometimes result in significant changes to processes and procedures used by the laboratory. Laboratory management evaluates the risks and benefits of change and includes in its implementation of change process, actions to minimize or eliminate any risk. The types of changes for which risk are considered and managed include: infrastructure change, change in analytical service offerings, certification or accreditation status, instrumentation, LIMS changes, and changes in key personnel.

4.13 Control of Records

A record is a piece of evidence about the past, especially an account of an act or occurrence kept in writing or some other permanent form. Laboratory records document laboratory activities and provide evidence of conformity to the requirements established in the quality management system. These records may be hardcopy or electronic on any form of media.

4.13.1 General Requirements

4.13.1.1 Procedure

The requirements for control of records is specified in corporate policy ENV-POL-CORQ-0013 *Record Management*. The procedure used to implement the policy is described in laboratory SOP *Document and Record Control* [QA SOP ME001HX].

The policy is established to assure quality and technical records are identified, retained, indexed, and filed to allow for retrieval during the entire retention time frame. During storage, records are kept secure and protected from deterioration. At the end of the retention time, the records are disposed of properly in order to maintain client confidentiality and to protect the interests of the company.

In general, laboratory records fall into three categories: quality, technical, and administrative.

Examples of each are provided in the following table:

Record Type	Includes Records of:
Quality	Document Types listed in SOP ENV-SOP-CORQ-0015 Audits: Internal and External Certificates and Scopes of Accreditation Corrective & Preventive Action Management Review Data Investigations Method Validation Instrument Verification Training Records
Technical	Raw Data Logbooks Certificates of Traceability Analytical Record Test Reports & Project Information Technical Training Records & Demonstration of Capability
Administrative	Personnel Records Finance/Business

4.13.1.2 Record Legibility and Storage

Records are designed to be legible and to clearly identify the information recorded. Manual entries are made in indelible ink; automated entries are in a typeface and of sufficient resolution to be read. The records identify laboratory personnel that performed the activity or entered the information. Records are archived and stored in a way that they are retrievable. Access to archived records is controlled and managed.

For records stored electronically, the capability to restore or retrieve the electronic record is maintained for the entire retention period. Hardcopy records are filed and stored in a suitable environment to protect from damage, deterioration, or loss. Hardcopy records may be scanned to PDF for retention. Scanned records must be checked against the hardcopy to verify the scan is complete and legible.

Administrative records are kept for a minimum of 5 years and technical and quality records are kept for 10 years unless otherwise specified by the client or regulatory program.

The date from which retention time is calculated depends on the record. In general, the retention time of technical records of original observation and measurement is calculated from the date the record is created. If the technical record is kept in a chronological logbook, the date of retention may be calculated from the date the logbook is archived. The retention time of test reports and project records, which are considered technical records, is calculated from the date the test report was issued. The retention time of quality records is usually calculated from the date the record is archived.

Refer to the record management policy and the laboratory SOP for more information.

4.13.1.3 Security

The laboratory is a secure facility and access to records is restricted to laboratory personnel.

4.13.1.4 Electronic Records

The data systems used to store electronic records is backed up in accordance with laboratory SOP *Laboratory Information Management System* (LIMS) [AD Sop ME00161]. Access to archived records stored electronically is maintained by personnel responsible for management of the electronic system.

4.13.1.5 Electronic Signature Policy

Work done by ENV locations include activities that require the application of a signature. Some of this work product is in electronic format and signatures are applied electronically.

The Electronic Signatures in Global and National Commerce Act (E-Sign Act) clarifies that electronic signatures are legally valid and enforceable under United States law.

ENV's policy for use and application of electronic signatures is specified in corporate policy ENV-POL-CORQ-0014 *Electronic Signature Policy*.

All employees of ENV, including temporary and contract personnel, must sign an Electronic Signature Agreement to acknowledge that they understand and accept that work activities performed by them may be authenticated with application of an electronic signature and that electronic signature has the same validity as a handwritten signature. Their signed agreement also confirms the individual has read and understands the policy and agrees to abide by the requirements for use of electronic signature stated in the policy.

4.13.2 Technical Records

In addition to the requirements specified in subsections 4.13.1.1 through 4.13.1.5, the requirements in the following subsections also apply to technical records.

4.13.2.1 Description

Technical records are the accumulation of data and information generated from the analytical process. These records may include forms, worksheets, workbooks, checklists, notes, raw data, calibration records, final test reports, and project record. The accumulated record essentially needs to provide adequate detail to historically reconstruct the process and identify the personnel that performed the tasks associated with a test result.

4.13.2.2 Real Time Recordkeeping

Personnel are instructed and expected to always record observations, data, and calculations at the time they are made. Laboratory managers are responsible to assure that data entries, whether made electronically or on hardcopy, are identifiable to the task.

4.13.2.3 Error Correction

Errors in records must never be erased, deleted or made illegible. Use of correction fluid, such as white-out is prohibited. In hardcopy records, the error is corrected by a single strike through the original entry and the new entry recorded alongside or footnoted to allow for readability. Corrections are initialed and dated by the person making the correction. If the correction is not self-explanatory, a reason for the correction is recorded.

For electronic records, equivalent measures of error correction or traceability of changes made is kept. For example, audit trails provide records of change.

Maintenance of proper practices for error correction is monitored through the tiered data review process described in Section 5.9.3. Laboratory records are reviewed throughout the data review process. Individuals performing these reviews flag errors that are not properly corrected and bring these to the attention of the department manager or supervisor of the work area in which the record was generated so that the problem may be addressed and corrected with the individual(s) that did not make the correction properly.

4.14 Audits

The laboratory performs internal systems and technical audits to assess implementation of the QMS and compliance to this manual and to procedures, such as policy, SOP and SWI. Since the processes in this manual are based on the relevant quality system standards and regulatory and accreditation/certification program requirements the laboratory provides services for, the internal audits also assess on-going compliance to these programs.

The laboratory is also audited by external parties such as regulatory agencies, customers, consultants and non-government assessment bodies (NGAB).

Information from internal and external audits is used by laboratory management to address compliance concerns and opportunities where improvement will increase the reliability of data.

Deficiencies, observations and recommendations from audits are managed by the local QM using the laboratory's formal CAPA process. See Section 4.11 for more information.

4.14.1 Internal Audit

The laboratory's internal audit program is managed by the local QM in accordance with an audit plan established at the beginning of each calendar year. The schedule is prepared to assure that all areas of the laboratory are reviewed over the course of the year. Conformance to the schedule is reported to both laboratory management and corporate quality personnel in a monthly QA report prepared by the quality manager.

Although the local QM creates the audit schedule, it is the shared responsibility of local management to assure the schedule is maintained. Laboratory supervisors cooperate with the quality personnel to provide the auditors with complete access to the work area, personnel, and records needed.

Internal audits are performed by personnel approved by the quality manager. In general, personnel may not audit their own activities unless it can be demonstrated that an effective and objective audit will be carried out. The auditor must be trained, qualified, and familiar enough with the objectives, principles, and procedures of laboratory operations to be able to perform a thorough and effective evaluation.

The laboratory's internal audit program ensures daily practice is consistent with laboratory's SOPs and to verify SOPs are compliant with policy and procedures. Test reports are audited to verify the final product is consistent with customer/project requirements, the work was carried out in accordance with policy and SOPs, the SOP complies with the cited reference method, test results are accurate, and of known and documented quality and properly qualified, when necessary.

Special audits are performed ad hoc to follow up on a specific issue such as a client complaint, negative feedback, concerns of data integrity or ethics, or a problem identified through other audits. Special audits may be scheduled or unscheduled. Unscheduled internal audits are conducted whenever doubts are cast on the laboratory's compliance with regulatory requirements or its own policies and procedures. These unscheduled internal audits may be conducted at any time and may be performed without an announcement to laboratory personnel.

When observations and findings from any audit (internal or external) cast doubt on the validity of the laboratory's testing results, the laboratory takes immediate action to initiate investigate the problem and take corrective action. (Also see 4.11 and 4.16)

The laboratory's internal audit program and auditing procedures are further described in laboratory SOP *Internal Audits* [QA SOP ME0015T].

4.14.1.1 Corporate Compliance Audit

ENV locations are also periodically audited by corporate quality personnel to assess the location's compliance to ENV's quality management program and to evaluate the effectiveness of implementation of the policies and procedures that make up the quality management system. The purpose of the compliance audit is to identify risks and opportunities and to assist laboratory management achieve the goals and objectives of the company's quality program.

4.15 Management Review

The management team formally reviews the management system of each location under their purview on an annual basis to assess for on-going suitability and effectiveness and to establish goals, objectives, and action plans for the upcoming year.

The process and procedures used to conduct this review are outlined in corporate SOP ENV-SOP-CORQ-0005 *Management Review*.

At a minimum, the following topics are reviewed and discussed:

- The on-going suitability of policies and procedures including EHS and waste management;
- Reports from managerial and supervisory personnel including topics discussed at regular management meetings held throughout the year;
- The outcome of recent internal audits;
- Corrective and preventive actions;
- Assessments by external bodies;
- The results of interlaboratory comparisons or proficiency tests;
- Changes in the volume and type of the work;
- Customer and personnel feedback, including complaints;
- Effectiveness of improvements / preventive actions made since last review;
- Internal and external issues of relevance and risk identification;
- A review of the status of actions from prior management reviews; and
- Other relevant factors, such as quality control activities, resources, and staff training.

The discussion and results of this review are documented in a formal report prepared by laboratory management. This report includes a determination of the effectiveness of the management system and its processes; goals and objectives for improvements in the coming year with timelines and responsibilities, and any other need for change.

Goals and action items from annual management systems review are shared with local employees and with corporate management to highlight focus areas for improvement in addition to areas in which the laboratory has excelled.

4.16 Data Integrity

ENV's procedures for the investigation and response to events that may affect data integrity are described in the corporate SOPs for data inquiries and data recall and corrective and preventive action, however named.

Customers whose data are affected by these events are notified in a timely manner, usually within 30 days after the impact of the problem is understood. Some accreditation programs also require notification to the accreditation body (AB) within a certain timeframe from date of discovery when

the underlying cause of the issue impacts accreditation. The laboratory follows any program or project specific client notification requirements for notification, when applicable.

5.0 TECHNICAL REQUIREMENTS

5.1 General

Many factors contribute to the correctness and reliability of the technical work performed by the laboratory. These factors fall under these general categories:

- Human Performance
- Facility and Environmental Conditions
- Test Method Performance and Validation
- Measurement Traceability
- Handling of Samples

The impact of each of these factors varies based on the type of work performed. To minimize negative effects from each of these factors, the laboratory accounts for the contribution from each of these categories when developing test method and process (administrative) SOPs, evaluating personnel qualifications and competence, and in the selection of equipment and supplies used.

5.2 Personnel

5.2.1 Personnel Qualifications

The laboratory's program for personnel management is structured to ensure personnel are selected, qualified, and competent to perform the roles and responsibilities of their position based on education, experience, and training.

Qualifications, duties, responsibilities, and authorities of each position are specified in job descriptions maintained by corporate HR (See Section 5.2.4). These job descriptions provide the general basis for the selection of personnel for hire and are used by the laboratory to communicate to personnel the duties, responsibilities, and authorities of their position.

The term "personnel" refers to individuals employed by the laboratory directly as full-time, part-time, or temporary, and individuals employed by the laboratory by contract, such as through an employment agency. The term "personnel" is used interchangeably with the term "employee" throughout this manual. For purposes of this manual, these terms are equivalent.

The personnel management program is structured to establish and maintain records for each of the following:

- Selection of personnel;
- Training of personnel;
- Supervision of personnel;
- Authorization of personnel; and
- Monitoring Competence of personnel.

5.2.1.1 Competence

Competence is the ability to apply a skill or series of skills to complete a task or series of tasks correctly within defined expectations.

Competence for technical personnel authorized by ENV to provide opinion and interpretation of data to customers also includes the demonstrated ability to:

- Apply knowledge, experience, and skills needed to safely and properly use equipment, instrumentation, and materials required to carry out testing and other work activities in accordance with manufacturer specifications and laboratory SOPs;
- Understand and apply knowledge of general regulatory requirements necessary to achieve regulatory compliance in work product; and
- Understand the significance of departures and deviations from procedure that may occur during the analytical testing process and the capability and initiative to troubleshoot and correct the problem, document the situation and decision-making process, and to properly qualify the data and analytical results.

The laboratory's requirements for the competence of personnel (education, qualification, work experience, technical skills, and responsibilities) are specified in job descriptions created by management and kept by human resources (HR). The job description provides the basis for the selection of personnel for each position.

An employee is considered competent when he/she has completed required training.

The policies and standard operating procedures (SOPs) for the following topics are established by management as minimum required training for all personnel:

- Ethics and Data Integrity
- Quality Manual
- Safety Manual
- Quality Management System
- Technical Process and Procedure relevant to their job tasks
- Successful Demonstration of Capability (DOC) – Analytical Personnel Only

Personnel are initially authorized competent to independently carry out their assigned duties when required training is complete and documented.

Records of required training and qualification provide the record of competence for the individual. Qualification records may include but are not limited to diploma, transcripts, and curriculum vitae (CV).

The on-going competence of each employee is monitored by laboratory management through on-the-job performance. Analytical employees are also required to successfully complete another demonstration of capability for each test method performed on an annual basis.

5.2.2 Training (Required)

ENV's training requirements are outlined in policies COR-POL-0023 *Mandatory Training Policy,* COR-POL-0004 *Ethics Policy*, and laboratory SOP *Employee Orientation and Training* [QA SOP ME003LN].

5.2.2.1 Required Training

The laboratory's training program includes these elements:

- Scheduling of Required Training
- Execution of Required Training
- Documentation and Tracking of Required Training
- Evaluation of Training Effectiveness

Required training is delivered using various methods that incorporate techniques that appeal to the main learning styles: visual, aural, linguistic, and kinesthetic. Techniques include, on-the-job, instructor-led, self-study, eLearning, and blended.

The employee's direct supervisor is responsible for oversight of completion of the employee's required training and for providing adequate time to the employee to complete training assignments. Both the supervisor and employee are responsible to make sure the employee's training status and training records for required training are current and complete.

The status of completion of required training is monitored by the local QM, who provides the status to the GM at least monthly or more frequently, if necessary, to ensure required training for personnel is complete and up to date.

The following subsections describe the elements of ENV's required training program.

5.2.2.1.1 New Hire Training

New hire training requirements apply to new personnel and to existing employees starting in a new position or different work area.

Required new hire training includes each of the following:

- Ethics and Data Integrity (See 5.2.2.1.3)
- Quality Manual / Quality Management System (See 5.2.2.1.4)
- Safety Manual and any training requirements specified in the manual.
- Policies & SOPs relevant to their job tasks
- Technical personnel that test samples must also successfully complete an initial demonstration of capability (IDOC) for the test methods performed before independently testing customer samples. (See 5.2.2.1.5). Independent testing means handling of client samples without direct supervision of the work activity by the supervisor or a qualified trainer.

All required training must be current and complete before the employee is authorized to work independently. Until then, the employee's direct supervisor is responsible for review and acceptance of the employee's work product.

5.2.2.1.2 On-Going Training

Personnel receive on-going training in each of the following topics:

- Ethics and Data Integrity (See 5.2.2.1.3)
- Quality Manual / Quality Management System (See 5.2.2.1.4)
- Safety Training
- Changes to Policies & SOPs
- Technical employees that carry out testing must also successfully complete on-going demonstration of capability (CDOC) for all test methods performed on an annual basis. (See 5.2.2.1.5)

Personnel are expected to maintain their DOCs current and complete and to complete training assignments in a timely manner.

5.2.2.1.3 Ethics and Data Integrity Training

Data integrity training is provided to all new personnel and refresher data integrity training is provided to all employees on an annual basis. Personnel are required to acknowledge they understand that any infractions of the laboratory data integrity procedures will result in a detailed investigation that could lead to very serious consequences including immediate termination, debarment, or civil/criminal prosecution.

Completion of data integrity training is documented by employee signature to provide evidence that the employee has participated in training on this topic and understand their obligations related to data integrity.

The following topics and activities are covered:

- Policy for honesty and full disclosure in all analytical reporting;
- Prohibited Practices;
- How and when to report data integrity issues;
- Record keeping. The training emphasizes the importance of proper written documentation on the part of the analyst with respect to those cases where analytical data may be useful, but are in one sense or another partially nonconforming;
- Training Program, including discussion regarding all data integrity procedures;
- Data integrity training documentation;

- In-depth procedures for data monitoring; and
- Specific examples of breaches of ethical behavior such as improper data manipulations, adjustments of instrument time clocks, and inappropriate changes in concentrations of standards.

All PAS personnel, including contract and temporary, are required to sign an "Attestation of Ethics and Confidentiality" at the time of employment and during annual refresher training. This document clearly identifies inappropriate and questionable behavior. Violations of this document result in serious consequences, including prosecution and termination, if necessary.

Also see SOP-ENV-COR-POL-0004 *Ethics Policy* for more information.

5.2.2.1.4 Management System Document Training

The Quality Manual and ENV manuals, policies, and SOPs are the documents used by regulatory bodies and PAS customers to verify the laboratory's capability, competency, and compliance with their requirements and expectations.

In addition to on-the-job training, employees must have a signed Read and Acknowledgement Statement (R&A) on record for the laboratory quality manual, and the policies and SOPs relating to his/her job responsibilities. This statement, whether signed by the employee electronically or by wet signature, confirms that the employee has received, read, and understands the content of the document, that the employee agrees to follow the document when carrying out their work tasks; and the employee understands that unauthorized change to procedures in an SOP is not allowed except in accordance with the SOP departure policy (See 4. 9.1).

See SOP ENV-CORQ-0016 *Standard Operating Procedures and Standard Work Instructions* for more information.

5.2.2.1.5 Demonstration of Capability (DOC)

Demonstration of capability is based on the employee's capability to achieve acceptable precision and accuracy for each analyte reported by the laboratory for the test method using the laboratory's test method SOP.

Technical employees must complete an initial demonstration of capability (IDOC) prior to independent work on client samples analyzed by the test methods they perform. After successful IDOC, the employee must demonstrate continued proficiency (CDOC) for the test method on an annual basis. If more than a year has passed since the employee last performed the method; then capability must be re-established with an IDOC.

Records of IDOC and CDOC are kept in the employee's training file.

5.2.2.2 Effectiveness of Training

The results of the performance measures used to identify training needs are the same measures used by the laboratory to measure effectiveness of the training program. Improvement in key performance measures suggest the training program is successful (See 5.2.2.1).

Effectiveness of individual employee training is measured by their demonstrated ability to comprehend the training material and apply knowledge and skills gained to their job task. Measurements include but are not limited to:

- Testing of the employee's knowledge of the quality management system, policies, and technical and administrative procedures through various mechanisms, such as quizzes, observation, and interviews.
- Demonstrated ability to convey information correctly and factually in written and verbal communication to internal and external parties.
- Demonstrated ability to carry out tasks in accordance with SOPs and other work instructions.
- Demonstrated ability to make sound decisions based on guidance and information available.
- Demonstrated initiative to seek help or guidance when the employee is unsure of how to proceed.

5.2.2.3 Supplemental Learning

Supplemental learning objectives are established for newly hired personnel to aid in their development of administrative and technical skills. These learning objectives and materials, referred to as Learning Plans (LP), are created and maintained by ENV's 3P program and managed by the employee's direct supervisor.

In addition to LPs, PAS maintains a wide variety of supplemental learning courses that are made available to all PAS employees for professional development. These learning materials, maintained by PAS's corporate training personnel, are accessed via the company's employee portal, PaceConnect. The learning may be self-initiated based on an employee's interest or may be assigned to the employee at the discretion of management as professional development as part of an employee's annual goals. Supplemental learning courses and learning plan activities are not prerequisites for competency (Section 5.2.1.1) and are not part of the required QMS training specified in Section 5.2.2.1.

5.2.3 Personnel Supervision

Every employee is assigned a direct supervisor, however named, who is responsible for their supervision. Supervision is the set of activities carried out by the supervisor to oversee the progress and productivity of the employees that report to them.

General supervisory responsibilities may include but are not limited to:

- Hiring Employees
- Training Employees
- Performance Management
- Development, oversight, and execution of personnel training plans
- Monitoring personnel work product to assure the work is carried out in accordance with this quality manual, policies, SOPs, and other documents that support the quality management system.

5.2.4 Job Descriptions

Job Descriptions that define the required education, qualifications, experience, skills, roles and responsibilities, and reporting relationships for each PAS position are established by top management and kept by corporate HR. PAS laboratories use these job descriptions as the source of positions and job titles for the laboratory. The job descriptions apply to employees who are directly employed by PAS, part-time, temporary, technical and administrative and by those that are under contract with PAS through other means.

The job descriptions include the education, expertise, and experience required for the position and the responsibilities and duties, including any supervisory or managerial duties assigned to the position.

5.2.5 Authorization of Technical Personnel

Laboratory management authorizes technical personnel to perform the technical aspects of their position after it has been verified that the employee meets the qualifications for the position, has successfully completed required training (Section 5.2.2.1), and the employee has completed initial demonstrated capability (Section 5.2.2.1.5). After initial authorization, technical personnel are expected to maintain a current and complete training record, demonstrate on-going capability at least annually for each test method performed, and produce reliable results through accurate analysis of certified reference materials, proficiency testing samples, and/or routine quality control samples in order to remain authorized to continue to perform their duties.

Records to support authorization including, education, experience, training, and other evaluations are kept by the laboratory.

5.3 Accommodations and Facilities

5.3.1 Facilities

The laboratory is designed to support the correct performance of procedures and to not adversely affect measurement integrity or safety. Access to the laboratory is controlled by various measures, such as card access, locked doors, main entry. Visitors to the laboratory are required to sign-in and to be escorted by laboratory personnel during their visit. A visitor is any person that is not an employee of the laboratory.

5.3.2 Environmental Conditions

The laboratory is equipped with energy sources, lighting, heating, and ventilation necessary to facilitate proper performance of calibrations and tests. The laboratory ensures that

housekeeping, electromagnetic interference, humidity, line voltage, temperature, sound and vibration levels are appropriately controlled to ensure the integrity of specific measurement results and to prevent adverse effects on accuracy or increases in the uncertainty of each measurement.

Environmental conditions are monitored, controlled, and recorded as required by the relevant specifications, methods, and procedures. Laboratory operations are stopped if it is discovered that the laboratory's environmental conditions jeopardize the analytical results.

5.3.3 Separation of Incompatible Activities

The layout and infrastructure of each work area including air handling systems, power supplies, and gas supplies of each laboratory work area is specifically designed for the type of analytical activity performed. Effective separation between incompatible work activities is maintained. For example, sample storage, preparation, and chemical handling for volatile organic analysis (VOA) is kept separate from semi-volatile organic (SVOA).

The laboratory separates samples known or suspected to contain high concentration of analytes from other samples to avoid the possibility for cross-contamination. If contamination is found, the source of contamination is investigated and resolved in accordance with laboratory SOPs.

5.3.4 Laboratory Security

Security is maintained by controlled access to the building and by surveillance of work areas by authorized personnel. Access is controlled to each area depending on the required personnel, the sensitivity of the operations performed, and possible safety concerns. The main entrance is kept unlocked during normal business hours for visitors and is continuously monitored by laboratory staff. All visitors must sign a visitor's log, and a staff member must accompany them during the duration of their stay.

5.3.5 Good Housekeeping

The laboratory ensures good housekeeping practices in work areas to maintain a standard of cleanliness necessary for analytical integrity and personnel health and safety. Minimally, these measures include regular cleaning of the work area. Where necessary, areas are periodically monitored to detect and resolve specific contamination and/or possible safety issues.

5.4 Test Methods

5.4.1 General Requirements

The laboratory uses test methods and procedures that are appropriate for the scope of analytical services the laboratory offers.

Instructions on the use and operation of equipment and sample handling, preparation, and analysis of samples are provided in SOPs. The instructions in SOPs may be supplemented with other documents including but not limited to, standard work instructions (SWI), manuals, guides, project documents and reference documents.

These documents are managed using the procedures described in SOP ENV-SOP-CORQ-0015 *Document Management and Control* and SOP ENV-SOP-CORQ-0016 *Standard Operating Procedures and Standard Work Instructions.*

5.4.2 Method Selection

The test methods and protocols used by the laboratory are selected to meet the needs of the customer, are appropriate for the item tested and intended use of the data, and to conform with regulatory requirements when regulatory requirements apply.

In general, the test methods offered are industry accepted methods published by international, regional, or national standards. The laboratory bases its procedure on the latest approved edition of a method unless it is not appropriate or possible to do so, or unless regulatory requirements specify otherwise.

The laboratory confirms that it can perform the test method and achieve desired outcome before analyzing samples (see section 5.4.5). If there is a change in the published analytical method, then the confirmation is repeated.

When a customer does not specify the test method(s) to be used, the laboratory may suggest test methods that are appropriate for the intended use of the data and the type of samples to be tested. The laboratory will also inform customers when test methods requested are considered inappropriate for their purpose and/or out of date. This discourse takes place during review of analytical service requests (See Section 4.4).

5.4.3 Laboratory Developed Methods

A laboratory developed method is a method developed from scratch (no published source method), a procedure that modifies the chemistry from the source method, or a procedure that exceeds the scope and application of the source method.

Laboratory developed methods must be validated prior to use (see section 5.4.5) and the procedure documented in a test method SOP.

The requirements for non-standard methods (Section 5.4.4) also apply to laboratory developed methods.

5.4.4 Non-standard Methods

A non-standard method is a method that is not published or approved for use by conventional industry standards for the intended purpose of the data. Non-standard methods must be validated prior to use (see section 5.4.5) and the procedure developed and documented in a test method SOP.

At a minimum, the following information must be included in the procedure:

- Title / Identification of Method;
- Scope and Application;
- Description of the type of item to be analyzed;
- Parameters or quantities and ranges to be determined;
- Apparatus and equipment, including technical performance requirements;
- Reference standards and reference materials required;
- Environmental conditions required and any stabilization period needed; and
- Description of the procedure, including:

- Affixing identification marks, handling, transporting, storing and preparing of items;
- Checks to be made before the work is started;
- Verifying equipment function and, where required, calibrating and/or adjusting the equipment before each use;
- Method of recording the observations and results;
- Any safety measures to be observed;
- Criteria and/or requirements for approval/rejection;
- Data to be recorded and method of analysis and presentation; and
- Uncertainty or procedure for estimating uncertainty.

Use of a non-standard method for testing must be agreed upon with the customer. The agreement, which is retained by the laboratory in the project record, must include the specifications of the client's requirements, the purpose of testing, and their authorization for use of the non-standard method.

5.4.5 Method Validation

5.4.5.1 Validation Description

Validation is the process of conformation and the provision of objective evidence that the stated requirements for a specific method/procedure are fulfilled.

The laboratory's requirements and procedures for method validation are outlined in SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification.*

5.4.5.2 Validation Summary

All test methods offered by the laboratory are validated before use to confirm the procedure works and the data and results achieved meet the goals for the method and repeated when there are major changes to the laboratory procedure.

Results of validation are retained are kept in accordance with method validation SOP and the corporate policy ENV-CORQ-POL-0013 *Record Management*.

5.4.5.3 Validation of Customer Need

The validation process includes review of accuracy, precision, sensitivity, selectivity, linearity, repeatability, reproducibility, robustness, and cross-sensitivity of the procedure against general customer needs to ensure the laboratory's procedure will meet those needs.

The following subsections highlight some of these concepts:

5.4.5.3.1 Accuracy

Accuracy is the degree to which the result of a measurement, calculation, or specification conforms to the correct value or a standard. When the result recovers within a range from the known

value (control limit); the result generated using the laboratory's test method SOP is considered accurate.

5.4.5.3.2 Precision

Precision refers to the closeness of two or more measurements to each other. It is generally measured by calculating the relative percent difference (RPD) or relative standard deviation (RSD) from results of separate analysis of the same sample. Precision provides information about repeatability, reproducibility, and robustness of the laboratory's procedure.

5.4.5.3.3 Limits of Detection (LOD) (Chemistry)

The LOD is the minimum result which can be reliably discriminated from a blank with a predetermined confidence level. The LOD establishes the limit of method sensitivity and is also known as the detection limit (DL) or the method detection limit (MDL).

Values below the LOD cannot be reliably measured and are not reported by the laboratory unless otherwise specified by regulatory program or test method.

The LOD is established during method validation and after major changes to the analytical system or procedure that affect sensitivity are made.

5.4.5.3.4 Limits of Quantitation (LOQ) and Reporting Limit (RL)

The LOQ is the minimum level, concentration, or quantity of a target analyte that can be reported with a specified degree of confidence. The LOQ is established at the same time as the LOD.

The LLOQ is the value of the lowest calibration standard included in the calibration curve. The LLOQ establishes the lower limit of quantitation.

The LOQ and LLOQ represent quantitative sensitivity of the test method.

- The LOQ must always be equal to or greater than the LLOQ and the LLOQ must always be greater than the LOD.
- Any reported value (detect or non-detect) less than the LLOQ is a qualitative value.

The RL is the value to which the presence of a target analyte is reported as detected or not detected. The RL is project-defined based on project data quality objectives (DQO). In the absence of project specific requirements, the RL is usually set to the LOQ or the LLOQ.

The laboratory's procedures for LOD/LOQ determination is detailed in laboratory SOP *Method Validation* [QA Policy ME003BF].

The local SOP is based on guidance provided by corporate quality and must comply with 40CFR 136 Appendix B and the TNI Standard.

5.4.5.3.5 Linearity

Linearity is a mathematical concept applied to calibration models that employ multiple points to establish a calibration range used for quantitative analysis. Linearity is measured differently based on the calibration model. In general, if linearity is demonstrated then the slope of the response of standards are sufficiently close to one another. The accuracy of the linear regression and non-linear curves is verified by checking percent error or relative standard error (RSE), which is the process of refitting calibration data back to the model to determine if the results are accurate. For linear curves that use average calibration or response factor, error is measured by relative standard difference (RSD).

Linearity also establishes the range of quantitation for the test method used which directly impacts the sensitivity of the test method and uncertainty in measurement results. As previously noted, the LLOQ establishes the lower limit of quantitation. Similarly, the upper range of linearity establishes the upper limit of quantitation. In general, results outside of this range are considered qualitative values. However, some inorganic methods allow for extension of the linear range above the upper limit of quantitation when accuracy at this value is verified.

Linearity can also be used to establish repeatability, reproducibility, and robustness of the laboratory's test method. When linearity is demonstrated using a specific calibration model during method validation, then use of this same calibration model to achieve linearity on a day to day basis confirms the laboratory's method is repeatable, reproducible, and robust.

5.4.5.3.6 Demonstration of Capability (DOC)

The DOC performed during method validation confirms that the procedure demonstrated acceptable precision and accuracy. The procedure used for DOC for method validation is the same as described in section 5.2.2.1.5 for demonstration of analyst capability.

5.4.6 Measurement Uncertainty

The laboratory provides an estimate of uncertainty in testing measurements when required or on client request. In general, the uncertainty of the test method is reflected in the control limits used to evaluate QC performance. (See 5.9.1.1.9). ISO/IEC supports this concept with language that reads when a well-recognized test method specifies limits to the values of the major source of uncertainty of measurement and specifies the form of presentation of

calculated results, the laboratory has satisfied the requirements on analytical uncertainty by following the test method and reporting instructions.

When measurement uncertainty cannot be satisfied through control limits, the laboratory will provide a reasonable estimation of uncertainty. A reasonable estimation is based on knowledge of method performance and previous experience. When estimating the analytical uncertainty, all uncertainty components which are of importance in the given situation are taken into account.

5.4.7 Control of Data

The laboratory has policies and processes in place to assure that reported data is free from calculation and transcription errors, that quality control is reviewed and evaluated before data is reported, and to address manual calculation and integration.

5.4.7.1 Calculations, Data Transfer, Reduction and Review

Whenever possible, calculations, transfer of data, and data reduction are performed using validated software programs (See 5.4.7.2).

If manual calculations are performed, the results of these calculations are verified during the data review process outlined in section 5.9.3.

5.4.7.1.1 Manual Integration

The laboratory's policy and procedures for manual integration are provided in corporate SOP ENV-SOP-CORQ-0006 *Manual Integration*.

This SOP includes the conditions under which manual integration is allowed and the requirements for documentation.

Required documentation of manual integration includes:

- complete audit trail to permit reconstruction of before and after results;
- identification of the analyst that performed the integration and the reason the integration was performed; and
- identification of the individual(s) that reviewed the integration and verified the integration was done and documented in compliance with the SOP.

5.4.7.2 Use of Computers and Automated Acquisition

Whenever possible the laboratory uses software and automation for the acquisition, processing, recording, reporting, storage, and/or retrieval of data.

Software applications developed by PAS are validated by corporate IT for adequacy before release for general use. Commercial off the shelf software is considered sufficiently validated when the laboratory follows the manufacturer or vendor's manual for set-up and use. Records of validation are kept by the corporate information technology (IT) group or by the local laboratory, whichever group performed the validation.

The laboratory's process for the protection of data stored in electronic systems include:

- Individual user names and passwords for Laboratory Information Management Systems (LIMS) and auxiliary systems used to store or process data.
- Employee Training in Computer Security Awareness
- Validation of spreadsheets used for calculations to verify formulas and logic yield correct results and protection of these cells to prevent unauthorized change.
- Operating system and file access safeguards
- Protection from Computer Viruses
- Regular system backup; and testing of retrieved data

The laboratory's process for software development and testing process includes:

- Verification the software application works as expected and is adequate for use and fulfills compliance requirements, such as the need to record date/time of data generation.
- Change control to assure requests for changes are reviewed and approved by management before the change is made.
- Communication channels to assure all staff are aware of changes made.
- Version Control and maintenance of historical records.

These procedures are detailed in laboratory SOPs *Laboratory Information Management System (LIMS)* [AD SOP ME00161] and *Document and Record Control* [QA SOP ME001HX].

5.5 Equipment

5.5.1 Availability of Equipment

The laboratory is furnished with all equipment and instrumentation necessary to correctly perform the tests offered in compliance with the specifications of the test method and to achieve the accuracy and sensitivity required.

5.5.2 Calibration

Equipment and instrumentation are checked prior to use to verify it performs within tolerance for its intended application.

Laboratory management is made aware of the status of equipment and instrumentation and any needs for either on a daily basis. This information is obtained during laboratory walkthroughs (LDM) that are conducted as part of the laboratory's lean program.

5.5.2.1 Support Equipment

The laboratory confirms support equipment is in proper working order and meets the specifications for general laboratory use prior to placement in service with

intermediate checks thereafter. Equipment that does not meet specifications is removed from service until repaired or replaced. Records of repair and maintenance activities are maintained.

Procedures used to carry out and record these checks are outlined laboratory in SOP *Equipment and Instrumentation* [QA SOP ME002JT].

5.5.2.2 Analytical Instruments

Analytical instruments are checked prior to placement in service in accordance with SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification.* After the initial service date, the calibration of instruments and verification calibration is performed in accordance with local test method SOPs.

The calibration procedures in the test method SOPs comply with the requirements for acceptable calibration practices outlined in corporate policy ENV-POL-CORQ-0005 *Acceptable Calibration Practices*, the reference methods, and any applicable regulatory or program requirements.

5.5.3 Equipment Use and Operation

Equipment is operated and maintained by laboratory personnel that are trained on the test method SOP. Up-to-date instructions and procedures for the use and maintenance of analytical equipment are included in SOPs and/or supplemental documents such as standard work instructions (SWI) or instrument manuals which are made readily accessible in the work area to all laboratory personnel.

5.5.4 Equipment Identification

The laboratory uniquely identifies equipment by serial number or any other unique ID system, when practical. The identifier is included in the equipment list maintained by the quality department.

5.5.5 Equipment Lists and Records

5.5.5.1 Equipment List

The laboratory maintains a master list of equipment that includes information about the equipment including a description, manufacturer, serial number, date placed in service, condition when received, identity, and the current location in the laboratory. The date of purchase is tracked by the procurement record. The equipment list(s) for each location covered by this manual is provided in Appendix E.

5.5.5.2 Equipment Records

In addition to the equipment list, the laboratory maintains records of equipment that include:

- Verification that equipment conforms with specifications.
- Calibration records including dates, results, acceptance criteria, and next calibration dates.
- Maintenance plan and records

- Records of damage, malfunction, or repair

The laboratory follows an equipment maintenance program designed to optimize performance and to prevent instrument failure which is described in laboratory SOP *Equipment and Instrumentation* [QA SOP ME002JT] or in individual test method SOPs.

The maintenance program includes routine maintenance activities which are performed as recommended by the manufacturer at the frequency recommended and non-routine maintenance, which is performed to resolve a specific problem such as degradation of peak resolution, shift in calibration relationship, loss of sensitivity, or repeat failure of instrument performance checks and quality control samples.

Maintenance is performed by laboratory personnel or by outside service providers.

All maintenance activities performed by laboratory personnel are recorded by the individual(s) that performed the activity at the time the maintenance was performed in an instrument maintenance log.

The maintenance record minimally includes the date of maintenance, the initials of the person(s) performing maintenance, a description of the activity performed, why (when the maintenance is non-routine), and the return to analytical control. When maintenance is performed by an external vendor, the laboratory staples the service record into hardcopy maintenance logs or scans the record for easy retrieval. The laboratory provides unrestricted access to instrument maintenance logs in order to promote good instrument maintenance and recordkeeping practices.

If an instrument must be moved, the laboratory will use safe practices for handling and transport to minimize damage and contamination.

5.5.6 Out of Service Protocol

Equipment that has been subjected to overloading, mishandling, gives suspect results, has been shown to be defective, or is performing outside of specified limits is taken out of service and either removed from the work area or labeled to prevent accidental use until it has been repaired and verified to perform correctly.

When analytical equipment is taken out of service, the laboratory examines the potential effect it may have had on previous analytical results to identify any non-conforming work. (See section 4.9).

5.5.7 Calibration Status

The laboratory labels support equipment to indicate calibration status, whenever practicable or otherwise maintains the calibration status in a visible location in the work area. These procedures are described in laboratory SOP *Equipment and Instrumentation* [QA SOP ME002JT].

The calibration status of analytical instruments is documented in the analytical record. Analysts verify on-going acceptability of calibration status prior to use and with instrument performance check standards. These procedures are described in test method SOPs.

5.5.8 Returned Equipment Checks

When equipment or an instrument is sent out of the laboratory for service, the laboratory ensures that the function and calibration status of the equipment is checked and shown to be

satisfactory before the equipment is returned to service. These procedures are outlined in SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification.*

5.5.9 Intermediate Equipment Checks

The laboratory performs intermediate checks on equipment to verify the on-going calibration status. For example, most test methods require some form of continuing calibration verification check and these procedures are included in the test method SOP. Periodic checks of support equipment are also performed; see laboratory *Equipment and Instrumentation* [QA SOP ME002JT]. for more information.

5.5.10 Safeguarding Equipment Integrity

The laboratory safeguards equipment integrity using a variety of mechanisms that include but are not limited to:

- Adherence to manufacturer's specification for instrument use so that settings do not exceed manufacturer's recommendation or stress the performance of the equipment.
- Established maintenance programs.
- Transparent maintenance records and unrestricted access to maintenance logs.
- Validation and approval of software before use.
- Audits to confirm instrument settings are consistent with SOPs.
- On-the-job training for safe and proper use of laboratory equipment.

5.6 Measurement Traceability

5.6.1 General

Measurement traceability refers to a property of a measurement result whereby the result can be related to a reference through an unbroken chain of calibration, each contributing to the measurement uncertainty. Traceability requires an established calibration hierarchy of equipment (instruments) used during testing including equipment used for subsidiary measurements. The laboratory assures this equipment is calibrated prior to being put into service and that the reference standard and materials used for calibration are traceable to the international standard of units (SI) or national measurement standard.

When strict traceability to SI units cannot be made, the laboratory establishes traceability with the use of reference standards and equipment obtained from competent suppliers that provide calibration certificates and/or certificates of analysis (COA).

5.6.2 Equipment Correction Factors

When correction factors are used to adjust results the laboratory will assure that results in computer software are also updated. For example, if the direct instrument or reading output must be corrected based on preparation factor or concentration factors, laboratory management will assure the corrected result is also updated in the software.

5.6.3 Specific Requirements

5.6.3.1 Requirements for Calibration Laboratories

The laboratory does not offer calibration services to customers.

5.6.3.2 Requirements for Testing Laboratories

The laboratory has procedures in place to verify equipment is calibrated prior to being put into service (See 5.5.2) and ensures the reference standard and materials used for calibration are traceable to the international standard of units (SI) or national measurement standard. When strict traceability to SI units cannot be made, the laboratory establishes traceability with the use of reference standards and equipment obtained from competent suppliers that provide calibration certificates and/or certificates of analysis (COA).

5.6.4 Reference Standards and Reference Materials

5.6.4.1 Reference Standards

The laboratory uses reference standards of measurement to verify adequacy of working weights and thermometers. The working weight is the weight(s) used for daily balance calibration checks and the working thermometers are used for temperature measurements on a daily basis.

Intermediate checks of the working reference measurement standards are performed to verify adequacy between calibration from an external calibration laboratory. The measurements from working weights and thermometers are compared to measurements taken by the reference standard which is traceable to SI or a national standard. The reference weights and thermometers are used solely for verification purposes unless the laboratory can prove that daily use does not adversely affect performance of the reference standard.

The laboratory performs intermediate checks of the working weights at least annually.

Working thermometers (glass and digital) are checked against the reference thermometer prior to placement in service to establish a correction factor and then rechecked annually (glass) or quarterly (digital) thereafter.

The calibration of liquid in glass reference thermometers is verified every 5 years and the calibration of digital reference thermometers is verified annually by an ISO/IEC 17025 accredited calibration laboratory or service provider that provides traceability to a national standard.

The calibration of the reference weight(s) is verified every 5 years by an ISO/IEC 17025 accredited calibration laboratory.

If criteria for the intermediate checks or recertification is not acceptable, the impact on previously reported results is evaluated using the process for evaluation of nonconforming work (See 4.9).

See laboratory SOPs *Equipment and Instrumentation* [QA SOP ME002JT] and *Procurement of Laboratory Supplies, Services, and Equipment* [AD SOP ME0015U] for more information about this process.

5.6.4.2 Reference Materials

The laboratory purchases chemical reference materials (also known as stock standards) from vendors that are accredited to ISO 17034 or Guide 34. Purchased

reference materials must be received with a Certificate of Analysis (COA) where available. If a reference material cannot be purchased with a COA, it must be verified by analysis and comparison to a certified reference material and/or there must be a demonstration of capability for characterization. COA are reviewed for adequacy and retained by the laboratory for future reference.

All prepared standards, reference materials, and reagents are verified to meet the requirements of the test method through routine analyses of quality control samples.

The laboratory procedure for traceability and use of these materials is provided in laboratory SOP *Procurement of Laboratory Supplies, Services, and Equipment* [AD SOP ME0015U] and *Preparation and Documentation of Laboratory Standards and Reagents* [QA SOP ME001HG].

This SOP includes each of the following requirements:

- Procedures for documentation of receipt and tracking. The record of entry includes name of the material, the lot number, receipt date, and expiration date.
- Storage conditions and requirements. Reference materials must be stored separately from samples, extracts, and digestates.
- Requirements to assure that preparations of intermediate or working solutions are recorded and assigned a unique identification number for tracking. Records of preparation include the lot number of the stock standard(s) used, the type and lot number of the solvent, the formulation, date, expiration date, and the preparer's initials. The lot number of the working standards is recorded in the analytical record to provide traceability to the standard preparation record. The preparation record provides traceability to the COA, which is traceable to SI or the national measurement standard.
- A requirement that the expiration dates of prepared standards may not exceed the expiration date of the parent standard. Standards, reference materials, and reagents are not used after their expiration dates unless it is not possible to procure a new standard and the reliability of the expired material is verified and documented by the laboratory using a procedure approved by corporate quality personnel. Otherwise, the expired material is promptly removed from the work area or clearly labeled as acceptable for qualitative/troubleshooting purposes only.
- The second source materials used for verification of instrument calibration are obtained from a different manufacturer or may be a different lot from the same manufacturer.
- Procedures to check reference materials for degradation and replacement of material if degradation or evaporation is suspected.
- Procedures for labeling. At a minimum the container must identify the material, the ID of the material and the expiration date. Original containers should also be labeled with date opened.

5.6.4.3 Intermediate Checks

Checks to confirm the calibration status of standards and materials are described in laboratory SOPs. These checks include use of second source standards and reference materials reserved only for the purpose of calibration checks.

5.6.4.4 Transport and Storage

The laboratory handles and transports reference standards and materials in a manner that protects the integrity of the materials. Reference standard and material integrity is protected by separation from incompatible materials and/or minimizing exposure to degrading environments or materials. Standards and reference materials are stored separately from samples, extracts, and digestates. All standards are stored according to the manufacturer's recommended conditions. Temperatures colder than the manufacturer's recommendation are acceptable if it does not compromise the integrity of the material (e.g. remains in liquid state and does not freeze solid). In the event a standard is made from more than a single source with different storage conditions, the standard will be stored according to the conditions specified in the analytical method.

See the applicable analytical SOPs for specific reference material storage and transport protocols.

5.7 Sampling

Sampling refers to the field collection of samples and to subsamples taken by the laboratory for analysis from the field collected sample.

Subsampling procedures are included in each test method SOP or a stand-alone SOP to assure the aliquot used for testing is representative of the field collected sample.

The requirements in the following subsections apply when field sampling is performed by the laboratory.

5.7.1 Sampling Plans and SOPs

When the laboratory performs field collection of samples, sampling is carried out in accordance with a written sample plan prepared by the customer or by the laboratory and by relevant sampling SOPs. These documents are made readily accessible at the sampling location. Sampling plans and SOPs are, whenever reasonable, based on appropriate governing methods and address the factors to be controlled to ensure the validity of the analytical results.

5.7.2 Customer Requested Deviations

When the customer requires deviations, additions, or exclusions from the documented laboratory sampling plan and/or procedure, the laboratory records the client's change request in detail with the sampling record, communicates the change to sampling personnel, and includes this information in the final test report.

5.7.3 Recordkeeping

The laboratory assures the sampling record includes the sampling procedure used, any deviations from the procedure, the date and time of sampling, the identification of the sampler, environmental conditions (if relevant), and the sampling location.

5.8 Sample Management & Handling

5.8.1 Procedures

The laboratory's procedures for sample management and handling are outlined in laboratory SOP *Sample Receiving* [AD SOP ME0013H].

The procedures in these SOPs are established to maintain the safe handling and integrity of samples from transport, storage, to disposal and during all processing steps to maintain client confidentiality, and to protect the interests of PAS and its customers.

5.8.1.1 Chain of Custody

All samples received by the laboratory must be accompanied with a Chain of Custody (COC) record. The COC provides information about the samples collected and submitted for testing and documents the possession of samples from time of collection to receipt by the laboratory.

The COC record must minimally include the following information:

- Client name, address, phone number;
- Project Reference;
- Client Sample Identification (Client ID);
- Date, Time, and Location of Sampling;
- Sampler's Name or Initials;
- Matrix;
- Type of container, and total number collected for each sample;
- Preservatives;
- Analyses Requested;
- Mode of collection;
- Any special instructions; and
- The date and time and signature of each sample transfer from time of collection to receipt in the laboratory. When the COC is transported inside the cooler, independent couriers do not sign the COC, the shipping manifests and/or air bills are the records of possession during transport. The shipping manifest must be retained as part of the COC record and included in the test report when required (See Section 5.10.3).

A complete and legible COC is required. If the laboratory observes that the COC is incomplete or illegible, the client is contacted for resolution. The COC must be filled out in indelible ink. Personnel correct errors by drawing a single line through the initial entry so the entry is not obscured, entering the correct information, and initialing, and dating the change.

5.8.1.2 Legal Chain of Custody

Legal chain of custody is a chain of custody protocol used for evidentiary or legal purposes. The protocol is followed by the laboratory when requested by customer or where mandated by a regulatory program.

Legal chain of custody (COC) protocol establishes an intact, continuous record of the physical possession*, storage, and disposal of "samples" which includes sample aliquots, and sample extracts/digestates/distillates.

Legal COC records account for all time periods associated with the samples and identifies all individuals who physically handled individual samples. Legal COC begins at the point established by legal authority, which is usually at the time the sample containers are provided by the laboratory for sample collect or when sample collection begins.

*A sample is in someone's custody if:

- It is in one's physical possession;
- It is in one's view after being in one's physical possession;
- It has been in one's physical possession and then locked or sealed so that no one can tamper with it; and/or
- It is kept in a secure area, restricted to authorized personnel only.

Refer to laboratory SOP *Sample Receiving* [AD SOP ME0013H] for more information.

5.8.2 Unique Identification

Each sample is assigned a unique identification number by the laboratory (Lab ID) after the sample has been checked and accepted by the laboratory in accordance with the laboratory's sample acceptance policy (See 5.8.3). The Lab ID is affixed to the sample container using a durable label.

The unique identification of samples also applies to subsamples, and prepared samples, such as extracts, digestates, etc.

The lab ID is linked to the field ID (client ID) in the laboratory's record. Both IDs are linked to the testing activities performed on the sample and the documentation records of the test.

Also see 5.8.4.

5.8.3 Sample Receipt Checks and Sample Acceptance Policy

The laboratory checks the condition and integrity of samples on receipt and compares the labels on the sample containers to the COC record. Any problem or discrepancy is recorded. If the problem impacts the suitability of the sample for analysis or if the documentation is incomplete, the client is notified for resolution. Decisions and instructions from the client are maintained in the project record.

5.8.3.1 Sample Receipt Checks

The following checks are performed:

- Verification that the COC is complete and legible.
- Verification that each sample's container label includes the client sample ID, the date and time of collection and the preservative in indelible ink.
- The container type and preservative are appropriate for each test requested.
- Adequate volume is received for each test requested.
- Visual inspection for damage or evidence of tampering.
- Visual inspection for presence of headspace in VOA vials. (VOA = volatile organic analysis).
- Thermal Preservation: Generally, for chemical testing methods for which thermal preservation is required, temperature on receipt is acceptable if the measurement is above freezing but <6°C. The requirements for thermal preservation vary based on test method or by regulatory program. For example, for microbiology, temperature on receipt is acceptable if the measurement is <10°C. Refer to the laboratory's SOP for sample receipt for specific requirements. For samples that are hand-delivered to the laboratory immediately after sample collection, there must be evidence that the chilling process began immediately after sample collection and prior to delivery of the samples to the laboratory or service center, such as arrival of the samples on ice.
- Chemical Preservation
- Holding Time: Sample receiving personnel are trained to recognize tests where the holding time is 48 hours or less and to expedite the log-in of these samples. Except for tests with immediate holding times (15 minutes from time of collection or less), when samples are received out of hold, the laboratory will notify the client and request instruction. If the decision is made to proceed with analysis, the final test report will include notation of this instruction.

5.8.3.2 Sample Acceptance Policy

The laboratory maintains a sample acceptance policy in accordance with regulatory guidelines to clearly establish the circumstances in which sample receipt is accepted or rejected.

When receipt does not meet criteria for any one of these conditions, the laboratory must document the noncompliance, contact the customer, and either reject the samples or fully document any decisions to proceed with testing. In accordance with regulatory specifications, test results associated with receipt conditions that do not meet criteria are qualified in the final test report.

All samples received must meet each of the following criteria:

- Be listed on a complete and legible COC;
- Be received in properly labeled sample containers;
- Be received in appropriate containers that identify preservative;

- The COC must include the date and time of collection for each sample;
- The COC must include the test method requested for each sample;
- Be in appropriate sample containers with clear documentation of the preservatives used;
- Be received within holding time. Any samples received beyond the holding time will not be processed without prior customer approval;
- Have sufficient sample volume to proceed with the analytical testing. If insufficient sample volume is received, analysis will not proceed without customer approval; and
- Be received within appropriate temperature ranges unless program requirements or customer contractual obligations mandate otherwise. The cooler temperature is recorded directly on the COC.

  Samples that are delivered to the laboratory immediately after collection are considered acceptable if there is evidence that the chilling process has been started. For example, by the arrival of the samples on ice. If samples arrive that are not compliant with these temperature requirements, the customer will be notified. The analysis will NOT proceed unless otherwise directed by the customer. If less than 72 hours remain in the hold time for the analysis, the analysis may be started while the customer is contacted to avoid missing the hold time. Data associated with any deviations from the above sample acceptance policy requirements will be appropriately qualified.

5.8.4 Sample Control and Tracking

The samples are controlled and tracked using the Laboratory Information Management System (LIMS). The LIMS stores information about the samples and project. The process of entering information into the LIMS is called log-in and these procedures are described in laboratory SOP *Sample Receiving* [AD SOP ME0013H]. After log-in, a label is generated and affixed to each sample container. Information on this label, such as the lab ID, links the sample container to the information in LIMS.

At a minimum, the following information is entered during log-in:

- Client Name and Contact Information;
- The laboratory ID linked to the client ID;
- Date and time of sample collection;
- Date and time of sample receipt;
- Matrix; and
- Tests Requested.

5.8.5 Sample Storage, Handling, and Disposal

The laboratory procedures for sample storage, handling and disposal are detailed in laboratory SOPs *Sample Receiving* [AD SOP ME0013H] and *Hazardous and Non-Hazardous Waste Management Plan* [HS SOP ME0012A.

5.8.5.1 Sample Storage

The samples are stored according to method and regulatory requirements as per test method SOPs. Samples are stored away from all standards, reagents, or other potential sources of contamination and stored in a manner that prevents cross contamination. Volatile samples are stored separately from other samples. All sample fractions, extracts, leachates, and other sample preparation products are stored in the same manner as actual samples or as specified by the analytical method.

Refrigerated storage areas are maintained at ≤6°C (but not frozen) and freezer storage areas are maintained at <-10°C, unless otherwise required per method or program. The temperature of each storage area is checked and documented at least once for each day of use. If the temperature falls outside the acceptable limits, then corrective actions are taken and appropriately documented.

The laboratory is operated under controlled access protocols to ensure sample and data integrity. Visitors must register at the front desk and be properly escorted while on-site. Samples are taken to the appropriate storage location immediately after sample receipt and log-in procedures are completed. All sample storage areas have limited access. Samples are removed from storage areas by designated personnel and returned to the storage areas as soon as possible after the required sample quantity has been taken.

5.8.5.2 Sample Retention and Disposal

The procedures used by the laboratory for sample retention and disposal are detailed in laboratory SOP *Hazardous and Non-Hazardous Laboratory Waste Management Plan* [HS SOP ME0012A].

In general, unused sample volume and prepared samples such as extracts, digestates, distillates and leachates (samples) are retained by the laboratory for the timeframe necessary to protect the interests of the laboratory and the customer.

Samples may be stored at ambient temperature when all analyses are complete, the hold time is expired, the report has been delivered, and/or when allowed by the customer or program. Samples requiring storage beyond the minimum sample retention time due to special requests or contractual obligations may be stored at ambient temperature unless the laboratory has a capacity and their presence does not compromise the integrity of other samples.

After this period expires, non-hazardous samples are properly disposed of as non-hazardous waste. The preferred method for disposition of hazardous samples is to return the excess sample to the customer.

5.9 Assuring the Quality of Test Results

5.9.1 Quality Control (QC) Procedures

The laboratory monitors the validity and reliability of test results using quality control (QC) samples that are prepared and analyzed concurrently with field samples in the same manner as field samples. QC results are always associated to and reported with the field samples they were prepared and analyzed with from the same preparation or analytical batch. See the glossary for definition of preparation and analytical batch.

The results of QC performed during the testing process are used by the laboratory to assure the results of analysis are consistent, comparable, accurate, and/or precise within a specified limit. When the results are not within acceptance criteria or expectations for method performance, correction and corrective action(s) are taken. These actions may include retesting or reporting of data with qualification to alert the end user of the situation.

Other QC measures performed include the use of certified reference materials (see 5.6.4), participation in interlaboratory proficiency testing (see 5.9.1.2), verification that formulae used for reduction of data and calculation of results is accurate (see 5.9.3), on-going monitoring of environmental conditions that could impact test results (see 5.3.2), and evaluation and verification of method selectivity and sensitivity (see 5.4.5).

QC results are also used by the laboratory to monitor performance statistical trends over time and to establish acceptance criteria when no method or regulatory criteria exist. (See 5.9.1.1.9)).

5.9.1.1 Essential QC

Although the general principles of QC for the testing process apply to all testing, the QC protocol used for each test depends on the type of test performed.

QC protocol used by the laboratory to monitor the validity of the test are specified in test method SOPs. The SOP includes QC type, frequency, acceptance criteria, corrective actions, and procedures for reporting of nonconforming work.

These requirements in the SOP conform to the reference method and any applicable regulations or certification and accreditation program requirement for which results of the test are used. When a project requires more stringent QC protocol than specified in the SOP, project specification is followed. When the project requires less stringent QC protocol, the project specification may be followed as an authorized departure from the SOP when the project specifications meet the requirements in the mandated method and any regulatory compliance requirements for which the data will be used.

The following are examples of essential QC for Chemistry:

5.9.1.1.1 Second Source Standard (ICV/QCS)

The second source standard is a standard obtained from a different vendor than the vendor of the standards used for calibration or it may be from a different lot from the same vendor when there are limited vendors that offer the material. It is a positive control used to verify the accuracy of a new calibration relative to the purity of the standards used for calibration. This check is referred to in test

method and quality system standards as the initial calibration verification (ICV) or quality control sample (QCS). The second source standard is analyzed immediately after the calibration and before analysis of any samples. When the ICV is not within acceptance criteria, a problem with the purity or preparation of the standards may be indicated.

5.9.1.1.2 Continuing Calibration Verification (CCV)

CCV results are used to determine if the analytical response has significantly changed since initial calibration. If the response of the CCV is within criteria, the calibration is considered valid. If not, there is a problem that requires further investigation. Actions taken are technology and method specific.

5.9.1.1.3 Method Blank (MB) / Other Blanks

A method blank is a negative control used to assess for contamination during the prep/analysis process. The MB consists of a clean matrix, similar to the associated samples that is known to be free of analytes of interest. The MB, unless otherwise specified by the test method, is processed with and carried through all preparation and analytical steps as the associated samples.

In general, contamination is suspected when the target analyte is detected in the MB above the reporting limit. Some programs may require evaluation of the MB to ½ the reporting limit or the detection limit. When contamination is evident, the source is investigated, and corrections are taken to reduce or eliminate it. Analytical results associated with MB that does not meet criteria are qualified in the final test report.

Other types of blanks that serve as negative controls in the process may include:

- Trip Blanks (VOA)
- Storage Blanks
- Equipment Blanks
- Field Blanks
- Calibration Blanks
- Cleanup Blanks
- Instrument Blanks

5.9.1.1.4 Laboratory Control Sample (LCS)

The LCS is positive control used to measure the accuracy of process in a blank matrix. The LCS is spiked by the laboratory with a known amount of analyte. The spike is a standard solution that is pre-made or prepared from a certified reference standard. Like the MB, unless otherwise specified in the test method, the LCS is processed with

and carried through all preparation and analytical steps as the associated samples.

When the percent recovery (%R) of the LCS is within the established control limit, sufficient accuracy has been achieved. If not, the source of the problem is investigated and corrected, and the procedure may be repeated. Analytical results associated with LCS that does not meet criteria are qualified in the final test report.

5.9.1.1.5 Matrix Spike (MS) and Matrix Spike Duplicate (MSD)

Matrix spikes measure the effect the sample matrix has on precision and accuracy of the determinative test method. The MS and MSD are replicates of a client sample that is spiked with known amount of target analyte.

Due to the heterogeneity of matrices even of the same general matrix type, matrix spike results mostly provide information on the effect of the matrix to the client whose sample was used and on samples of the same matrix from the same sampling site. Therefore, MS should be client-specific when the impact of matrix on accuracy and precision is a project data quality objective. When there is not a client-specified MS for any sample in the batch, the laboratory randomly selects a sample from the batch; the sample selected at random is called a "batch" matrix spike.

The MS/MSD results for percent recovery and relative percent difference are checked against control limits. Because the performance of matrix spikes is matrix-dependent, the result of matrix spikes is not used to determine the acceptability of the test.

5.9.1.1.6 Sample Duplicate (SD)

A sample duplicate is a second replicate of sample that is prepared and analyzed in the laboratory along another replicate. The SD is used to measure precision.

The relative percent difference between replicates are evaluated against the method or laboratory derived criteria for relative percent difference (RPD), when this criterion is applicable. If RPD is not met, associated test results are reported with qualification.

5.9.1.1.7 Surrogates

Surrogates are compounds that mimic the chemistry of target analytes but are not expected to occur naturally in real world samples. Surrogates are added to each sample and matrix QC samples (MS, MSD, SD) at known concentration to measure the impact of the matrix on the accuracy of method performance. Surrogates are also added to the positive and negative control samples (MB, LCS) to evaluate performance in a clean matrix, and included in the calibration standards and calibration check standards.

The percent recovery of surrogates is evaluated against method-specified limits or statistically derived in-house limits. Project-specific limits and/or program-specific limits are used when required. Results with surrogate recovery out of limits in samples are reported with qualification. Samples with surrogate failures can also be re-extracted and/or re-analyzed to confirm that the out-of-control value was caused by the matrix of the sample and not by some other systematic error.

5.9.1.1.8 Internal Standards

Internal Standards are compounds not expected to occur naturally in field samples. They are added to every standard and sample at a known concentration prior to analysis for the purpose of adjusting the response factor used in quantifying target analytes. The laboratory follows specific guidelines for the treatment of internal standard recoveries and further information can be found in the applicable laboratory SOP.

5.9.1.1.9 QC Acceptance Criteria and Control Limits

The QC acceptance criteria are specified in test method SOPs. The criteria in the SOP are based on the requirements in the published test method or regulatory program. When there are no established acceptance criteria, the laboratory develops acceptance criteria in accordance with recognized industry standards.

Some methods and programs require the laboratory to establish control limits for LCS, MS/MSD, and surrogate evaluation using historical data. Laboratory developed limits are referred to as "in-house" control limits. In-house control limits represent ± 3 Standard Deviations (99% confidence level) from the average recovery of at least 20 data points generated using the same preparation and analytical procedure in a similar matrix.

See laboratory SOP *Trend Analysis of Data Using Control Charts* [QA Policy ME001IW] for more information about the procedures used to establish in-house control limits.

5.9.1.2 Proficiency Testing (PT)

The laboratory participates in interlaboratory proficiency testing (PT) studies to measure performance of the test method and to identify or solve analytical problems. PT samples measure laboratory performance through the analysis of unknown samples provided by an external source.

The PT samples are obtained from accredited proficiency testing providers (PTP) and handled as field samples which means they are included in the laboratory's normal analytical processes and do not receive extraordinary attention due to their nature.

The laboratory does not share PT samples with other laboratories, does not communicate with other laboratories regarding current PT sample results during the

duration of the study, and does not attempt to obtain the assigned value of any PT sample from the PT provider.

The laboratory investigates and implements corrective action whenever PT results are scored unacceptable by the PT provider.

The frequency of PT participation is based on the certification and accreditation requirements held by the laboratory.

5.9.2 QC Corrective Action

When the results of QC are not within acceptance criteria or expectations for method performance, correction and corrective action(s) are taken per the specifications in the test method SOP. These actions may include retesting or reporting of data with qualification to alert the end user of the situation.

5.9.3 Data Review

The laboratory uses a tiered system for data review. The tiered process provides sequential checks to verify data transfer is complete; manual calculations, if performed, are correct, manual integrations are appropriate and documented, calibration and QC requirements are met, appropriate corrective action was taken when required, test results are properly qualified, process and test method SOPs were followed, project specific requirements were met, when applicable, and the test report is complete.

The sequential process includes three tiers referred to as primary review, secondary review, and administrative/completeness review.

Detailed procedures for the data review process are described in laboratory SOP *Data Review* [QA SOP ME003LP]. The general expectations for the tiered review process are described in the following sections:

5.9.3.1 Primary Review

Primary review is performed by the individual that performed the task. All laboratory personnel are responsible for review of their work product to assure it is complete, accurate, documented, and consistent with policy and SOPs.

Checks performed during primary review include but are not limited to:

- Verification that data transfer and acquisition is complete
- Manual calculations, if performed, are documented and accurate
- Manual integrations, if performed, are documented and comply with SOP ENV-SOP-CORQ-006 *Manual Integration*
- Calibration and QC criteria were met, and/or proper correction and corrective actions were taken, and data and test results associated with QC and criteria exceptions are properly qualified
- Work is consistent with SOPs and any other relevant instructional document such as SWI, program requirements, or project QAPP

5.9.3.2 Secondary Review

Secondary review is performed by a qualified peer or supervisor. Secondary review is essentially a repeat of the checks performed during primary review by another person. In addition to the checks of primary review, secondary review includes chromatography review to check the accuracy of quantitative analyte identification.

5.9.3.3 Completeness Review

Completeness review is an administrative review performed prior to release of the test report to the customer. Completeness review verifies that the final test report is complete and meets project specification. This review also assures that information necessary for the client's interpretation of results are explained in the case narrative or footnoted in the test report.

5.9.3.4 Data Audits

In addition to the 3-tier data review process, test reports may be audited by local quality personnel to verify compliance with SOPs and to check for data integrity, technical accuracy, and regulatory compliance. These audits are not usually done prior to issuance of the test report to the customer. The reports chosen for the data audits are selected at random.

If any problems with the data or test results are found during the data audit, the impact of the nonconforming work is evaluated using the process described in Section 4.9.

Also see Section 4.14 for internal audits.

5.9.4 Calibration Certificates

The laboratory does not perform calibration activities for its customers and calibration certificates are not offered or issued.

5.9.5 Opinions and Interpretations

The laboratory provides objective data and information to its customers of sufficient detail for their interpretation and decision making. Objective data and information are based solely on fact and does not attempt to explain the meaning (interpret) or offer a view or judgement (opinion). Sometimes the customer may request the laboratory provide opinion or interpretation to assist them with their decisions about the data.

When opinions and interpretations are included in the test report, the laboratory will document the basis upon which the opinions and interpretations have been made and clearly identify this content as opinion or interpretation in the test report.

Examples of opinion and interpretation include but are not limited to:

- The laboratory's viewpoint on how a nonconformance impacts the quality of the data or usability of results.
- The laboratory's judgment of fulfillment of contractual requirements.
- Recommendations for how the customer should use the test results and information.
- Suggestions or guidance to the customer for improvement.

When opinions or interpretations are verbally discussed with the customer, the content of these conversations is summarized by the laboratory and kept in the project record.

5.9.6 Subcontractor Reports

When analytical work has been subcontracted to an organization external to PAS, the test report from the subcontractor is included in its entirety as an amendment to the final test report.

Test results performed by multiple locations within the PAS network may be merged into a single test report. The test report issued clearly identifies the location and address of each network location that performed testing, and which tests they performed. (See 5.10.2)

5.9.7 Electronic Transmission of Results

When test results and/or reports are submitted to the customer through electronic transmission, the procedures established in this manual for confidentiality and protection of data apply.

5.9.8 Format of Test Reports

The test formats offered by the laboratory are designed to accommodate each type of analytical test method carried out by the laboratory and to minimize the possibility of misunderstanding or misuse of analytical results. The format of electronic data deliverables (EDD) follow the specifications for the EDD.

5.9.9 Amendments to Test Reports

Test reports that are revised or amended by the laboratory after date of release of the original final test report to the customer are issued as a new test report that is clearly identified as an amendment or revision and that includes a reference to the originally issued final test report.

The customer is the organization doing business with PAS external to PAS.

Changes made to test results and data before the final test report is issued to the customer are not amendments or revisions, these are corrections to errors found during the laboratory's data verification and review process.

The laboratory's procedure for report amendments and revision are outlined in laboratory SOP *Project Management* [AD SOP ME001HD].

5.10 Reporting

5.10.1 General Requirements

The laboratory reports results of testing in a way that assures the results are clear, and unambiguous. All data and results are reviewed prior to reporting to assure the results reported are accurate and complete.

Test results are summarized in test reports that include all information necessary for the customer's interpretation of the test results. Additional information necessary to clarify the data or disclose nonconformance, exceptions, or deviations that occurred during the analytical process are also reported to the customer in the test report.

The specifications for test reports and EDD are established between the laboratory and the customer at the time the request for analytical services is initiated. The report specifications include the test report format, protocol for the reporting limit (RL), conventions for the reporting of results less than the limit of quantitation (LOQ), and specification for the use of

project or program specific data qualifiers. Information about review of analytical service requests is provided in Section 4.4.

5.10.2 Test Reports: Required Items

Test Reports are prepared by the laboratory at the end of the testing process. The format of the report depends on the level of reporting requested by the customer. The laboratory offers a variety of standardized test report formats and can provide custom test report formats, when necessary.

The level of detail required in the test report depends on the customer's needs for data verification, validation, and usability assessments that occur after the laboratory releases the test report to the customer. The test report formats offered by the laboratory provide gradient levels of detail to meet the unique needs of each customer. The laboratory project manager helps the customer select the test report format that best meets their needs. When a specific report format or protocol is required for a regulatory or program compliance, the laboratory project manager must ensure the test report selected meets those requirements.

Every test report issued by the laboratory includes each of the following items:

a) Title

b) Name and phone number of a point of contact from the laboratory issuing the report.

c) Name and address of the laboratory where testing was performed. When testing is done at multiple locations within network (IRWO), the report must clearly identify which network laboratory performed each test and must include the physical address of each laboratory.

d) Unique identification of the test report and an identifier on each page of the report to link each page to the test report and clear identification of the end of the report.

e) The name and address of the customer

f) Identification of test methods used

g) Cross reference between client sample identification number (Sample ID) and the laboratory's identification number for the sample (Lab ID) to provide unambiguous identification of samples.

h) The date of receipt of samples, condition of samples on receipt, and identification of any instance where receipt of the samples did not meet sample acceptance criteria.

i) Date and times of sample collection, receipt, preparation, and analysis.

j) Test results and units of measurement, and qualification of results associated with QC criteria exceptions, and identification of reported results outside of the calibration range.

k) All chains of custody (COC) including records of internal transfer between locations within the PAS network.

l) Name, title, signature of the person(s) authorizing release of the test report and date of release.

m) A statement that the results in the test report relate only to the items tested.

n) Statement that the test report may not be reproduced except in full without written approval from the laboratory.

5.10.3 Test Reports: Supplemental Items

5.10.3.1 Supplemental Requirements

The following items are included in the test report when required or relevant:

a) Shipping manifests / bill of ladings as applicable when common couriers are utilized for shipment of samples,

b) Explanation of departure from test method SOPs including, what the departure was and why it was necessary.

c) Statistical methods used. (Required for Whole Effluent Toxicity)

d) For solid samples, specification that results are reported on a dry weight or wet weight basis.

e) Signed Affidavit, when required by client or regulatory agency.

f) A statement of compliance / non-compliance with requirements or specifications (client, program, or standard) that includes identification of test results that did not meet acceptance criteria.

g) When requested by the client, statement of estimated measurement uncertainty. In general, for environmental testing, estimated uncertainty of measurement is extrapolated from LCS control limits. Control limits incorporate the expected variation of the data derived from the laboratory's procedure. When the control limits are specified by the test method or regulatory program, the control limits represent the expected variation of the test method and/or matrices for which the test method was designed.

h) Opinions and Interpretations

i) If a claim of accreditation/certification is included in the test report, identification of any test methods or analytes for which accreditation/certification is not held by the laboratory if the accrediting body offers accreditation/certification for the test method/analyte. The fields of accreditation/certification vary between agencies and it cannot be presumed that because accreditation/certification is not held that it is offered or required.

j) Certification Information, including certificate number and issuing body.

5.10.3.2 Test Reports: Sampling Information

The following items are included in the test report when samples are collected by the laboratory or when this information is necessary for the interpretation of test results:

a) Date of Sampling.

b) Unambiguous identification of material samples.

c) Location of sampling including diagrams, sketches, or photographs.

d) Reference to the sampling plan and procedures used.

e) Details of environmental conditions at time of sample that may impact test results.

f) Any standard or other specification for the sampling method or procedure, and deviations, additions to or exclusions from the specification concerned.

6.0 REVISION HISTORY

This Version:

Section	Description of Change
Manual Approval Signatory Page	Added "Quality" before Manual. Updated the list of required signatories and changed job titles to match current job descriptions.
All	Replaced "PAS" with "ENV" to denote that ENV is division of PAS. References to PAS were left in some sections when the policy or procedure applies to all business units in addition to environmental sciences.
All	Corrected spelling, typographical, and format errors.
All	Changed "laboratory" to "location" when requirement applies to non-testing locations, such as service centers.
All	References to "Local QA" was replaced with "Local QM"
1.2.1	Changed frequency of review from every 2 years to annually.
1.2.2	Clarified local management refers to the signatories of the manual.
2.0	Replaced reference "current version" for ISO Standard with 2nd and 3rd Editions and publication dates.
4.1.3	Removed table and inserted reference to Title Page, where locations covered by the manual are listed.
4.1.4.1	Updated content to match current organization structure and job titles maintained by corporate HR.
4.5.1.1	Updated content to match current organization structure and job titles maintained by corporate HR.
4.5.1.2	Added new positions, updated job titles to current HR job titles, removed obsolete job titles.
4.5.2.1	Added timeframe for AB notification for absence of acting TNI Technical Manager.
4.2.2.1	Replaced term "tertiary" with completeness and replaced reference to MintMiner with data surveillance.
4.2.5.1	Updated definition of Guide; Added Guidance
4.5	Changed reference to procurement program to vendor qualification program.
4.6	Added reference to corporate SOP for vendor qualification.
4.7.1	Removed reference to SME, the SME program was not formalized as planned.
4.7.2	Removed reference to monthly; the frequency of management reports is established by the executive leadership team based on need.
4.11	Replaced reference to local SOP with corporate SOP. The corporate SOP replaced all local SOPs for the process. Updated 7 Stage process to match SOP.
4.11.1	Changed reference to root cause analysis to cause analysis.
4.12	Removed 7 step process for preventive action. PA is rolled into the 7 stage process for CAPA.
4.12.1	Removed reference to preventive action SOP – this was a typo for this section.
4.13.1	Added reference to the corporate policy for records.
4.13.1.2	Updated record retention time frame to match policy.

4.13.1.5	Added this section to incorporate electronic signature policy.
4.14.1	Rewrote paragraph that describes audit program.
4.15	Replaced reference to local SOP with corporate SOP. The corporate SOP replaced all local SOPs for the process.
4.16	Rewrote paragraph for clarity and updated SOP references.
5.2.2	Updated section to match current requirements.
5.2.2.1	Changed "monitor" to "tracks" to clarify expectation.
5.2.2.3	Added this section.
5.2.2.1.3	Changed "attendance sheet" to signature record.
5.4.5.2	Replaced local SOP reference with referral to corporate policy.
5.4.5.3.4	Fixed typographical error related to RL as qualitative/quantitative value. Moved SOP reference from section 5.4.5.3.3 to this section.
5.5.2.2	Updated policy reference.
5.5.9	Replaced typographical error reference to Appendix E with reference to local SOP.
5.6.4.2	Clarified requirements for expired reference materials.
5.8.1.1	Added requirement for locations to retain shipping manifest as COC record.
5.8.3.1	Updated requirements for thermal preservation.
5.9.1.1.1	Updated section to specify the second source standard may also be a different lot from the same manufacturer.
5.9.1.1.3	Added unless otherwise specified by test method exception.
5.9.1.1.4	Added unless otherwise specified by test method exception.
5.9.1.1.9	Clarified that in-house limits are calculated using historical data.
5.10.2	Added requirement that all test reports must include copies of the COCs, including COC for in-network transfer. (CAR to State Audit Deficiency)
Glossary	Added Definition of MRL (CAR to State Audit Deficiency)
Glossary	Changed definition of MintMiner
Appendix 8.1	Added DoD/DOE requirements for LOD/LOQ
Appendix B	Added footnote specification for test methods that are not TNI accredited; applies to TNI accredited labs only.

This document supersedes the following documents:

Document Number	Title	Version
ME0012K	Quality Assurance Management Plant	14

7.0 APPENDICES

7.1 Appendix A: Certification / Accreditation Listing

The certifications / accreditation lists provided in this manual represent those that were held by the named location on the effective date of this manual. This information is subject to change without notice and must not be considered valid proof of certification or accreditation status. Current certificates are maintained by the local QM and a copy of the certificate is posted to ENV eDMS Portal for access by all ENV employees. External parties should contact the laboratory for the most current information.

7.1.1 PAS-WCOL [Analytical Laboratory]

Authority	ID	Authority	ID
US EPA CLP	Contract EP-W-14-035	**Kentucky DEP – WW**	KY98037
US EPA CLP	Contract 68HERH20D0015	**Massachusetts DEP**	NA [Approval Letter]
Alabama DEM	42000	**Michigan DEQ**	9999
Alaska DEC	20-002	**Michigan EGLE**	9999
Alaska DEC (DW)	SC00162	**New Jersey DEP – NELAP**	SC006
California NELAP	3049	**North Carolina DEQ – WW & GW**	329
Delaware ODW	NA [Approval Letter]	**Ohio EPA**	41251
ANAB ISO/IEC 17025-2017 and DoD ELAP	L2224	**North Carolina DEQ – WW & GW – Charlotte Service Center [Field]**	5639
ANAB ISO/IEC 17025-2017 and DOE ELAP	L2224.01	**South Carolina DHEC – Main Laboratory**	32010001
Florida – NELAP [Primary AB]	E87653	**South Carolina DHEC – Charlotte Service Center [Field]**	99064001
Georgia DPD	C049	**South Carolina DHEC – Greenville Service Center [Field]**	23615001
Illinois EPA	200055	**Virginia DLCS – NELAP**	10541
Kansas DHE	E-10417	**Wisconsin DNR**	NA [Approval Letter]
Kentucky DEP – UST Branch	AI# 103582		

7.1.2 PAS-WCOL [Charlotte Service Center]

Authority	ID	Authority	ID
North Carolina DEQ	5639	**South Carolina DHEC**	99064001

7.1.3 PAS-WCOL [Greenville Service Center]

Authority	ID	Authority	ID
South Carolina DHEC	23615001		

7.2 Appendix B: Capability Listing

The capabilities listed in this Appendix were held by the location referenced on the effective date of this manual. This information is subject to change without notice. External parties should contact the laboratory for the most current information.

Table Legend:

- Air = Air
- DW = Drinking Water
- NPW = Non-Potable Water
- SCM = Solid and Chemical Materials
- Waste = Non-Aqueous Phase Liquid (NAPL), Oil
- Tissue = Biota and Tissue

7.2.1 PAS-WCOL [Analytical Laboratory]

Parameter	Method	Matrices			
		DW	NPW	SCM	Tissue
Alkalinity as CaCO3 (Titration)	SM 2320 B-2011	X	X		
Alkalinity, Bicarbonate (Calculation)[1]	SM 2320 B-2011		X		
Alkalinity, Carbonate (Calculation) [1]	SM 2320 B-2011		X		
Alkalinity, Hydroxide (Calculation) [1]	SM 2320 B-2011		X		
Ammonia - N by Gas Diffusion	EPA 350.1		X	X	
Anions by IC	EPA 300.0	X	X	X	
Anions by IC	EPA 9056A		X	X	
Bacteria - Biosolids Preparation	EPA/625/R-92/013 APP F		X		
Bacteria - Escherichia coli (MPN)	SM 9223 B-2004	X	X		
Bacteria - Fecal Coliform	COLILERT®-18		X		
Bacteria - Fecal Coliform (MF)	SM 9222 D-2006		X		
Bacteria - Fecal Coliform (MPN) [1]	SM 9221-C E-2006		X		
Bacteria - Heterotrophic[1]	SIMPLATE	X			
Bacteria - Total Coliforms	SM 9223 B-2004	X			
Biochemical Oxygen Demand (BOD)	SM 5210 B-2011	X	X		
Calcium Hardness (Titration) [1]	SM 3500-Ca B-2011	X			
Carbon Dioxide (CO2) (Calculation) [1]	SM 4500-CO2 D-2011		X		
Carbonaceous BOD (CBOD)	SM 5210 B-2011		X		
Chemical Oxygen Demand (COD)	SM 5220 D-2011		X		
Chlorine, Residual (DPD) [1]	SM 4500-CL G-2011	X	X		

Parameter	Method	Matrices			
		DW	NPW	SCM	Tissue
Color, ADMI Weighted-Ordinate	SM 2120 F-2011		X		
Color, Platinum Cobalt	SM 2120 B-2011	X	X		
Corrosivity (Electrode)	EPA 9040C		X	X	
Corrosivity (Electrode)	EPA 9045C/D			X	
Cyanide, Amenable to Chlorination	EPA 9012B		X		
Cyanide, Amenable to Chlorination	Kelada-01	X			
Cyanide, Amenable to Chlorination	SM 4500-CN^- G-2011		X		
Cyanide, Total	Kelada-01	X	X		
Cyanide, Total (UV/VIS)	EPA 335.4	X	X		
Cyanide, Total (UV/VIS)	EPA 9012B		X	X	
Cyanide, Total (UV/VIS)	SM 4500 CN^- E-2011		X		
Dissolved Oxygen (DO) [1]	HACH 10360		X		
Dissolved Oxygen (DO) [1]	SM 4500-O G-2011		X		
Ferrous Iron (UV/VIS) [1]	SM 3500-Fe B-2011		X		
Hardness, Total (as CaC3) (Calculation) [1]	SM 2340 B-2011		X		
Hardness, Total (as CaCO3) (Titration)	SM 2340 C-2011		X		
Hexavalent Chromium (Discrete Analyzer)	SM 3500-Cr B-2011		X		
Hexavalent chromium (IC)	EPA 218.6		X		
Hexavalent chromium (IC)	EPA 7199		X	X	
Hexavalent chromium (UV/VIS)	EPA 7196A		X	X	
Ignitability (Pensky-Martens)	EPA 1010A/B		X	X	
Metals - Determination in Water Samples and Waste Extracts or Digests by ICP/MS	EPA 6020A/B		X	X	
Metals - Low Level Mercury (CVAA)	EPA 1631E		X		
Metals - Mercury (CVAA)	EPA 245.1	X	X		
Metals - Mercury (CVAA)	EPA 7470A		X	X	
Metals - Mercury (CVAA)	EPA 7471B			X	
Metals - Trace Elements in Aqueous Solutions by ICP/AES	EPA 6010C/D		X	X	
Metals - Trace Elements in Waters and Wastes by ICP/MS	EPA 200.8	X	X		
Metals - Trace Metals by ICP/AES	EPA 200.7	X	X		
Nitrate as N (IC)	EPA 300.0	X	X	X	

Parameter	Method	Matrices			
		DW	NPW	SCM	Tissue
Nitrate as N (IC)	EPA 9056A		X	X	
Nitrate as N (UV/VIS) - NO3-NO2 Minus NO2	EPA 353.2	X	X	X	
Nitrate-Nitrite (UV/VIS)	EPA 353.2	X	X	X	
Nitrite as N (IC)	EPA 300.0	X	X	X	
Nitrite as N (IC)	EPA 9056A		X	X	
Nitrite as N (UV/VIS)	EPA 353.2	X	X	X	
Oil & Grease	EPA 9071B		X	X	
Oil & Grease (Gravimetric)	EPA 1664B		X		
Orthophosphate (UV/VIS)	EPA 365.1	X	X		
Paint Filter Test (Filtration)	EPA 9095B		X	X	
Per- and Polyfluoroalkyl Substances (PFAS - DAI) by LC/MS/MS	SES SOP ME00217/LC-MS-MS ID		X		
Per- and Polyfluoroalkyl Substances (PFAS - ID) by LC/MS/MS	SES SOP ME00213/LC-MS-MS ID		X	X	
Per- and Polyfluoroalkyl Substances (PFAS) LC/MS/MS	EPA 533	X			
Per- and Polyfluoroalkyl Substances (PFAS) LC/MS/MS	EPA 537	X			
Per- and Polyfluoroalkyl Substances (PFAS) LC/MS/MS	EPA 537.1	X			
Per- and Polyfluoroalkyl Substances (PFAS) LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15		X	X	
pH (Electrode)	EPA 150.1	X			
pH (Electrode)	SM 4500-H+ B-2011	X	X		
Phenolics (UV/VIS)	EPA 420.4		X		
Phenolics (UV/VIS)	EPA 9065		X	X	
Phenolics (UV/VIS) [1]	EPA 9066		X	X	
Phosphorus (UV/VIS)	EPA 365.1	X	X	X	
Preparation - Acid Digestion of Aqueous Samples and TCLP/SPLP Extracts for ICP analysis	EPA 3010A		X	X	
Preparation - Acid Digestion of Aqueous Samples for ICP Spectroscopy	EPA 3030C		X		
Preparation - Acid Digestion of Waters for Total or Dissolved Metals	EPA 3005A			X	
Preparation - Alkaline Digestion for Hexavalent Chromium	EPA 3060A		X	X	
Preparation - Bomb	EPA 5050			X	

Parameter	Method	Matrices			
		DW	NPW	SCM	Tissue
Preparation - Closed-System Purge-and-Trap for Volatile Organics in Soil and Waste Samples	EPA 5035		X	X	
Preparation - Continuous Liquid-Liquid Extraction	EPA 3520C		X		
Preparation - Continuous Liquid-Liquid Extraction, Reduced Volume	EPA 3520C-RVE			X	
Preparation - Florisil Cleanup Procedure	EPA 3620B		X	X	
Preparation - GPC Cleanup Procedure	EPA 3640C		X	X	
Preparation - Metals Digestion, Soils by Hotblock	EPA 3050B			X	
Preparation - Metals Digestion, Waters by Hotblock	EPA 3005A		X		
Preparation - Micro-Dist for Cyanide	Lachat 10-204-00-1-X		X		
Preparation - Microwave Extraction	EPA 3546			X	
Preparation - Preliminary Treatment and Distillation for Cyanide	SM 4500-CN^- B,C-2011		X		
Preparation - Purge-and-Trap for Aqueous Samples	EPA 5030B		X	X	
Preparation - Soil by Sonication	EPA 3550C			X	
Preparation - Solid-Phase Extraction (SPE)	EPA 3535A		X		
Preparation - Soxhlet Extraction	EPA 3540C				X
Preparation - Sulfur Cleanup Procedure	EPA 3660B		X	X	
Preparation - Sulfuric Acid Cleanup Procedure	EPA 3665A		X	X	
Preparation - Synthetic Precipitation Leaching Procedure (SPLP)	EPA 1312		X	X	
Preparation - Toxicity Characteristic Leaching Procedure (TCLP)	EPA 1311		X	X	
Preparation - Waste Dilution	EPA 3580A		X	X	
Preparation - Waste Dilution for Volatile Organics	EPA 3585		X	X	
Reactive Cyanide	Sec. 7.3.3 SW-846		X	X	
Reactive Sulfide	Sec. 7.3.4 SW-846		X	X	
Salinity	SM 2520 B-2011		X		
Sampling for Low-Level Metals	EPA 1669		X		
Solids - Residue-total (TS)	SM 2540 B-2011		X		
Solids - Total Dissolved Solids (TDS)	SM 2540 C-2011	X	X		

Parameter	Method	Matrices			
		DW	NPW	SCM	Tissue
Solids - Total Fixed and Volatile Solids (TVS) [1]	SM 2540 G-2011		X		
Solids - Total Suspended Solids (TSS)	SM 2540 D-2011		X		
Solids - Volatile Residue (VS) [1]	EPA 160.4		X		
Specific Conductance	SM 2510 B-2011	X	X		
Specific Conductance (Electrode)	EPA 120.1		X		
Sulfide (Titration)	SM 4500 S^{2-} F-2011		X		
Sulfide Preparation	SM 4500-S^{2-} C-2011		X		
Sulfite-SO3 (Titration)	SM 4500 $SO3^{2-}$ B-2011		X		
Surfactants - CTAS[1]	SM 5540 D-2011		X		
Surfactants – MBAS	SM 5540 C-2011		X		
Surfactants - Separation by Sublation	SM 5540 B-2011		X		
SVOC - Base/Neutrals and Acids - GC/MS	EPA 625.1-RVE		X		
SVOC - Base/Neutrals and Acids by GC/MS	EPA 625.1		X		
SVOC - Determination by GC/MS	EPA 8270D				X
SVOC - Determination by GC/MS	EPA 8270C,D,E		X	X	
SVOC - Determination by GC/MS	EPA 8270E SIM		X	X	
SVOC - Diesel Range Organics (DRO) by GC/FID	EPA 8015C		X	X	
SVOC - Diesel Range Organics (DRO) by GC/FID	MADEP-EPH (MA-EPH)		X		
SVOC - EDB & DBCP by GC/ECD	EPA 8011		X	X	
SVOC - EDB, DBCP, TCP by GC/ECD	EPA 504.1	X			
SVOC - Explosives by HPLC[1]	EPA 8330A		X	X	
SVOC - Explosives by HPLC	EPA 8330B		X	X	
SVOC - Extractable Petroleum Hydrocarbons (EPH) Modified by GC/FID	MADEP-EPH		X	X	
SVOC - Herbicides GC	EPA 8151A		X	X	
SVOC - Organochlorine Pesticides & PCBs	EPA 608.3		X		
SVOC - Organochlorine Pesticides & PCBs	EPA 608.3-RVE		X		
SVOC - PCBs	EPA 8081		X		
SVOC - PCBs GC	EPA 8082A		X	X	X
SVOC - Pesticides GC	EPA 8081B		X	X	X
SVOC - Total Petroleum Hydrocarbons (TPH) by GC/FID	MADEP-EPH (MA-EPH)			X	

Parameter	Method	Matrices			
		DW	NPW	SCM	Tissue
SVOC - Total Petroleum Hydrocarbons (TPH) GC	FL-PRO		X	X	
SVOC - WI-Diesel Range Organics (DRO) by GC/FID	EPA 8015C		X	X	
Temperature	SM 2550 B-2010	X	X		
TKN (UV/VIS)	EPA 351.2		X	X	
TOC (Heated Persulfate - Wet Oxidation)	SM 5310 C-2011		X		
TOC (Titration)	Walkley-Black			X	
TOC (Wet Oxidation)	EPA 9060A		X	X	
Turbidity (Electrode)	EPA 180.1	X	X		
VOC - 1,4-Dioxane by GC/MS	EPA 8260B,C SIM		X	X	
VOC - 1,4-Dioxane by GC/MS	EPA 8260D SIM			X	
VOC - Alcohols and Glycols by Direct Aqueous Injection GC/FID	EPA 8015C		X	X	
VOC - Gasoline Range Organics (GRO) by GC/FID	EPA 8015C		X	X	
VOC - Gasoline Range Organics (GRO) by Purge and Trap GC/PID/FID	MADEP-VPH (MA-VPH)		X	X	
VOC - Methane, Ethane, Ethene by GC/FID	RSK - 175		X		
VOC - Oxyenate Volatile Organics by GC/MS	EPA 8260B-OXY			X	
VOC - Purgeable Organic Compounds by GC/MS	EPA 524.2	X			
VOC - Purgeable Organics by GC/MS	EPA 624.1		X		
VOC - Purgeable Organics by GC/MS[1]	SM 6200 B-2011		X		
VOC - Volatile Petroleum Hydrocarbons (VPH) by Purge & Trap GC/PID/FID	MADEP-VPH		X	X	
VOC - Volatiles by GC/MS	EPA 8260D		X	X	
VOC - WI-Gasoline Range Organics (GRO) by GC/FID[1]	EPA 8015C		X	X	

[1] = Laboratory does not hold TNI Accreditation for this test method.

7.2.2 PAS-WCOL [Charlotte Service Center]

Parameter	Method	Matrices			
		DW	NPW	SCM	Tissue
Chlorine, Residual (DPD)	SM 4500-CL G-2011		X		

Parameter	Method	Matrices			
		DW	NPW	SCM	Tissue
Specific Conductance (Electrode)	EPA 120.1		X		
Dissolved Oxygen (DO)	SM 4500-O G-2011		X		
Temperature	SM 2550 B-2010		X		
pH (Electrode)	SM 4500-H+ B-2011		X		

7.2.3 PAS-WCOL [Greenville Service Center]

Parameter	Method	Matrices			
		DW	NPW	SCM	Tissue
Chlorine, Residual (DPD)	SM 4500-CL G-2011		X		
Dissolved Oxygen (DO)	SM 4500-O G-2011		X		
Temperature	SM 2550 B-2010		X		
pH (Electrode)	SM 4500-H+ B-2011		X		

7.3 Appendix C: Glossary

This glossary provides common terms and definitions used in the laboratory. **It is not intended to be a complete list of all terms and definitions used.** The definitions have been compiled mostly from the TNI Standard and DoD QSM. Although this information has been reproduced with care, errors cannot be entirely excluded. Definitions for the same term also vary between sources. When the meaning of a term used in a laboratory document is different from this glossary or when the glossary does not include the term, the term and definition is included or defined in context in the laboratory document.

Term	Definition
3P Program	PAS-The continuous improvement program used by PAS that focuses on Process, Productivity, and Performance.
Acceptance Criteria	TNI- Specified limits placed on characteristics of an item, process, or service defined in requirement documents.
Accreditation	TNI- The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. DoD- Refers to accreditation in accordance with the DoD ELAP.
Accreditation Body (AB)	TNI- The organization having responsibility and accountability for environmental laboratory accreditation and which grants accreditation under this program. DoD- Entities recognized in accordance with the DoD-ELAP that are required to operate in accordance with ISO/IEC 17011, *Conformity assessment: General requirements for accreditation bodies accrediting conformity assessment bodies.* The AB must be a signatory, in good standing, to the International Laboratory Accreditation Cooperation (ILAC) mutual recognition arrangement (MRA) that verifies, by evaluation and peer assessment, that its signatory members are in full compliance with ISO/IEC 17011 and that its accredited laboratories comply with ISO/IEC 17025.
Accuracy	TNI- The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations; a data quality indicator.
Activity, Absolute	TNI- Rate of nuclear decay occurring in a body of material, equal to the number of nuclear disintegrations per unit time. NOTE: Activity (absolute) may be expressed in becquerels (Bq), curies (Ci), or disintegrations per minute (dpm), and multiples or submultiples of these units.

Term	Definition
Activity, Areic	TNI- Quotient of the activity of a body of material and its associated area.
Activity, Massic	TNI- Quotient of the activity of a body of material and its mass; also called specific activity.
Activity, Volumic	TNI- Quotient of the activity of a body of material and its volume; also called activity concentration. NOTE: In this module [TNI Volume 1, Module 6], unless otherwise stated, references to activity shall include absolute activity, areic activity, massic activity, and volumic activity.
Activity Reference Date	TNI- The date (and time, as appropriate to the half-life of the radionuclide) to which a reported activity result is calculated. NOTE: The sample collection date is most frequently used as the Activity Reference Date for environmental measurements, but different programs may specify other points in time for correction of results for decay and ingrowth.
Aliquot	DoD- A discrete, measured, representative portion of a sample taken for analysis.
American Society for Testing and Materials (ASTM)	An international standards organization that develops and publishes voluntary consensus standards for a wide range of materials, products, systems and services.
Analysis	DoD- A combination of sample preparation and instrument determination.
Analysis Code (Acode)	All the set parameters of a test, such as Analytes, Method, Detection Limits and Price.
Analysis Sequence	A compilation of all samples, standards and quality control samples run during a specific amount of time on a particular instrument in the order they are analyzed.
Analyst	TNI- The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.
Analyte	TNI- A substance, organism, physical parameter, property, or chemical constituent(s) for which an environmental sample is being analyzed. DoD- The specific chemicals or components for which a sample is analyzed; it may be a group of chemicals that belong to the same chemical family and are analyzed together.
Analytical Method	DoD- A formal process that identifies and quantifies the chemical components of interest (target analytes) in a sample.
Analytical Uncertainty	TNI- A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis.
Aliquot	DoD- A discrete, measured, representative portion of a sample taken for analysis.
Annual (or Annually)	Defined by PAS as every 12 months ± 30 days.
Assessment	TNI - The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its system to defined criteria (to the standards and requirements of laboratory accreditation). DoD- An all-inclusive term used to denote any of the following: audit, performance evaluation, peer review, inspection, or surveillance conducted on-site.
Atomic Absorption Spectrometer	Instrument used to measure concentration in metals samples.
Atomization	A process in which a sample is converted to free atoms.
Audit	TNI- A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives.
Batch	TNI- Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one to 20 environmental samples of the same quality systems matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours or the time-frame specified by the regulatory program. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed 20 samples.
Batch, Radiation Measurements (RMB)	TNI- An RMB is composed of 1 to 20 environmental samples that are counted directly without preliminary physical or chemical processing that affects the outcome of the test (e.g., non-destructive gamma spectrometry, alpha/beta counting of air filters, or swipes on gas proportional detectors). The samples in an RMB share similar physical and chemical parameter, and analytical configurations (e.g., analytes, geometry, calibration, and background corrections). The maximum time between the start of processing of the first and last in an RMB is 14 calendar days.

Term	Definition
Bias	TNI- The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value).
Blank	TNI and DoD- A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results (See Method Blank). DoD- Blank samples are negative control samples, which typically include field blank samples (e.g., trip blank, equipment (rinsate) blank, and temperature blank) and laboratory blank samples (e.g., method blank, reagent blank, instrument blank, calibration blank, and storage blank).
Blind Sample	A sub-sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.
BNA (Base Neutral Acid compounds)	A list of semi-volatile compounds typically analyzed by mass spectrometry methods. Named for the way they can be extracted out of environmental samples in an acidic, basic or neutral environment.
BOD (Biochemical Oxygen Demand)	Chemical procedure for determining how fast biological organisms use up oxygen in a body of water.
Calibration	TNI- A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. 1) In calibration of support equipment, the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI); 2) In calibration according to test methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.
Calibration Curve	TNI- The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.
Calibration Method	A defined technical procedure for performing a calibration.
Calibration Range	DoD- The range of values (concentrations) between the lowest and highest calibration standards of a multi-level calibration curve. For metals analysis with a single-point calibration, the low-level calibration check standard and the high standard establish the linear calibration range, which lies within the linear dynamic range.
Calibration Standard	TNI- A substance or reference material used for calibration.
Certified Reference Material (CRM)	TNI- Reference material accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute.
Chain of Custody	An unbroken trail of accountability that verifies the physical security of samples, data, and records.
Chain of Custody Form (COC)	TNI- Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and type of containers; the mode of collection, the collector, time of collection; preservation; and requested analyses.
Chemical Oxygen Demand (COD)	A test commonly used to indirectly measure the amount of organic compounds in water.
Client (referred to by ISO as Customer)	Any individual or organization for whom items or services are furnished or work performed in response to defined requirements and expectations.
Code of Federal Regulations (CFR)	A codification of the general and permanent rules published in the Federal Register by agencies of the federal government.
Comparability	An assessment of the confidence with which one data set can be compared to another. Comparable data are produced through the use of standardized procedures and techniques.
Completeness	The percent of valid data obtained from a measurement system compared to the amount of valid data expected under normal conditions. The equation for completeness is: % Completeness = (Valid Data Points/Expected Data Points)*100
Confirmation	TNI- Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to: second-column confirmation; alternate wavelength; derivatization; mass spectral interpretation; alternative detectors; or additional cleanup procedures. DoD- Includes verification of the identity and quantity of the analyte being measured by another means (e.g., by another determinative method, technology, or column). Additional cleanup procedures alone are not considered confirmation techniques.

Term	Definition
Conformance	An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements.
Congener	A member of a class of related chemical compounds (e.g., PCBs, PCDDs).
Consensus Standard	DoD- A standard established by a group representing a cross-section of a particular industry or trade, or a part thereof.
Continuing Calibration Blank (CCB)	A blank sample used to monitor the cleanliness of an analytical system at a frequency determined by the analytical method.
Continuing Calibration Check Compounds (CCC)	Compounds listed in mass spectrometry methods that are used to evaluate an instrument calibration from the standpoint of the integrity of the system. High variability would suggest leaks or active sites on the instrument column.
Continuing Calibration Verification	DoD- The verification of the initial calibration. Required prior to sample analysis and at periodic intervals. Continuing calibration verification applies to both external and internal standard calibration techniques, as well as to linear and non-linear calibration models.
Continuing Calibration Verification (CCV) Standard	Also referred to as a Calibration Verification Standard (CVS) in some methods, it is a standard used to verify the initial calibration of compounds in an analytical method. CCVs are analyzed at a frequency determined by the analytical method.
Continuous Emission Monitor (CEM)	A flue gas analyzer designed for fixed use in checking for environmental pollutants.
Continuous Improvement Plan (CIP)	The delineation of tasks for a given laboratory department or committee to achieve the goals of that department.
Contract Laboratory Program (CLP)	A national network of EPA personnel, commercial labs, and support contractors whose fundamental mission is to provide data of known and documented quality.
Contract Required Detection Limit (CRDL)	Detection limit that is required for EPA Contract Laboratory Program (CLP) contracts.
Contract Required Quantitation Limit (CRQL)	Quantitation limit (reporting limit) that is required for EPA Contract Laboratory Program (CLP) contracts.
Control Chart	A graphic representation of a series of test results, together with limits within which results are expected when the system is in a state of statistical control (see definition for Control Limit)
Control Limit	A range within which specified measurement results must fall to verify that the analytical system is in control. Control limit exceedances may require corrective action or require investigation and flagging of non-conforming data.
Correction	DoD- Action taken to eliminate a detected non-conformity.
Corrective Action	DoD- The action taken to eliminate the causes of an existing non-conformity, defect, or other undesirable situation in order to prevent recurrence. A root cause analysis may not be necessary in all cases.
Corrective and Preventative Action (CAPA)	The primary management tools for bringing improvements to the quality system, to the management of the quality system's collective processes, and to the products or services delivered which are an output of established systems and processes.
Critical Value	TNI- Value to which a measurement result is compared to make a detection decision (also known as critical level or decision level). NOTE: The Critical Value is designed to give a specified low probability α of false detection in an analyte-free sample, which implies that a result that exceeds the Critical Value, gives high confidence $(1 - \alpha)$ that the radionuclide is actually present in the material analyzed. For radiometric methods, α is often set at 0.05.
Customer	DoD- Any individual or organization for which products or services are furnished or work performed in response to defined requirements and expectations.
Data Integrity	TNI- The condition that exists when data are sound, correct, and complete, and accurately reflect activities and requirements.
Data Quality Objective (DQO)	Systematic strategic planning tool based on the scientific method that identifies and defines the type, quality, and quantity of data needed to satisfy a specified use or end user.
Data Reduction	TNI- The process of transforming the number of data items by arithmetic or statistical calculation, standard curves, and concentration factors, and collating them into a more usable form.
Definitive Data	DoD- Analytical data of known quantity and quality. The levels of data quality on precision and bias meet the requirements for the decision to be made. Data that is suitable for final decision-making.

Term	Definition
Demonstration of Capability (DOC)	TNI- A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. DoD- A procedure to establish the ability of the analyst to generate analytical results by a specific method that meet measurement quality objectives (e.g., for precision and bias).
Department of Defense (DoD)	An executive branch department of the federal government of the United States charged with coordinating and supervising all agencies and functions of the government concerned directly with national security.
Detection Limit (DL)	DoD- The smallest analyte concentration that can be demonstrated to be different than zero or a blank concentration with 99% confidence. At the DL, the false positive rate (Type 1 error) is 1%. A DL may be used as the lowest concentration for reliably reporting a detection of a specific analyte in a specific matrix with a specific method with 99% confidence.
Detection Limit (DL) for Safe Drinking Water Act (SDWA) Compliance	TNI- Laboratories that analyze drinking-water samples for SDWA compliance monitoring must use methods that provide sufficient detection capability to meet the detection limit requirements established in 40 CFR 141. The SDWA DL for radioactivity is defined in 40 CFR Part 141.25.c as the radionuclide concentration, which can be counted with a precision of plus or minus 100% at the 95% confidence level (1.96σ where σ is the standard deviation of the net counting rate of the sample).
Deuterated Monitoring Compounds (DMCs)	DoD- SIM specific surrogates as specified for GC/MS SIM analysis.
Diesel Range Organics (DRO)	A range of compounds that denote all the characteristic compounds that make up diesel fuel (range can be state or program specific).
Digestion	DoD- A process in which a sample is treated (usually in conjunction with heat and acid) to convert the target analytes in the sample to a more easily measured form.
Document Control	The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed.
Documents	DoD- Written components of the laboratory management system (e.g., policies, procedures, and instructions).
Dry Weight	The weight after drying in an oven at a specified temperature.
Duplicate (also known as Replicate or Laboratory Duplicate)	The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results of duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory.
Electron Capture Detector (ECD)	Device used in GC methods to detect compounds that absorb electrons (e.g., PCB compounds).
Electronic Data Deliverable (EDD)	A summary of environmental data (usually in spreadsheet form) which clients request for ease of data review and comparison to historical results.
Eluent	A solvent used to carry the components of a mixture through a stationary phase.
Elute	To extract, specifically, to remove (absorbed material) from an absorbent by means of a solvent.
Elution	A process in which solutes are washed through a stationary phase by movement of a mobile phase.
Environmental Data	DoD- Any measurements or information that describe environmental processes, locations, or conditions; ecological or health effects and consequences; or the performance of environmental technology.
Environmental Monitoring	The process of measuring or collecting environmental data.
Environmental Protection Agency (EPA)	An agency of the federal government of the United States which was created for the purpose of protecting human health and the environment by writing and enforcing regulations based on laws passed by Congress.

Term	Definition
Environmental Sample	A representative sample of any material (aqueous, non-aqueous, or multimedia) collected from any source for which determination of composition or contamination is requested or required. Environmental samples can generally be classified as follows: • Non Potable Water (Includes surface water, ground water, effluents, water treatment chemicals, and TCLP leachates or other extracts) • Drinking Water - Delivered (treated or untreated) water designated as potable water • Water/Wastewater - Raw source waters for public drinking water supplies, ground waters, municipal influents/effluents, and industrial influents/effluents • Sludge - Municipal sludges and industrial sludges. • Soil - Predominately inorganic matter ranging in classification from sands to clays. • Waste - Aqueous and non-aqueous liquid wastes, chemical solids, and industrial liquid and solid wastes
Equipment Blank	A sample of analyte-free media used to rinse common sampling equipment to check effectiveness of decontamination procedures.
Extracted Internal Standard Analyte	Isotopically labeled analogs of analytes of interest added to all standards, blanks and samples analyzed. Added to samples and batch QC samples prior to the first step of sample extraction and to standards and instrument blanks prior to analysis. Used for isotope dilution methods.
Facility	A distinct location within the company that has unique certifications, personnel and waste disposal identifications.
False Negative	DoD- A result that fails to identify (detect) an analyte or reporting an analyte to be present at or below a level of interest when the analyte is actually above the level of interest.
False Positive	DoD- A result that erroneously identifies (detects) an analyte or reporting an analyte to be present above a level of interest when the analyte is actually present at or below the level of interest.
Field Blank	A blank sample prepared in the field by filling a clean container with reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken.
Field Measurement	Determination of physical, biological, or radiological properties, or chemical constituents that are measured on-site, close in time and sPAS to the matrices being sampled/measured, following accepted test methods. This testing is performed in the field outside of a fixed-laboratory or outside of an enclosed structure that meets the requirements of a mobile laboratory.
Field of Accreditation	TNI- Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.
Field of Proficiency Testing (FoPT)	TNI- Matrix, technology/method, analyte combinations for which the composition, spike concentration ranges and acceptance criteria have been established by the PTPEC.
Finding	TNI- An assessment conclusion referenced to a laboratory accreditation standard and supported by objective evidence that identifies a deviation from a laboratory accreditation standard requirement. DoD- An assessment conclusion that identifies a condition having a significant effect on an item or activity. An assessment finding may be positive, negative, or neutral and is normally accompanied by specific examples of the observed condition. The finding must be linked to a specific requirement (e.g., this standard, ISO requirements, analytical methods, contract specifications, or laboratory management systems requirements).
Flame Atomic Absorption Spectrometer (FAA)	Instrumentation used to measure the concentration of metals in an environmental sample based on the fact that ground state metals absorb light at different wavelengths. Metals in a solution are converted to the atomic state by use of a flame.
Flame Ionization Detector (FID)	A type of gas detector used in GC analysis where samples are passed through a flame which ionizes the sample so that various ions can be measured.
Gas Chromatography (GC)	Instrumentation which utilizes a mobile carrier gas to deliver an environmental sample across a stationary phase with the intent to separate compounds out and measure their retention times.
Gas Chromatograph/ Mass Spectrometry (GC/MS)	In conjunction with a GC, this instrumentation utilizes a mass spectrometer which measures fragments of compounds and determines their identity by their fragmentation patterns (mass spectra).
Gasoline Range Organics (GRO)	A range of compounds that denote all the characteristic compounds that make up gasoline (range can be state or program specific).
Graphite Furnace Atomic Absorption Spectrometry (GFAA)	Instrumentation used to measure the concentration of metals in an environmental sample based on the absorption of light at different wavelengths that are characteristic of different analytes.

Term	Definition
High Pressure Liquid Chromatography (HPLC)	Instrumentation used to separate, identify and quantitate compounds based on retention times which are dependent on interactions between a mobile phase and a stationary phase.
Holding Time	TNI- The maximum time that can elapse between two specified activities. 40 CFR Part 136- The maximum time that samples may be held prior to preparation and/or analysis as defined by the method and still be considered valid or not compromised. For sample prep purposes, hold times are calculated using the time of the start of the preparation procedure. DoD- The maximum time that may elapse from the time of sampling to the time of preparation or analysis, or from preparation to analysis, as appropriate.
Homogeneity	The degree to which a property or substance is uniformly distributed throughout a sample.
Homologue	One in a series of organic compounds in which each successive member has one more chemical group in its molecule than the next preceding member. For instance, methanol, ethanol, propanol, butanol, etc., form a homologous series.
Improper Actions	DoD- Intentional or unintentional deviations from contract-specified or method-specified analytical practices that have not been authorized by the customer (e.g., DoD or DOE).
Incremental Sampling Method (ISM)	Soil preparation for large volume (1 kg or greater) samples.
In-Depth Data Monitoring	TNI- When used in the context of data integrity activities, a review and evaluation of documentation related to all aspects of the data generation process that includes items such as preparation, equipment, software, calculations, and quality controls. Such monitoring shall determine if the laboratory uses appropriate data handling, data use and data reduction activities to support the laboratory's data integrity policies and procedures.
Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)	Analytical technique used for the detection of trace metals which uses plasma to produce excited atoms that emit radiation of characteristic wavelengths.
Inductively Coupled Plasma- Mass Spectrometry (ICP/MS)	An ICP that is used in conjunction with a mass spectrometer so that the instrument is not only capable of detecting trace amounts of metals and non-metals but is also capable of monitoring isotopic speciation for the ions of choice.
Infrared Spectrometer (IR)	An instrument that uses infrared light to identify compounds of interest.
Initial Calibration (ICAL)	The process of analyzing standards, prepared at specified concentrations, to define the quantitative response relationship of the instrument to the analytes of interest. Initial calibration is performed whenever the results of a calibration verification standard do not conform to the requirements of the method in use or at a frequency specified in the method.
Initial Calibration Blank (ICB)	A blank sample used to monitor the cleanliness of an analytical system at a frequency determined by the analytical method. This blank is specifically run in conjunction with the Initial Calibration Verification (ICV) where applicable.
Initial Calibration Verification (ICV)	DoD- Verifies the initial calibration with a standard obtained or prepared from a source independent of the source of the initial calibration standards to avoid potential bias of the initial calibration.
Injection Internal Standard Analyte	Isotopically labeled analogs of analytes of interest (or similar in physiochemical properties to the target analytes but with a distinct response) to be quantitated. Added to all blanks, standards, samples and batch QC after extraction and prior to analysis.
Instrument Blank	A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination.
Instrument Detection Limits (IDLs)	Limits determined by analyzing a series of reagent blank analyses to obtain a calculated concentration. IDLs are determined by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day.
Interference, spectral	Occurs when particulate matter from the atomization scatters incident radiation from the source or when the absorption or emission from an interfering species either overlaps or is so close to the analyte wavelength that resolution becomes impossible.
Interference, chemical	Results from the various chemical processes that occur during atomization and later the absorption characteristics of the analyte.
Internal Standard	TNI and DoD- A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.

Term	Definition
International Organization for Standardization (ISO)	An international standard-setting body composed of representatives from various national standards organizations.
Intermediate Standard Solution	Reference solutions prepared by dilution of the stock solutions with an appropriate solvent.
International System of Units (SI)	The coherent system of units adopted and recommended by the General Conference on Weights and Measures.
Ion Chromatography (IC)	Instrumentation or process that allows the separation of ions and molecules based on the charge properties of the molecules.
Isomer	One of two or more compounds, radicals, or ions that contain the same number of atoms of the same element but differ in structural arrangement and properties. For example, hexane (C6H14) could be n-hexane, 2-methylpentane, 3-methylpentane, 2,3-dimethylbutane, 2,2-dimethylbutane.
Laboratory	A body that calibrates and/or performs testing..
Laboratory Control Sample (LCS)	TNI- (also known as laboratory fortified blank (LFB), spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes and taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to evaluate the performance of all or a portion of the measurement system.
Laboratory Duplicate	Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.
Laboratory Information Management System (LIMS)	DoD- The entirety of an electronic data system (including hardware and software) that collects, analyzes, stores, and archives electronic records and documents.
Learning Management System (LMS)	A web-based database used by the laboratories to track and document training activities. The system is administered by the corporate training department and each laboratory's learn centers are maintained by a local administrator.
Legal Chain-of-Custody Protocols	TNI- Procedures employed to record the possession of samples from the time of sampling through the retention time specified by the client or program. These procedures are performed at the special request of the client and include the use of a Chain-of-Custody (COC) Form that documents the collection, transport, and receipt of compliance samples by the laboratory. In addition, these protocols document all handling of the samples within the laboratory.
Limit(s) of Detection (LOD)	TNI- The minimum result, which can be reliably discriminated from a blank with predetermined confidence level. DoD- The smallest concentration of a substance that must be present in a sample in order to be detected at the DL with 99% confidence. At the LOD, the false negative rate (Type II error) is 1%. A LOD may be used as the lowest concentration for reliably reporting a non-detect of a specific analyte in a specific matrix with a specific method at 99% confidence.
Limit(s) of Quantitation (LOQ)	TNI- The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. DoD- The smallest concentration that produces a quantitative result with known and recorded precision and bias. For DoD/DOE projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard and within the calibration range.
Linear Dynamic Range	DoD- Concentration range where the instrument provides a linear response.
Liquid chromatography/ tandem mass spectrometry (LC/MS/MS)	Instrumentation that combines the physical separation techniques of liquid chromatography with the mass analysis capabilities of mass spectrometry.
Lot	TNI- A definite amount of material produced during a single manufacturing cycle, and intended to have uniform character and quality.
Management	Those individuals directly responsible and accountable for planning, implementing, and assessing work.
Management System	System to establish policy and objectives and to achieve those objectives.
Manager (however named)	The individual designated as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual.
Matrix	TNI- The substrate of a test sample.
Matrix Duplicate	TNI- A replicate matrix prepared in the laboratory and analyzed to obtain a measure of precision.

Term	Definition
Matrix Spike (MS) (spiked sample or fortified sample)	TNI- A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.
Matrix Spike Duplicate (MSD) (spiked sample or fortified sample duplicate)	TNI- A replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.
Measurement Performance Criteria (MPC)	DoD- Criteria that may be general (such as completion of all tests) or specific (such as QC method acceptance limits) that are used by a project to judge whether a laboratory can perform a specified activity to the defined criteria.
Measurement Quality Objective (MQO)	TNI- The analytical data requirements of the data quality objectives are project- or program-specific and can be quantitative or qualitative. MQOs are measurement performance criteria or objectives of the analytical process. Examples of quantitative MQOs include statements of required analyte detectability and the uncertainty of the analytical protocol at a specified radionuclide activity, such as the action level. Examples of qualitative MQOs include statements of the required specificity of the analytical protocol, e.g., the ability to analyze for the radionuclide of interest given the presence of interferences.
Measurement System	TNI- A method, as implemented at a particular laboratory, and which includes the equipment used to perform the test and the operator(s). DoD- A test method, as implemented at a particular laboratory, and which includes the equipment used to perform the sample preparation and test and the operator(s).
Measurement Uncertainty	DoD- An estimate of the error in a measurement often stated as a range of values that contain the true value within a certain confidence level. The uncertainty generally includes many components which may be evaluated from experimental standard deviations based on repeated observations or by standard deviations evaluated from assumed probability distributions based on experience or other information. For DoD/DOE, a laboratory's Analytical Uncertainty (such as use of LCS control limits) can be reported as the minimum uncertainty.
Method	TNI- A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, quantification), systematically presented in the order in which they are to be executed.
Method Blank	TNI- A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.
Method Detection Limit (MDL)	TNI- One way to establish a Detection Limit; defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.
Method of Standard Additions	A set of procedures adding one or more increments of a standard solution to sample aliquots of the same size in order to overcome inherent matrix effects. The procedures encompass the extrapolation back to obtain the sample concentration.
Minimum Detectable Activity (MDA)	TNI- Estimate of the smallest true activity that ensures a specified high confidence, $1 - \beta$, of detection above the Critical Value, and a low probability β of false negatives below the Critical Value. For radiometric methods, β is often set at 0.05. NOTE 1: The MDS is a measure of the detection capability of a measurement process and as such, it is an a priori concept. It may be used in the selection of methods to meet specified MQOs. Laboratories may also calculate a "sample specific" MDA, which indicates how well the measurement process is performing under varying real-world measurement conditions, when sample-specific characteristics (e.g., interferences) may affect the detection capability. However, the MDA must never be used instead of the Critical Value as a detection threshold. NOTE 2: For the purpose of this Standard, the terms MDA and minimum detectable concentration (MDC) are equivalent.
Minimum Reporting Limit (MRL)	the lowest concentration of standard used for calibration – Drinking Water Manual
MintMiner	Commercial software program used to scan large amounts of chromatographic data to monitor for errors or data integrity issues.
Mobile Laboratory	TNI- A portable enclosed structure with necessary and appropriate accommodation and environmental conditions for a laboratory, within which testing is performed by analysts. Examples include but are not limited to trailers, vans, and skid-mounted structures configured to house testing equipment and personnel.

Term	Definition
National Environmental Laboratory Accreditation Conference (NELAC)	See definition of The NELAC Institute (TNI).
National Institute of Occupational Safety and Health (NIOSH)	National institute charged with the provision of training, consultation and information in the area of occupational safety and health.
National Institute of Standards and Technology (NIST)	TNI- A federal agency of the US Department of Commerce's Technology Administration that is designed as the United States national metrology institute (or NMI).
National Pollutant Discharge Elimination System (NPDES)	A permit program that controls water pollution by regulating point sources that discharge pollutants into U.S. waters.
Negative Control	Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.
Nitrogen Phosphorus Detector (NPD)	A detector used in GC analyses that utilizes thermal energy to ionize an analyte. With this detector, nitrogen and phosphorus can be selectively detected with a higher sensitivity than carbon.
Nonconformance	An indication or judgment that a product or service has not met the requirement of the relevant specifications, contract, or regulation; also the state of failing to meet the requirements.
Not Detected (ND)	The result reported for a compound when the detected amount of that compound is less than the method reporting limit.
Operator Aid	DoD- A technical posting (such as poster, operating manual, or notepad) that assists workers in performing routine tasks. All operator aids must be controlled documents (i.e., a part of the laboratory management system).
Performance Based Measurement System (PBMS)	An analytical system wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner.
Physical Parameter	TNI- A measurement of a physical characteristic or property of a sample as distinguished from the concentrations of chemical and biological components.
Photo-ionization Detector (PID)	An ion detector which uses high-energy photons, typically in the ultraviolet range, to break molecules into positively charged ions.
Polychlorinated Biphenyls (PCB)	A class of organic compounds that were used as coolants and insulating fluids for transformers and capacitors. The production of these compounds was banned in the 1970's due to their high toxicity.
Positive Control	Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.
Post-Digestion Spike	A sample prepared for metals analyses that has analytes spike added to determine if matrix effects may be a factor in the results.
Power of Hydrogen (pH)	The measure of acidity or alkalinity of a solution.
Practical Quantitation Limit (PQL)	Another term for a method reporting limit. The lowest reportable concentration of a compound based on parameters set up in an analytical method and the laboratory's ability to reproduce those conditions.
Precision	TNI- The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.
Preservation	TNI and DoD- Any conditions under which a sample must be kept in order to maintain chemical, physical, and/or biological integrity prior to analysis.
Primary Accreditation Body (Primary AB)	TNI- The accreditation body responsible for assessing a laboratory's total quality system, on-site assessment, and PT performance tracking for fields of accreditation.
Procedure	TNI- A specified way to carry out an activity or process. Procedures can be documented or not.
Proficiency Testing (PT)	TNI- A means to evaluate a laboratory's performance under controlled conditions relative to a given set of criteria, through analysis of unknown samples provided by an external source.
Proficiency Testing Program (PT Program)	TNI- The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories.
Proficiency Testing Provider (PT Provider)	TNI- A person or organization accredited by a TNI-approved Proficiency Testing Provider Accreditor to operate a TNI-compliant PT Program.

Term	Definition
Proficiency Testing Provider Accreditor (PTPA)	TNI- An organization that is approved by TNI to accredit and monitor the performance of proficiency testing providers.
Proficiency Testing Reporting Limit (PTRL)	TNI- A statistically derived value that represents the lowest acceptable concentration for an analyte in a PT sample, if the analyte is spiked into the PT sample. The PTRLs are specified in the TNI FoPT tables.
Proficiency Testing Sample (PT)	TNI- A sample, the composition of which is unknown to the laboratory, and is provided to test whether the laboratory can produce analytical results within the specified acceptance criteria.
Proficiency Testing (PT) Study	TNI- a) Scheduled PT Study: A single complete sequence of circulation and scoring of PT samples to all participants in a PT program. The study must have the same pre-defined opening and closing dates for all participants; b) Supplemental PT Study: A PT sample that may be from a lot previously released by a PT Provider that meets the requirements for supplemental PT samples given in Volume 3 of this Standard [TNI] but that does not have a pre-determined opening date and closing date.
Proficiency Testing Study Closing Date	TNI- a) Scheduled PT Study: The calendar date by which all participating laboratories must submit analytical results for a PT sample to a PT Provider; b) Supplemental PT Study: The calendar date a laboratory submits the results for a PT sample to the PT Provider.
Proficiency Testing Study Opening Date	TNI- a) Scheduled PT Study: The calendar date that a PT sample is first made available to all participants of the study by a PT Provider; b) Supplemental PT Study: The calendar date the PT Provider ships the sample to a laboratory.
Protocol	TNI- A detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) that must be strictly followed.
Qualitative Analysis	DoD- Analysis designed to identify the components of a substance or mixture.
Quality Assurance (QA)	TNI- An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client.
Quality Assurance Manual (QAM)	A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.
Quality Assurance Project Plan (QAPP)	A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved.
Quality Control (QC)	TNI- The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality.
Quality Control Sample (QCS)	TNI- A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control.
Quality Manual	TNI- A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.
Quality System	TNI and DoD- A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required quality assurance and quality control activities.

Term	Definition
Quality System Matrix	TNI and DoD- These matrix definitions shall be used for purposes of batch and quality control requirements and may be different from a field of accreditation matrix: • **Air and Emissions**: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device • **Aqueous**: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, groundwater effluents, and TCLP or other extracts. • **Biological Tissue**: Any sample of a biological origin such as fish tissue, shellfish or plant material. Such samples shall be grouped according to origin. • **Chemical Waste**: A product or by-product of an industrial process that results in a matrix not previously defined. • **Drinking Water**: Any aqueous sample that has been designated a potable or potentially potable water source. • **Non-aqueous liquid**: Any organic liquid with <15% settleable solids • **Saline/Estuarine**: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake. • **Solids**: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.
Quantitation Range	DoD- The range of values (concentrations) in a calibration curve between the LOQ and the highest successively analyzed initial calibration standard used to relate instrument response to analyte concentration. The quantitation range (adjusted for initial sample volume/weight, concentration/dilution and final volume) lies within the calibration range.
Quantitative Analysis	DoD- Analysis designed to determine the amounts or proportions of the components of a substance.
Random Error	The EPA has established that there is a 5% probability that the results obtained for any one analyte will exceed the control limits established for the test due to random error. As the number of compounds measured increases in a given sample, the probability for statistical error also increases.
Raw Data	TNI- The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records.
Reagent Blank (method reagent blank)	A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps.
Reagent Grade	Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents that conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.
Records	DoD- The output of implementing and following management system documents (e.g., test data in electronic or hand-written forms, files, and logbooks).
Reference Material	TNI- Material or substance one or more of whose property values are sufficiently homogenized and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.
Reference Method	TNI- A published method issued by an organization generally recognized as competent to do so. (When the ISO language refers to a "standard method", that term is equivalent to "reference method"). When a laboratory is required to analyze by a specified method due to a regulatory requirement, the analyte/method combination is recognized as a reference method. If there is no regulatory requirement for the analyte/method combination, the analyte/method combination is recognized as a reference method if it can be analyzed by another reference method of the same matrix and technology.
Reference Standard	TNI- Standard used for the calibration of working measurement standards in a given organization or at a given location.
Relative Percent Difference (RPD)	A measure of precision defined as the difference between two measurements divided by the average concentration of the two measurements.
Reporting Limit (RL)	The level at which method, permit, regulatory and customer-specific objectives are met. The reporting limit may never be lower than the Limit of Detection (i.e., statistically determined MDL). Reporting limits are corrected for sample amounts, including the dry weight of solids, unless otherwise specified. There must be a sufficient buffer between the Reporting Limit and the MDL. DoD- A customer-specified lowest concentration value that meets project requirements for quantitative data with known precision and bias for a specific analyte in a specific matrix.

Term	Definition
Reporting Limit Verification Standard (RLVS)	A standard analyzed at the reporting limit for an analysis to verify the laboratory's ability to report to that level.
Representativeness	A quality element related to the ability to collect a sample reflecting the characteristics of the part of the environment to be assessed. Sample representativeness is dependent on the sampling techniques specified in the project work plan.
Requirement	Denotes a mandatory specification; often designated by the term "shall".
Retention Time	The time between sample injection and the appearance of a solute peak at the detector.
Revocation	TNI- The total or partial withdrawal of a laboratory's accreditation by an accreditation body.
Sample	Portion of material collected for analysis, identified by a single, unique alphanumeric code. A sample may consist of portions in multiple containers, if a single sample is submitted for multiple or repetitive analysis.
Sample Condition Upon Receipt Form (SCURF)	Form used by sample receiving personnel to document the condition of sample containers upon receipt to the laboratory (used in conjunction with a COC).
Sample Delivery Group (SDG)	A unit within a single project that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer field samples within a project, received over a period of up to 14 calendar days. Data from all samples in an SDG are reported concurrently.
Sample Receipt Form (SRF)	Letter sent to the client upon login to show the tests requested and pricing.
Sample Tracking	Procedures employed to record the possession of the samples from the time of sampling until analysis, reporting and archiving. These procedures include the use of a chain-of-custody form that documents the collection, transport, and receipt of compliance samples to the laboratory. In addition, access to the laboratory is limited and controlled to protect the integrity of the samples.
Sampling	TNI- Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.
Selected Ion Monitoring (SIM)	A mode of analysis in mass spectrometry where the detector is set to scan over a very small mass range, typically one mass unit. The narrower the range, the more sensitive the detector. DoD- Using GC/MS, characteristic ions specific to target compounds are detected and used to quantify in applications where the normal full scan mass spectrometry results in excessive noise.
Selectivity	TNI- The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system.
Sensitivity	TNI- The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.
Serial Dilution	The stepwise dilution of a substance in a solution.
Shall	Denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification as long as the requirement is fulfilled.
Should	Denotes a guideline or recommendation whenever noncompliance with the specification is permissible.
Signal-to-Noise Ratio (S/N)	DoD- A measure of signal strength relative to background noise. The average strength of the noise of most measurements is constant and independent of the magnitude of the signal. Thus, as the quantity being measured (producing the signal) decreases in magnitude, S/N decreases and the effect of the noise on the relative error of a measurement increases.
Source Water	TNI- When sampled for drinking water compliance, untreated water from streams, rivers, lakes, or underground aquifers, which is used to supply private and public drinking water supplies.
Spike	A known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.
Standard (Document)	TNI- The document describing the elements of a laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies.
Standard (Chemical)	Standard samples are comprised of a known amount of standard reference material in the matrix undergoing analysis. A standard reference material is a certified reference material produced by US NIST and characterized for absolute content, independent of analytical test method.
Standard Blank (or Reagent Blank)	A calibration standard consisting of the same solvent/reagent matrix used to prepare the calibration standards without the analytes. It is used to construct the calibration curve by establishing instrument background.

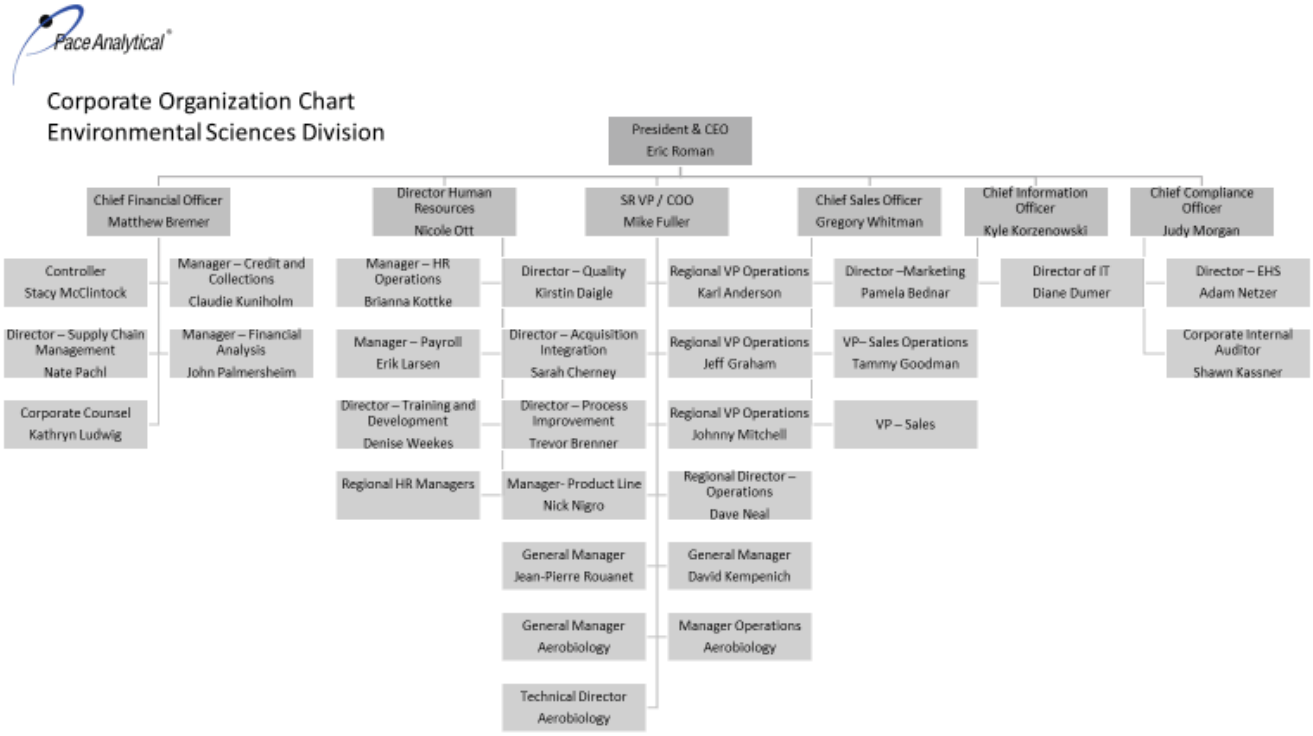
Term	Definition
Standard Method	A test method issued by an organization generally recognized as competent to do so.
Standard Operating Procedure (SOP)	TNI- A written document that details the method for an operation, analysis, or action with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks.
Standard Reference Material (SRM)	A certified reference material produced by the US NIST or other equivalent organization and characterized for absolute content, independent of analytical method.
Statement of Qualifications (SOQ)	A document that lists information about a company, typically the qualifications of that company to compete on a bid for services.
Stock Standard	A concentrated reference solution containing one or more analytes prepared in the laboratory using an assayed reference compound or purchased from a reputable commercial source.
Storage Blank	DoD- A sample of analyte-free media prepared by the laboratory and retained in the sample storage area of the laboratory. A storage blank is used to record contamination attributable to sample storage at the laboratory.
Supervisor	The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses.
Surrogate	DoD- A substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for quality control purposes.
Suspension	TNI- The temporary removal of a laboratory's accreditation for a defined period of time, which shall not exceed 6 months or the period of accreditation, whichever is longer, in order to allow the laboratory time to correct deficiencies or area of non-conformance with the Standard.
Systems Audit	An on-site inspection or assessment of a laboratory's quality system.
Target Analytes	DoD- Analytes or chemicals of primary concern identified by the customer on a project-specific basis.
Technical Director	Individual(s) who has overall responsibility for the technical operation of the environmental testing laboratory.
Technology	TNI- A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.
Test	A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate.
Test Method	DoD- A definitive procedure that determines one or more characteristics of a given substance or product.
Test Methods for Evaluating Solid Waste, Physical/ Chemical (SW-846)	EPA Waste's official compendium of analytical and sampling methods that have been evaluated and approved for use in complying with RCRA regulations.
Test Source	TNI- A radioactive source that is tested, such as a sample, calibration standard, or performance check source. A Test Source may also be free of radioactivity, such as a Test Source counted to determine the subtraction background, or a short-term background check.
The NELAC Institute (TNI)	A non-profit organization whose mission is to foster the generation of environmental data of known and documented quality through an open, inclusive, and transparent process that is responsive to the needs of the community. Previously known as NELAC (National Environmental Laboratory Accreditation Conference).
Total Petroleum Hydrocarbons (TPH)	A term used to denote a large family of several hundred chemical compounds that originate from crude oil. Compounds may include gasoline components, jet fuel, volatile organics, etc.
Toxicity Characteristic Leaching Procedure (TCLP)	A solid sample extraction method for chemical analysis employed as an analytical method to simulate leaching of compounds through a landfill.
Traceability	TNI- The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical conditions or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project.
Training Document	A training resource that provides detailed instructions to execute a specific method or job function.

Term	Definition
Trip Blank	This blank sample is used to detect sample contamination from the container and preservative during transport and storage of the sample. A cleaned sample container is filled with laboratory reagent water and the blank is stored, shipped, and analyzed with its associated samples.
Tuning	A check and/or adjustment of instrument performance for mass spectrometry as required by the method.
Ultraviolet Spectrophotometer (UV)	Instrument routinely used in quantitative determination of solutions of transition metal ions and highly conjugated organic compounds.
Uncertainty, Counting	TNI- The component of Measurement Uncertainty attributable to the random nature of radioactive decay and radiation counting (often estimated as the square root of observed counts (MARLAP). Older references sometimes refer to this parameter as Error, Counting Error or Count Error (c.f., Total Uncertainty).
Uncertainty, Expanded	TNI- The product of the Standard Uncertainty and a coverage factor, k, which is chosen to produce an interval about the result that has a high probability of containing the value of the measurand (c.f., Standard Uncertainty). NOTE: Radiochemical results are generally reported in association with the Total Uncertainty. Either if these estimates of uncertainty can be reported as the Standard Uncertainty (one-sigma) or as an Expanded Uncertainty (k-sigma, where k > 1).
Uncertainty, Measurement	TNI- Parameter associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand.
Uncertainty, Standard	TNI- An estimate of the Measurement Uncertainty expressed as a standard deviation (c.f., Expanded Uncertainty).
Uncertainty, Total	TNI- An estimate of the Measurement Uncertainty that accounts for contributions from all significant sources of uncertainty associated with the analytical preparation and measurement of a sample. Such estimates are also commonly referred to as Combined Standard Uncertainty or Total Propagated Uncertainty, and in some older references as the Total Propagated Error, among other similar items (c.f., Counting Uncertainty).
Unethical actions	DoD- Deliberate falsification of analytical or quality control results where failed method or contractual requirements are made to appear acceptable.
United States Department of Agriculture (USDA)	A department of the federal government that provides leadership on food, agriculture, natural resources, rural development, nutrition and related issues based on public policy, the best available science, and effective management.
United States Geological Survey (USGS)	Program of the federal government that develops new methods and tools to supply timely, relevant, and useful information about the Earth and its processes.
Unregulated Contaminant Monitoring Rule (UCMR)	EPA program to monitor unregulated contaminants in drinking water.
Validation	DoD- The confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled.
Verification	TNI- Confirmation by examination and objective evidence that specified requirements have been met. In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.
Voluntary Action Program (VAP)	A program of the Ohio EPA that gives individuals a way to investigate possible environmental contamination, clean it up if necessary and receive a promise from the State of Ohio that no more cleanup is needed.
Whole Effluent Toxicity (WET)	The aggregate toxic effect to aquatic organisms from all pollutants contained in a facility's wastewater (effluent).

7.4 Appendix D: Organization Chart(s)

7.4.1 Corporate Organization Chart

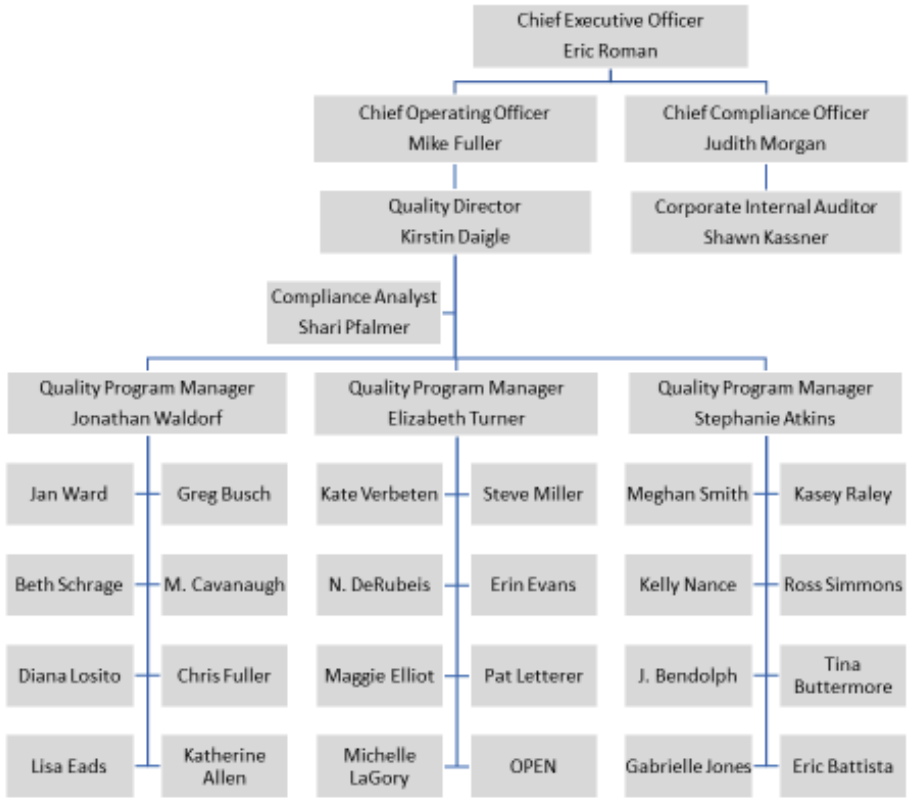
Disclaimer: The following organization chart shows the structure of the and the relationships and relative ranks of its parts and positions/jobs in place on the date this version of this manual was published. This information is subject to change; contact the Quality Manager for the most current version.



7.4.2 Quality Systems Management

Disclaimer: The following organization chart shows the structure of the and the relationships and relative ranks of its parts and positions/jobs in place on the date this version of this manual was published. This information is subject to change; contact the Quality Manager for the most current version.

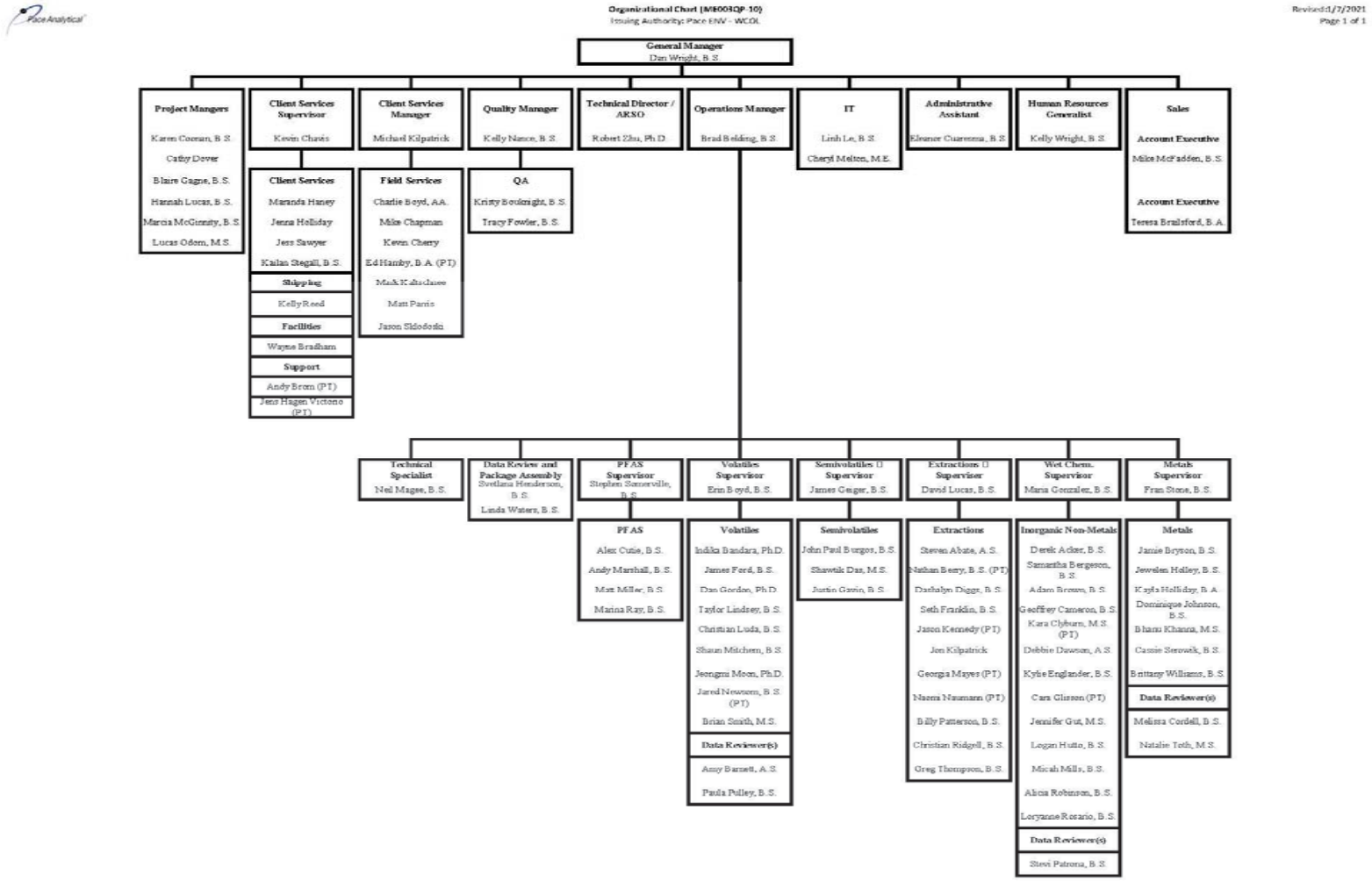
Quality Systems Management
Environmental Sciences Division



Local Quality Managers

Each QM has a direct reporting relationship to a Quality Program Manager and an indirect reporting relationship to the General Manager of each location for which the QM is assigned.

7.4.3 PAS-WCOL [Analytical Laboratory] – Organization Chart

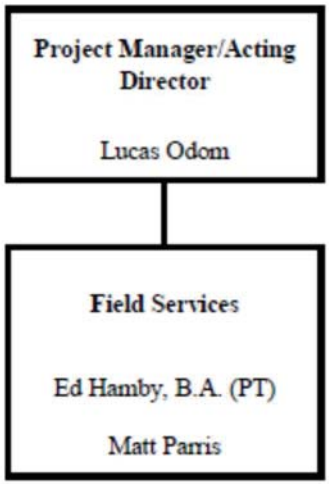


7.4.4 PAS-WCOL [Charlotte Service Center] – Organization Chart

Organizational Chart
PAS ENV - WCOL [Charlotte Service Center]

7.4.5 PAS-WCOL [Greenville Service Center] – Organization Chart

Organizational Chart
PAS ENV - WCOL [Greenville Service Center]



7.5 Appendix E: Equipment Listing

The equipment listed represents equipment were held by each location on the effective date of this manual. This information is subject to change without notice. External parties should contact the location for the most current information.

7.5.1 PAS-WCOL [Analytical Laboratory]

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Composite Sampler	Isco	3710	218J00263	09/01/2018	New	Field Services - Columbia	Composite Sampler #18-0901	EQLIMS
Composite Sampler	Isco	3710	205E01372	Unknown	Unknown	Field Services - Columbia	Composite Sampler #18-1115A	EQLIMS
Composite Sampler	Isco	3710	200A01650	Unknown	Unknown	Field Services - Columbia	Composite Sampler #18-1115B	EQLIMS
Composite Sampler	Isco	3710	212B00320	Unknown	Unknown	Field Services - Columbia	Composite Sampler #18-1115C	EQLIMS
Composite Sampler	Isco	3710	205K0152	Unknown	Unknown	Field Services - Columbia	Composite Sampler #18-1115E	EQLIMS
Composite Sampler	Isco	3710	213F01221	Unknown	Unknown	Field Services - Columbia	Composite Sampler #18-1115F	EQLIMS
Composite Sampler	Isco	3710	218M00077	01/09/2019	New	Field Services - Columbia	Composite Sampler #19-0109B	EQLIMS
DO Meter	YSI	Pro ODO	18B103680	03/08/2018	New	Field Services - Columbia	DO Meter #6	EQLIMS
DO Probe	YSI	Pro ODO Probe	18B103921	03/08/2018	New	Field Services - Columbia	DO Meter #6 Probe	EQLIMS
Multimeter	YSI	Pro1030	16K102983	09/11/2016	Unknown	Field Services - Columbia	Multimeter #2	EQLIMS

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Peristaltic Pump	Isco	150	203G01007	2004	Unknown	Field Services - Columbia	Peristaltic Pump #1	NA
pH Meter	ThermoFisher Scientific	Orion Star A321	G07576	10/06/2016	New	Field Services - Columbia	pH Meter #101	EQLIMS
pH Meter	YSI	Pro 10	18B102977	03/08/2018	New	Field Services - Columbia	pH Meter #104	EQLIMS
pH Meter	ThermoFisher Scientific	STARA3210	G12519	11/25/2019	New	Field Services - Columbia	pH Meter #19-1125	EQLIMS
pH Meter	ThermoFisher Scientific	Orion Star A321	G03795	02/04/2013	Unknown	Field Services - Columbia	pH Meter #37	EQLIMS
TRC Meter	Hach	Pocket Colorimeter II	18060E360317	05/31/2019	New	Field Services - Columbia	TRC Meter #19-0531	EQLIMS
TRC Meter	Hach	Pocket Colorimeter II	12070E203659	10/09/2012	Unknown	Field Services - Columbia	TRC Meter #TRCM-1210	EQLIMS
Turbidimeter	Hach	2100Q	13080C027705	11/14/2013	Unknown	Field Services - Columbia	Turbidimeter #TM-1114	EQLIMS
Balance - Top Loader	Mettler Toledo	ME303E	B908280591	03/18/2019	New	Metals	Balance #19-0318	EQLIMS
Centrifuge	VWR International	Clinical 200	68165129	03/13/2017	New	Metals	Centrifuge #4	EQLIMS
Chiller	ThermoFisher Scientific	121123010000008	1171044001200304	6/8/2020	New	Metals	Chiller # 11	NA
Chiller	ThermoFisher Scientific	121123010000008	0110795501131104	Unknown	Unknown	Metals	Chiller #1	NA
Chiller	ThermoFisher Scientific	121123010000008	1122870601191031	Unknown	Unknown	Metals	Chiller #10	NA

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Chiller	ThermoFisher Scientific	121123010000008	1171199501200813	10/22/2020	New	Metals	Chiller #12	NA
Chiller	ThermoFisher Scientific	121123010000008	1171283901201019	11/1/2020	New	Metals	Chiller #13	NA
Chiller	ThermoFisher Scientific	121123010000008	0110399701130116	Unknown	Unknown	Metals	Chiller #2	NA
Mercury Analyzer CVAA	Leeman Labs	Hydra AA	4004	02/2014	Unknown	Metals	Hg #4	Software
Quick Trace Mercury Analyzer (CVAF)	Teledyne Cetac Technologies	M-8000	US17076012	02/05/2017	New	Metals	Hg #5	Software
Mercury Analyzer CVAA	Leeman Labs	Hydra IIAA	US18193023	09/10/2018	Unknown	Metals	Hg #6	Software
Quick Trace Autosampler	Teledyne Cetac Technologies	ASX-560	03174A560	02/05/2017	Unknown	Metals - Attached to Hg # 5	Hg Autosampler #5	NA
Hot Block Graphite Heater	Environmental Express	SC154	2018CECW4978	09/26/2018	New	Metals	Hot Block #10	NA
Hot Block Graphite Heater	Environmental Express	SC154	2019CECw5105	06/18/2019	New	Metals	Hot Block #11	NA
Hot Block Graphite Heater	Environmental Express	SC154	3098CEC15414	01/04/2011	Unknown	Metals - Storage	Hot Block #4	NA
Hot Block Graphite Heater	CPI	MOD/BLOCK	NA	02/2016	Unknown	Metals - Storage	Hot Block #6	NA
Hot Block Graphite Heater	CPI	MOD/BLOCK	NA	06/2016	Unknown	Metals	Hot Block #7	NA
Hot Block Graphite Heater	Environmental Express	SC154	2018CEC4882	06/28/2018	New	Metals	Hot Block #9	NA

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Inductively Coupled Plasma-Trace	ThermoFisher Scientific	iCAP6500	20074003	01/8/2002	New	Metals	ICP #4	Software
Inductively Coupled Plasma-Trace	ThermoFisher Scientific	iCAP7600	IC76DU134102	01/2014	New	Metals	ICP #5	Software
ICP Autosampler	Teledyne Cetac Technologies	ASX-560	111742A560	01/19/2018	Unknown	Metals - Attached to ICP-MS # 4	ICP Autosampler #5	NA
ICP Autosampler	Teledyne Cetac Technologies	ASX-560	1118107A560	12/21/2018	Unknown	Metals - Storage	ICP Autosampler #6	Software
ICP Autosampler	Teledyne Cetac Technologies	ASX-560	011969A560	03/15/2019	Unknown	Metals - Attached to ICP # 5	ICP Autosampler #7	NA
ICP Autosampler	Teledyne Cetac Technologies	ASX-560	0319125A560	04/15/2019	Unknown	Metals - Attached to ICP-MS # 2	ICP Autosampler #8	NA
ICP Autosampler	Teledyne Cetac Technologies	ASX-560	121845A560	04/22/2019	Unknown	Metals - Attached to ICP # 4	ICP Autosampler #9	NA
ICP-MS	ThermoFisher Scientific	XSERIES 2	01686C	06/2011	New	Metals	ICP-MS #2	Software
ICP-MS	ThermoFisher Scientific	iCAP RQ	iCAPRQ00715	01/11/2018	New	Metals	ICP-MS #4	Software
Autoclave	Market Forge	Sterilmatic	NA	05/30/2013	Unknown	Microbiology	Autoclave #3	NA
Black Light Lamp	Spectroline	B-260	1959859	09/30/2015	Unknown	Microbiology	Black Light Lamp #1	NA
Blender	Black & Decker	PB1002	NA	08/31/2015	Unknown	Microbiology	Blender #M2	NA
Homgenizer	VWR International	250	V-28-045	2017	New	Microbiology	Homgenizer #1	NA

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Incubator	Thelco	51220112	699010336	04/1999	Unknown	Microbiology	Incubator #M3	NA
Incubator	Thelco	51221118	604081265	8/2004	Unknown	Microbiology	Incubator #M4	NA
Microscope	LW Scientific	Dual Mag	HG814817	08/02/2017	New	Microbiology	Microscope #2	NA
Quanti-Tray Sealer	IDEXX	89-0003936-00	QTP131716024 19	06/23/2017	New	Microbiology	Quanti-Tray Sealer #2	NA
Quanti-Tray Sealer	IDEXX	89-0003936-00	QTP132014009 63	6/18/2020	New	Microbiology	Quanti-Tray Sealer #4	NA
Refrigerator	ThermoFisher Scientific	GT20FREEFSA	300420890	10/8/2020	New	Microbiology	Refrigerator # 33	NA
Water Bath	ThermoFisher Scientific	TSCIR35	300182961	12/29/2017	New	Microbiology	Water Bath #M10	NA
Water Bath	ThermoFisher Scientific	TSCIR35	300261529	7/26/2019	New	Microbiology	Water Bath #M11	NA
Water Bath	Shel Lab	SWB2	8018015	08/15/2010	New	Microbiology	Water Bath #M8	NA
Balance - Top Loader	Mettler Toledo	ML802E/03	B326486754	10/16/2013	Unknown	Organic Prep.	Balance #13-1016	EQLIMS
Balance - Top Loader	Mettler Toledo	PB1502-S	1120493684	08/08/2012	Unknown	Organic Prep.	Balance #25B	EQLIMS
Chiller	ThermoFisher Scientific	121123010000 008	112261770119 0523	06/05/2019	New	Organic Prep.	Chiller #7	NA
Chiller	ThermoFisher Scientific	121123010000 008	112277790119 0903	09/10/2019	New	Organic Prep.	Chiller #9	NA

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Drying Oven	Baxter	DK43	189001	1998	Unknown	Organic Prep.	Drying Oven #5	NA
Drying Oven	Quincy Lab	10GCE	G1E-00440	06/06/2018	New	Organic Prep.	Drying Oven #7	NA
Freezer	Frigidaire	FFU1464FWO	WB64933777	02/04/2012	Unknown	Organic Prep.	Freezer #16	NA
Freezer	Fisher Scientific	20FFEEFSA	015849640116 0516	05/24/2016	New	Organic Prep.	Freezer #17	NA
Muffle Furnace	ThermoScientific Lindberg/Blue M	BF51828C-1	J11A-164949-JA	09/17/2015	New	Organic Prep.	Furance #5	EQLIMS
Muffle Furnace	ThermoScientific Lindberg/Blue M	BF51828C-1	P04N-623993-PN	06/2003	Unknown	Organic Prep.	Furnace #1	EQLIMS
Muffle Furnace	ThermoScientific Lindberg/Blue M	BF51828C-1	112630560119 0221	03/05/2019	New	Organic Prep.	Furnace #6	EQLIMS
Muffle Furnace	ThermoScientific Lindberg/Blue M	BF51828C-1	112630590119 0222	03/05/2019	New	Organic Prep.	Furnace #7	EQLIMS
GPC	J2 Scientific	AccuPrep	03H-1083-3.0	12/2003	Unknown	Organic Prep.	GPC #1	Software
GPC	J2 Scientific	AccuPrep MPS	05C-1148-4.0	01/05/2011	Unknown	Organic Prep.	GPC #2	Software
Microwave Reaction System	CEM	MARS6 230/60	MJ6109	05/2015	New	Organic Prep.	MCW #2	Software
Refrigerator	Fisher Scientific	20FREEFSA	012542840115 0205	12/02/2015	New	Organic Prep.	Refrigerator #24	NA
Refrigerator	Black & Decker	BCRK43	184300118	12/02/2019	Unknown	Organic Prep.	Refrigerator #32	NA

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Shaker	Glas-Col	099A DPM12	11905743	05/24/2019	New	Organic Prep.	Shaker #2	EQLIMS
Speed Vap	Horizon Tech	Speed-vap IV	15-0039	09/17/2015	New	Organic Prep.	Speed Vap #2	Software
Hazardous Waste Filtration System	Millipore Corporation	YT30142HW	NA	Unknown	New	Organic Prep.	TCLP Filter System # 1	NA
TCLP Rotary Sample Agiator	Environmental Express	EN60034-1	GF189N060-BMYJ1C	2/17/2020	New	Organic Prep.	TCLP Rotary Sample Agitator # 4	NA
TCLP Rotary Sample Agitator	Associated Design	3740-12-BRE 12 Station	1528	1998	Unknown	Organic Prep.	TCLP Rotary Sample Agitator #1	NA
TCLP Rotary Sample Agitator	Associated Design	3740-12-BRE 12-11	1957	03/27/2019	Unknown	Organic Prep.	TCLP Rotary Sample Agitator #3	NA
Turbo Vap II Concentrator	Biotage	Turbo Vap II	181200445	06/06/2018	New	Organic Prep.	Turbo Vap II Concentrator #10	Software
Turbo Vap II Concentrator	Biotage	Turbo Vap II	181400458	06/06/2018	New	Organic Prep.	Turbo Vap II Concentrator #11	Software
Turbo Vap II Concentrator	Biotage	Turbo Vap II	193101237	11/12/2019	New	Organic Prep.	Turbo Vap II Concentrator #13	Software
Turbo Vap II Concentrator	Biotage	Turbo Vap II	194501417	01/23/2020	Unknown	Organic Prep.	Turbo Vap II Concentrator #14	Software
Turbo Vap II Concentrator	Biotage	Turbo Vap II	TV1434N20575	10/23/2014	Unknown	Organic Prep.	Turbo Vap II Concentrator #6	Software
Turbo Vap II Concentrator	Biotage	Turbo Vap II	172500072	09/12/2017	New	Organic Prep.	Turbo Vap II Concentrator #8	Software
Turbo Vap II Concentrator	Biotage	Turbo Vap II	173000101	09/13/2017	New	Organic Prep.	Turbo Vap II Concentrator #9	Software

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Automated Turner	DFIT	Custom Build	Custom Build	05/01/2017	New	Organic Prep.	Turner #1	NA
Ultrasonic Disruptor	Misonix (Qsonica)	Sonicator 3000	R02998	2006	Unknown	Organic Prep.	Ultrasonic Disruptor #1	NA
Ultrasonic Disruptor	Misonix (Qsonica)	Sonicator 3000	R2374	Unknown	Unknown	Organic Prep.	Ultrasonic Disruptor #2	NA
Ultrasonic Processor	Misonix (Qsonica)	Sonicator Q700	78274E	01/16/2014	Unknown	Organic Prep.	Ultrasonic Processor #2	NA
Ultrasonic Processor	Qsonica	Sonicator Q700	86849J	09/22/2015	New	Organic Prep.	Ultrasonic Processor #3	NA
Vortex Mixer	Fisher Scientific	SA8	R800010121	Unknown	Unknown	Organic Prep.	Vortex Mixer #3	NA
Water Bath	Boekel	1494	190921153	05/24/2019	New	Organic Prep.	Water Bath #4	EQLIMS
Zero Headspace Extractor	Associated Design	3745 ZHE	NA	Unknown	New	Organic Prep.	ZHE # 2	NA
Zero Headspace Extractor	Associated Design	3745 ZHE	RW-273	Unknown	New	Organic Prep.	ZHE # 273	NA
Zero Headspace Extractor	Associated Design	3745 ZHE	RW-274	Unknown	New	Organic Prep.	ZHE # 274	NA
Zero Headspace Extractor	Associated Design	3745 ZHE	RW-275	Unknown	New	Organic Prep.	ZHE # 275	NA
Zero Headspace Extractor	Associated Design	3745 ZHE	RW-276	Unknown	New	Organic Prep.	ZHE # 276	NA
Zero Headspace Extractor	Associated Design	3745 ZHE	RW-277	Unknown	New	Organic Prep.	ZHE # 277	NA

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Zero Headspace Extractor	Associated Design	3745 ZHE	NA	Unknown	New	Organic Prep.	ZHE # 3	NA
Zero Headspace Extractor	Associated Design	3745 ZHE	NA	Unknown	New	Organic Prep.	ZHE # 4	NA
Zero Headspace Extractor	Associated Design	3745 ZHE	NA	Unknown	New	Organic Prep.	ZHE # 5	NA
Zero Headspace Extractor	Associated Design	3745 ZHE	NA	Unknown	New	Organic Prep.	ZHE # 7	NA
Zero Headspace Extractor	Associated Design	3745 ZHE	NA	Unknown	New	Organic Prep.	ZHE #1	NA
Centrifuge	Hettich Zentrifugen	Rotanta 460	0000280-03-01	5/22/2020	Used	PFAS (LC/MS/MS)	Centrifuge # 5	NA
HPLC / Triple Quad Detector	Agilent Technologies	1260 HiP Degasser (G4225A) 4465800	JPAAB07343	11/01/2016	New	PFAS (LC/MS/MS)	LCMSMS1	Software
HPLC / Triple Quad Detector	Agilent Technologies	1260 HiP Degasser (G4225A) 4465800	JPAAB08258	10/24/2018	New	PFAS (LC/MS/MS)	LCMSMS2	Software
Shaker	VWR International	3500 Std. 120V	17127011	03/14/2017	New	PFAS (LC/MS/MS)	Shaker #1	NA
Sonicator	VWR International	97043-976	1717A0842	04/2017	New	PFAS (LC/MS/MS)	Sonicator #1	NA
Turbo Vap LV	Biotage	Turbo Vap LV	174100205	07/10/2018	DEMO	PFAS (LC/MS/MS)	TurboVap LV #1	Software
Vortex Mixer	Fisher Scientific	SA8	R800010110	Unknown	Unknown	PFAS (LC/MS/MS)	Vortex Mixer # 2	NA

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
HPLC / Triple Quad Detector	Agilent Technologies	1260 Binary Pump (G1312B)	DEACB10794	11/01/2016	New	PFAS (LC/MS/MS) - Attached to LCMSMS1	NA	Software
HPLC / Triple Quad Detector	Agilent Technologies	1260 ALS (G1329B)	DEAAC42719	11/01/2016	New	PFAS (LC/MS/MS) - Attached to LCMSMS1	NA	Software
HPLC / Triple Quad Detector	Agilent Technologies	1260 TCC (G1316A)	DEACN45680	11/01/2016	New	PFAS (LC/MS/MS) - Attached to LCMSMS1	NA	Software
HPLC / Triple Quad Detector	Sciex	5020102	BJ29321608	11/01/2016	New	PFAS (LC/MS/MS) - Attached to LCMSMS1	NA	Software
HPLC / Triple Quad Detector	Agilent Technologies	1260 Bin Pump (G1312B)	DEACB11117	10/24/2018	New	PFAS (LC/MS/MS) - Attached to LCMSMS2	NA	Software
HPLC / Triple Quad Detector	Agilent Technologies	1290 Thermostat (G1330B)	DEBAK32602	10/24/2018	New	PFAS (LC/MS/MS) - Attached to LCMSMS2	NA	Software
HPLC / Triple Quad Detector	Agilent Technologies	1260 TCC (G1316A)	DEACN47046	10/24/2018	New	PFAS (LC/MS/MS) - Attached to LCMSMS2	NA	Software
HPLC / Triple Quad Detector	Sciex	5060884 (QTRAP 4500)	EB250231807	10/24/2018	New	PFAS (LC/MS/MS) - Attached to LCMSMS2	NA	Software
Pancake Probe	Ludlum Measurements Inc	44-9	PR385660	08/13/2019	Unknown	Sample Receiving	Pancake Probe #1	NA
Walk-in Cooler	Polar King	DT820	A16097900	11/1/2019	Used	Sample Receiving	Polar Walk-In Cooler #30	NA

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Survey Meter	Ludlum Measurements Inc	12	162119	10/2014	Unknown	Sample Receiving	Survey Meter #SM-2	NA
Walk-in Cooler (SR)	Climate Control	Unknown	Unknown	Unknown	Unknown	Sample Receiving	Walk-in Cooler #1	NA
Autosampler	Agilent Technologies	7683B	CN54028057	11/2005	Unknown	Semi-Volatiles	Autosampler #27	Software
Autosampler	Hewlett Packard	Series 7683	US95110869	08/2009	Unknown	Semi-Volatiles	Autosampler #29	NA
Autosampler	Agilent Technologies	7683	US92107420	02/10/2006	Unknown	Semi-Volatiles	Autosampler #31	Software
GC Dual ECD	Agilent Technologies	6890N	CN10518005	06/2005	Unknown	Semi-Volatiles	GC #10	EQLIMS
GC Dual ECD	Agilent Technologies	6890N	CN10633092	02/10/2006	Unknown	Semi-Volatiles	GC #11	EQLIMS
GC Dual FID	Agilent Technologies	6890N	CN10650056	07/2007	Unknown	Semi-Volatiles	GC #12	EQLIMS
GC Dual ECD	Agilent Technologies	7890B	US13463049	12/2013	Unknown	Semi-Volatiles	GC #14	Software
GC Dual ECD	Hewlett Packard	6890	US00022182	08/1998	Unknown	Semi-Volatiles	GC #5	EQLIMS
GC Dual ECD	Agilent Technologies	6890N	US10212124	2001	Unknown	Semi-Volatiles	GC #7	EQLIMS
HPLC	Agilent Technologies	1200 Series Degasser	JP62357625	07/2007	Unknown	Semi-Volatiles	HPLC #1	Software
HPLC	Agilent Technologies	1260 Series Quat Pump	DEAB702492	12/15/2002	Unknown	Semi-Volatiles	HPLC #2	Software

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Hydrogen Generator	Parker	60HMD	13HMD0167	2015	New	Semi-Volatiles	Hydrogen Generator #1	NA
GC/MS	Agilent Technologies	GC-6890N / MS-5975	US10542069 / US53931266	11/2005	Unknown	Semi-Volatiles	MSD #10	EQLIMS
GC/MS	Agilent Technologies	GC-6890N / MS-5975B	CN10633016 / US53931266	02/10/2006	Unknown/ New	Semi-Volatiles	MSD #11	EQLIMS
GC/MS	Agilent Technologies	GC- 6890 / MS-5973	CN10305019 / US33220170	08/2009	Unknown	Semi-Volatiles	MSD #12	EQLIMS
GC/MS	Agilent Technologies	GC- G3440B / MS- G7077B	US18073048 / US1807M004	04/01/2018	New	Semi-Volatiles	MSD #16	Software
GC/MS	Hewlett Packard	GC- 6890 / MS-5973	US00009998 / US72010678	1997	Unknown	Semi-Volatiles	MSD #4	EQLIMS
Refrigerator	Fisher Scientific	3751FS	Unknown	01/03/2013	Unknown	Semi-Volatiles	Refrigerator #20	NA
Refrigerator	Fisher Scientific	20FREEFSA	015849620116 0516	05/26/2016	New	Semi-Volatiles	Refrigerator #26	NA
Refrigerator	Fisher Scientific	20FREEFSA	116354150119 1121	11/22/2019	New	Semi-Volatiles	Refrigerator #31	NA
Zero Air Generator	Parker	UHP35ZASW	13Z016	11/2016	New	Semi-Volatiles	Zero Air Generator #2	NA
Autosampler	Agilent Technologies	7683	US12419286	08/1998	Unknown	Semi-Volatiles - Attached to GC # 10	Autosampler #34	NA
Autosampler	Agilent Technologies	G2913A	CN52125269	08/2019	Unknown	Semi-Volatiles - Attached to GC # 11	Autosampler #39	NA
Autosampler	Agilent Technologies	G2613A	US90204405	7/10/2020	Unknown	Semi-Volatiles - Attached to GC # 12	Autosampler #42	Software

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Autosampler	Agilent Technologies	7693	CN 13450127	12/2013	Unknown	Semi-Volatiles - Attached to GC # 14	Autosampler #30	NA
Autosampler	Agilent Technologies	7683	UF91606711	08/07/2009	Unknown	Semi-Volatiles - Attached to GC # 5	Autosampler MSD #12	NA
Autosampler	Agilent Technologies	7683	US81501052	2001	Unknown	Semi-Volatiles - Attached to GC # 7	Autosampler #33	NA
HPLC	Agilent Technologies	1260 Series DAD	DEAA301229	12/23/2019	Unknown	Semi-Volatiles - Attached to HPLC # 1	NA	Software
HPLC	Agilent Technologies	1200 Series Quat Series	DE43631807	1/29/2019	Unknown	Semi-Volatiles - Attached to HPLC # 1	NA	Software
HPLC	Agilent Technologies	1200 Series ALS	DE64761071	07/2007	Unknown	Semi-Volatiles - Attached to HPLC # 1	NA	Software
HPLC	Agilent Technologies	1200 Series TCC	DE63061942	07/2007	Unknown	Semi-Volatiles - Attached to HPLC # 1	NA	Software
HPLC	Agilent Technologies	1260 Series DAD	DEAA301472	10/5/2018	Unknown	Semi-Volatiles - Attached to HPLC # 2	NA	Software
HPLC	Agilent Technologies	1260 Series ALS	DEAB305754	12/15/2002	Unknown	Semi-Volatiles - Attached to HPLC # 2	NA	Software
HPLC	Agilent Technologies	1290 Series Thermostat	DEBAK13589	12/15/2002	Unknown	Semi-Volatiles - Attached to HPLC # 2	NA	Software
HPLC	Agilent Technologies	1260 Series TCC	DEACN06491	12/15/2002	Unknown	Semi-Volatiles - Attached to HPLC # 2	NA	Software
Autosampler	Agilent Technologies	7683B	CN62940495	02/10/2006	Unknown	Semi-Volatiles - Attached to MSD # 10	Autosampler #26	NA
Autosampler	Agilent Technologies	7683B	CN53827750	01/14/2019	Unknown	Semi-Volatiles - Attached to MSD # 11	Autosampler #37	NA

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Autosampler	Agilent Technologies	7683B / 7683	CN71343516 / US12419304	07/2007	Unknown	Semi-Volatiles - Attached to MSD # 12	Autosampler #GC 12	NA
Autosampler	Agilent Technologies	G4513A	CN18090084	04/01/2018	Unknown	Semi-Volatiles - Attached to MSD # 16	Autosampler #38	NA
Autosampler	Hewlett Packard	HP6890	US73503279	1997	Unknown	Semi-Volatiles - Attached to MSD # 4	Autosampler #28	NA
Geiger Counter	Eberline Instrument Corporation	E-120E	13618	05/2012	Unknown	Storage - RSO	Geiger Counter #1	NA
Geiger Counter Probe	ThermoFisher Scientific	HP-190A	1092	Unknown	Unknown	Storage - RSO	Geiger Counter Probe #1	NA
Survey Meter	Ludlum Measurements Inc	12	305064	10/21/2014	Unknown	Storage - RSO	Survey Meter #SM-1	NA
Tritium Detector, Large Area	Ludlum Measurements Inc	44-110	PR327579	10/21/2014	Unknown	Storage - RSO	Tritium Detector #TD-1	NA
Tritium Detector, Large Area	Ludlum Measurements Inc	44-110	PR149565	05/2012	Unknown	Storage - RSO	Tritium Detector #TD-2	NA
Water Bath	Boekel	1494	52202304	06/22/2005	Unknown	Storage in SU3	Water Bath #3	NA
Microwave Reaction System	CEM	MARS6	MN2648	09/26/2018	New	Storage in SU4	MCW #3	Software
Balance - Top Loader	Sartorius	Practum 412-1S	32850362	05/19/2015	New	Volatiles	Balance #VOA-2	EQLIMS
Centrifuge	VWR International	Clinical 200	68145107	11/2014	Unknown	Volatiles	Centrifuge #3	EQLIMS
Freezer	Frigidaire	FFU1464FWO	WB64933778	01/2007	New	Volatiles	Freezer #15	NA

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Freezer	Fisher Scientific	20LFEEFSA	116279080117 0807	08/09/2017	New	Volatiles	Freezer #29	NA
GC PID/FID	Agilent Technologies	GC 7890A	CN10815015	02/2009	Unknown	Volatiles	GC #13	Software
GC Dual FID	Hewlett Packard	GC 6890	US00026561	06/1999	Unknown	Volatiles	GC #6	EQLIMS
GC Dual FID	Agilent Technologies	GC 6890	US00042711	10/2002	Unknown	Volatiles	GC #8	EQLIMS
GC PID/FID	Hewlett Packard	GC 6890N	US10403005	04/2004	New	Volatiles	GC #9	EQLIMS
Hydrogen Generator	Parker	H2PEMPD-510-100	19PLD5086	6/28/2019	New	Volatiles	Hydrogen Generator #2	NA
GC/MS	Agilent Technologies	GC 5973 / MS 6890	CN10508043 / US44621283	10/13/2011	Unknown	Volatiles	MSD #13	Software
GC/MS	Agilent Technologies	GC 7890A / MS 5975C	CN12071133 / US11092707	03/2012	Unknown	Volatiles	MSD #14	Software
GC/MS	Agilent Technologies	GC 7890B / MS 5975C	CN14323093 / US13433B01	10/2014	Unknown	Volatiles	MSD #15	Software
GC/MS	Agilent Technologies	GC 5977B (G7077B) / MS 7890B (G3442B)	US1844M023 / CN18033077	01/29/2019	New	Volatiles	MSD #17	Software
GC/MS	Agilent Technologies	GC 8860 / MS 5977B	CN1925C013 / US1925R048	09/30/2019	New	Volatiles	MSD #18	Software
GC/MS	Agilent Technologies	GC 6890 / MS 5973	US00021398 / US80210977	05/1998	Unknown	Volatiles	MSD #5	EQLIMS
GC/MS	Agilent Technologies	GC 6890 / MS 5973N	US00032609 / US90160010	03/2000	Unknown	Volatiles	MSD #6	EQLIMS

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
GC/MS	Agilent Technologies	GC-6890 / MS-5973N	US00034585 / US01150087	05/2000	Unknown	Volatiles	MSD #7	EQLIMS
GC/MS	Agilent Technologies	GC 6890N / MS 5973N	US10150052 / US10451993	01/2002	Unknown	Volatiles	MSD #8	EQLIMS
GC/MS	Agilent Technologies	GC 6890 / MS 5973I	CN10339035 / US33220060	03/2004	Unknown	Volatiles	MSD #9	EQLIMS
Refrigerator	Kenmore	253.60722	WA73802498	12/2007	Unknown	Volatiles	Refrigerator #18	NA
Refrigerator / Freezer	Whirlpool	ET18JK	E81221051	1991	Unknown	Volatiles	Refrigerator / Freezer #2	NA
Refrigerator / Freezer	General Electric	TBX18G	RT599810	03/1999	Unknown	Volatiles	Refrigerator / Freezer #4	NA
Refrigerator / Freezer	Frigidaire	FRT18B4AW6	BA31031244	08/29/2003	Unknown	Volatiles	Refrigerator / Freezer #9	NA
Ultrasonic Cleaner	VWR International	97043-964	1612A6033	Unknown	Unknown	Volatiles	Ultrasonic #1	NA
Vortex Mixer	Fisher Scientific	G-560	2-32318	Unknown	Unknown	Volatiles	Vortex Mixer #1	NA
Walk-in Cooler	HotPack	1208401	73160	1992	Unknown	Volatiles	Walk-in Cooler #8	NA
Zero Air Generator	Parker	UHP35ZASW	14Z0024	2015	New	Volatiles	Zero Air Generator #1	NA
Autosampler	O.I Analytical	4551-A	D81445B984	05/29/2008	Unknown	Volatiles - Attached to GC # 13	Autosampler #9	NA
Purge & Trap Concentrator	O.I Analytical	4660 Eclipse	B42546655P	10/2003	Unknown	Volatiles - Attached to GC # 13	Purge & Trap #2	NA

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Autosampler	Agilent Technologies	7683B	CN54028055	10/2006	Unknown	Volatiles - Attached to GC # 6	Autosampler #7	NA
Autosampler	EST Analytical	LGX50	LGX11062315	08/2015	Unknown	Volatiles - Attached to GC # 8	Autosampler #16	NA
Autosampler	EST Analytical	LGX50	LGX120070119	08/01/2019	Unknown	Volatiles - Attached to GC # 9	Autosampler #24	NA
Autosampler	O.I Analytical	4100	B508410225	Unknown	Unknown	Volatiles - Attached to MSD # 13	Autosampler #21	NA
Purge & Trap Concentrator	O.I Analytical	4660 Eclipse	C504466513P	06/2005	Unknown	Volatiles - Attached to MSD # 13	Purge & Trap #5	NA
Autosampler	O.I Analytical	4551-A	F00545B633	02/04/2020	Unknown	Volatiles - Attached to MSD # 14	Autosampler #41	NA
Purge & Trap Concentrator	O.I Analytical	4660	D534466729	Unknown	Unknown	Volatiles - Attached to MSD # 14	Purge & Trap #18	NA
Autosampler	O.I Analytical	4100	A428410167	10/2014	Unknown	Volatiles - Attached to MSD # 15	Autosampler #15-1	NA
Purge & Trap Concentrator	O.I Analytical	4660 (eclipse)	K430466776P	10/2014	Unknown	Volatiles - Attached to MSD # 15	Purge & Trap #15-2	NA
Autosampler	O.I Analytical	4100	E916410597	05/14/2019	Unknown	Volatiles - Attached to MSD # 17	Autosampler #23	NA
Purge & Trap Concentrator	O.I Analytical	4660 Eclipse	H301466056P	04/2013	Unknown	Volatiles - Attached to MSD # 17	Purge & Trap #10	NA
Purge & Trap Concentrator	O.I Analytical	4760 Eclipse	A925447209	02/13/2020	Unknown	Volatiles - Attached to MSD # 18	Purge & Trap #21	NA
Autosampler	O.I Analytical	4100	B509410485	Unknown	Unknown	Volatiles - Attached to MSD # 5	Autosampler #20	NA

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Purge & Trap Concentrator	O.I Analytical	4660 Eclipse	A352466312P	02/2005	Unknown	Volatiles - Attached to MSD # 5	Purge & Trap #3	NA
Autosampler	O.I Analytical	4551-A	D51945B229	06/2005	Unknown	Volatiles - Attached to MSD # 6	Autosampler #3	NA
Purge & Trap Concentrator	O.I Analytical	4660 Eclipse	G129466450P	10/2011	Unknown	Volatiles - Attached to MSD # 6	Purge & Trap #8	NA
Autosampler	O.I Analytical	4551-A	F30845B200	04/2013	Unknown	Volatiles - Attached to MSD # 7	Autosampler #14	NA
Purge & Trap Concentrator	O.I Analytical	4660 (eclipse)	K433466705P	10/2014	Unknown	Volatiles - Attached to MSD # 7	Purge & Trap #15-1	NA
Autosampler	O.I Analytical	4551-A	F30845B199	04/2013	Unknown	Volatiles - Attached to MSD # 8	Autosampler #12	NA
Purge & Trap Concentrator	O.I Analytical	4660	C519466231	09/08/2016	Unknown	Volatiles - Attached to MSD # 8	Purge & Trap #17	NA
Autosampler	O.I Analytical	4551-A	F30845B198	04/2013	Unknown	Volatiles - Attached to MSD # 9	Autosampler #13	NA
Purge & Trap Concentrator	O.I Analytical	4660 Eclipse	D637466747P	06/2005	Unknown	Volatiles - Attached to MSD # 9	Purge & Trap #4	NA
Autosampler	Teledyne Cetac Technologies	Atomx XYZ	US18354015	01/29/2019	New	Volatiles - Storage	Autosampler #22	Software
Auto-Titrator System	Man-Tech	PCM-1002/4	NA	12/13/2017	New	Wet Chem.	#AT	EQLIMS
Auto Analyzer System	Lachat	Quick-Chem 8000	A83000-1990	2002	Unknown	Wet Chem.	AA #2	EQLIMS
Automated Chemistry Analyzer 3700	O.I Analytical	329995	B550837557	01/2016	New	Wet Chem.	AA #3	Software

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Automated Chemistry Analyzer 3700	O.I Analytical	329995	B549837495	01/2016	New	Wet Chem.	AA #4	Software
Phosphorus and Orthophosate Chemistry Unit	Skalar Analytical	5000	182889	02/11/2019	New	Wet Chem.	AA #5	EQLIMS
Aquakem 200 Analyzer	ThermoFisher Scientific	984206 (type: 973)	22831	05/2013	Unknown	Wet Chem.	Aquakem Analyzer #2	EQLIMS
Balance - Analytical	Mettler Toledo	MS204S	B427764304	06/08/2014	New	Wet Chem.	Balance #11A	EQLIMS
Balance - Analytical	Mettler Toledo	MS-204TS	B539502279	01/10/2015	New	Wet Chem.	Balance #12	EQLIMS
BOD Incubator	Fisher Scientific	304	UF30K-5020/03B-868521	12/2003	Unknown	Wet Chem.	BOD Incubator #B4	NA
BOD Incubator	VWR International	2020	90100	04/2007	Unknown	Wet Chem.	BOD Incubator #B5	NA
BOD Incubator	Fisher Scientific	11-679-25C	2.01808E+12	10/29/2012	Unknown	Wet Chem.	BOD Incubator #B6	NA
BOD Incubator	Fisher Scientific	3720	136748-668	07/16/2013	Unknown	Wet Chem.	BOD Incubator #B8	NA
BOD Incubator	Fisher Scientific	3720A	300044697	09/15/2011	Unknown	Wet Chem.	BOD Incubator #B9	NA
Bomb Calorimeter	Parr	1341EB	7303	1997	Unknown	Wet Chem.	Bomb Calorimeter #1	NA
Centrifuge	VWR International	Clinical 200	68175072	02/07/2018	Unknown	Wet Chem.	Centrifuge #5	EQLIMS
Chiller	Caron	2050-1	2050-1-673	07/05/2019	New	Wet Chem.	Chiller #8	NA

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
COD Digestion Block	Columbia Analytical Instruments	Smart Block 226	NA	10/02/2014	Unknown	Wet Chem.	COD Digestion Block #3	NA
Conductivity Meter	Extech Instruments	EC100	301884	02/06/2016	New	Wet Chem.	Conductivity Meter #AT2	Software
Conductivity Meter	Man-Tech	4510	66959	12/13/2017	New	Wet Chem.	Conductivity Meter #AT3	Software
Cyanide Hotblock	Andrews	110-10-12	A5P0842	12/14/2015	Unknown	Wet Chem.	Cyanide Hotblock #3	NA
Cyanide Hotblock	Andrews	110-10-P	86N0892	01/25/2017	New	Wet Chem.	Cyanide Hotblock #4	NA
Luminescence Dissolved Oxygen Meter	YSI	ProODO	16B100776	02/18/2016	New	Wet Chem.	DO Meter #5	EQLIMS
ProODO Probe	YSI	ProODO Probe	16B100987	02/18/2016	New	Wet Chem.	DO Meter #5 Probe	NA
Luminescence Dissolved Oxygen Meter	YSI	ProODO	18E102940	05/03/2018	New	Wet Chem.	DO Meter #7	EQLIMS
ProODO Probe	YSI	ProODO Probe	18H108982	01/18/2019	New	Wet Chem.	DO Probe #19-0118	NA
ProODO Probe	YSI	ProODO Probe	19A105401	01/03/2019	New	Wet Chem.	DO Probe #19-0130	NA
Drying Oven	Lindberg/Blue M	G01350C	P18G-33084-PG	1998	Unknown	Wet Chem.	Drying Oven #3	NA
Drying Oven	ThermoFisher Scientific	Heratherm GS180 510288	41746692	01/09/2015	Unknown	Wet Chem.	Drying Oven #6	EQLIMS
Drying Oven	Fisher Scientific	151030520	42516465	2/14/2020	New	Wet Chem.	Drying Oven #8	NA

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Flashpoint Tester	Koehler	K16225	00020A	6/19/2019	New	Wet Chem.	Flashpoint Tester #3	NA
Hot Block Graphite Heater	Environmental Express	SC154	NA	11/06/2019	New	Wet Chem.	Hot Block #12	NA
Ion Chromatograph	Dionex	ICS-2100	9050165	06/08/2012	New	Wet Chem.	IC #4	Software
Ion Chromatograph	ThermoFisher Scientific	Dionex ICS-2100	13010346	10/15/2015	New	Wet Chem.	IC #5	Software
Ion Chromatograph	ThermoFisher Scientific	Dionex Integrion RFIC	19010010	02/19/2019	New	Wet Chem.	IC #6	Software
Ion Chromatograph	ThermoFisher Scientific	Dionex Integrion RFIC	16033206	08/13/2019	New	Wet Chem.	IC #7	Software
IC Pump	ThermoFisher Scientific	Dionex AXP	Z0053278	12/2014	New	Wet Chem.	IC Pump #1	Software
Micro Dist Hot Block	Lachat	Micro Dist	11010000294	11/08/2011	Unknown	Wet Chem.	Micro Dist Hot Block #1	EQLIMS
Micro Dist Hot Block	Lachat	Micro Dist A17102	1.412E+11	11/2014	New	Wet Chem.	Micro Dist Hot Block #2	EQLIMS
pH Meter	VWR International	SB70P	D05976	11/22/2010	New	Wet Chem.	pH Meter #12	EQLIMS
pH Meter	ThermoFisher Scientific	STARA1110	J14050	09/08/2016	New	Wet Chem.	pH Meter #14	EQLIMS
Refrigerator	Fisher Scientific	20FREEFSA	1.15067E+15	04/18/2019	New	Wet Chem.	Refrigerator #19-0418	NA
Spectrophotometer	Hach	DR 3900	1606201	06/30/2015	New	Wet Chem.	Spectrophotometer #4	EQLIMS

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
TDS Stable Weigh Filling Station	Environmental Express	TDS600F	37-7264	11/01/2017	New	Wet Chem.	TDS Filling Station #1	NA
TDS Static Guard	Haug	1781305	15426	11/01/2017	New	Wet Chem.	TDS Static Guard #1	NA
TKN Hot Block	Environmental Express	TKN054	2020TKN203	08/05/2020	New	Wet Chem.	TKN Hot Block #4	EQLIMS
TOC Analyzer	O.I Analytical	1030	J019730196	06/2010	Unknown	Wet Chem.	TOC #1	EQLIMS
Turbidimeter	HF Scientific	Micro 100	201610276	05/12/2016	Unknown	Wet Chem.	Turbidimeter #3	EQLIMS
Walk-in Cooler (INM)	NA	NA	NA	06/29/2018	Unknown	Wet Chem.	Walk-in Cooler #3	NA
IC Variable Wavelength Detector	ThermoFisher Scientific	Dionex VWD	14101375	12/2014	New	Wet Chem. - Attached to IC # 5	IC VWD #1	NA
IC Autosampler	ThermoFisher Scientific	Dionex AS-DV	181214361	02/19/2019	New	Wet Chem. - Attached to IC # 6	IC Autosampler #6	NA
IC Autosampler	ThermoFisher Scientific	Dionex AS-DV	190614911	08/13/2019	New	Wet Chem. - Attached to IC # 7	IC Autosampler #7	NA
Ignition Unit	Parr	2901EB	NA	04/10/2012	Unknown	Wet Chem. - Attached to the Bomb Colorimeter # 1	Ignition Unit #1U1012	Software
Autosampler	Man-Tech	PC-1000-688	261C4N066	2002	Unknown	Wet Chem. - Attached to #AT	NA	EQLIMS
Buret Module	Man-Tech	PC-1000-1040	MT-1G7-200	12/13/2017	New	Wet Chem. - Attached to #AT	NA	EQLIMS
GT-ID-II	Man-Tech	PCM-1000-495	CIL-00296	2002	Unknown	Wet Chem. - Attached to #AT	NA	EQLIMS

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Mini Mix Stirrer Controller	Man-Tech	PC-1000-388	MT-1C7-375	12/13/2017	New	Wet Chem. - Attached to #AT	NA	EQLIMS
PC-Tis Interface Module	Man-Tech	PC-1000-102	MS-0E2-308	2002	Unknown	Wet Chem. - Attached to #AT	NA	EQLIMS
PC-Titrate Titra-Rinse/A Module	Man-Tech	PCM-1000-408	MS-0G0-118	2002	Unknown	Wet Chem. - Attached to #AT	NA	EQLIMS
PC-Titrate Titra-Rinse/A Module	Man-Tech	PCM-1000-408	MS-0F1-137	2002	Unknown	Wet Chem. - Attached to #AT	NA	EQLIMS
Peristaltic Pump	Man-Tech	PC-1000-475	MT-1H7-420	12/13/2017	New	Wet Chem. - Attached to #AT	NA	EQLIMS
Titrasip Module	Man-Tech	PC-1300-475	MT-1E7-1014	12/13/2017	New	Wet Chem. - Attached to #AT	NA	EQLIMS
Turbidity Assay Plus	Man-Tech	PC-20013A	209090	2002	Unknown	Wet Chem. - Attached to #AT	NA	EQLIMS
Autosampler	O.I Analytical	ASX-520	0915907A520	01/2016	New	Wet Chem. - Attached to AA # 3	AA #3 Autosampler	NA
Autosampler	O.I Analytical	ASX-130	111504A130	01/2016	New	Wet Chem. - Attached to AA # 4	AA #4 Autosampler	NA
Autosampler	Skalar Analytical	21050900-01	181797	02/11/2019	New	Wet Chem. - Attached to AA #5	NA	EQLIMS
Backpressure Regulator	Skalar Analytical	5530	181210	02/11/2019	New	Wet Chem. - Attached to AA #5	NA	EQLIMS
Cyanide and Phenol Chemistry Unit	Skalar Analytical	5000	182885	02/11/2019	New	Wet Chem. - Attached to AA #5	NA	EQLIMS
Cyanide Kelada	Skalar Analytical	5565	19123	02/11/2019	New	Wet Chem. - Attached to AA #5	NA	EQLIMS

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Mixing Device	Skalar Analytical	21050325	18417	02/11/2019	New	Wet Chem. - Attached to AA #5	NA	EQLIMS
Power Supply for UV Lamp	Skalar Analytical	PS-4	NA	02/11/2019	New	Wet Chem. - Attached to AA #5	NA	EQLIMS
Stir Base	Environmental Express	SC160	711326	7/28/2020	New	Wet Chem. - Attached to Hot Block # 12	Stir Base # 1	NA
IC Autosampler	ThermoFisher Scientific	Dionex AS-DV	180513735	06/14/2018	Unknown	Wet Chem. - Attached to IC # 4	IC Autosampler #5	NA
IC Autosampler	ThermoFisher Scientific	Dionex AS-DV	150810037	10/15/2015	New	Wet Chem. - Attached to IC # 5	IC Autosampler #4	NA
TKN Hot Block Controller	Environmental Express	TKN100	2019TKNBC164	08/05/2020	New	Wet Chem. - Attached to TKN # 4	TKN Block Controller #4	NA
TOC Autosampler	O.I Analytical	1088 AS	E018788107	06/2010	Unknown	Wet Chem. - Attached to TOC # 1	TOC Autosampler #1	NA
Luminescence Dissolved Oxygen Meter	YSI	ProODO	15K102490	11/16/2015	New	Wet Chem. - Storage	DO Meter #4	EQLIMS
Luminescence Dissolved Oxygen Meter	YSI	4010-3W	20411391	11/16/2020	New	Wet Chem. - Attached to BOD Robot # 1	DO Meter #8	EQLIMS
BOD Robot	Skalar Analytical	21088903-01	206074	11/16/2020	New	Wet Chem.	BOD Robot # 1	EQLIMS
Dilution Pump	Skalar Analytical	21007985-01	20617	11/16/2020	New	Wet Chem. - Attached to BOD Robot # 1	Pump 3 - Dilution Liquid #1	EQLIMS
Balance - Top Loader	Sartorius	Practum 412-1S	39550090	11/23/2020	New	PFAS (LC/MS/MS)	Balance # 20-1123	NA
Balance - Top Loader	Mettler Toledo	ML802E/03	B345970999	01/2014	New	Storage - QA	Balance # 13-1224	NA

7.5.2 PAS-WCOL [Charlotte Service Center]

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Composite Sampler	Isco	3710	212K00187	Unknown	Unknown	Field Services - Charlotte	Composite Sampler #18-1115G	EQLIMS
Composite Sampler	Isco	3700	09078-115	Unknown	Unknown	Field Services - Charlotte	Composite Sampler #18-1115I	EQLIMS
Composite Sampler	Isco	3710	200B02589	Unknown	Unknown	Field Services - Charlotte	Composite Sampler #18-1115K	EQLIMS
Composite Sampler	Isco	2910	197M01047	Unknown	Unknown	Field Services - Charlotte	Composite Sampler #18-1115M	EQLIMS
Composite Sampler	Isco	2910	07983-001	Unknown	Unknown	Field Services - Charlotte	Composite Sampler #18-1115N	EQLIMS
Composite Sampler	Isco	2910	197B01220	Unknown	Unknown	Field Services - Charlotte	Composite Sampler #18-1115P	EQLIMS
Composite Sampler	Isco	3710	217J00432	Unknown	New	Field Services - Charlotte	Composite Sampler #18-1115Q	EQLIMS
Composite Sampler	Isco	3710	212I11172	Unknown	Used	Field Services - Charlotte	Composite Sampler #19-0122	EQLIMS
Conductivity Meter	YSI	32/11SPD	04IJ071BC	05/2007	Unknown	Field Services - Charlotte	Conductivity Meter #1-C	Software
DO Meter	YSI	Pro 20	11A100588	2006	Unknown	Field Services - Charlotte	DO Meter #1	EQLIMS
DO Meter	YSI	Pro 20	13A101442	01/2013	Unknown	Field Services - Charlotte	DO Meter #2	EQLIMS
Low Level TRC Meter	Hach	DR2700	1353696	03/29/2011	Unknown	Field Services - Charlotte	LL TRC Meter #1	Software

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Low Level TRC Meter	Hach	DR2800	1177995	Unknown	Used	Field Services - Charlotte	LL TRC Meter #2	Software
pH Meter	ThermoFisher Scientific	Orion Star A221	G07625	03/23/2016	New	Field Services - Charlotte	pH Meter #100	EQLIMS
pH Meter	ThermoFisher Scientific	Orion Star A221	K00642	Unknown	Unknown	Field Services - Charlotte	pH Meter #102	EQLIMS
pH Meter	ThermoFisher Scientific	Orion Star A221	H01668	Unknown	Unknown	Field Services - Charlotte	pH Meter #13-820	EQLIMS
pH Meter	ThermoFisher Scientific	Orion Star A321	G12116	11/21/2019	Unknown	Field Services - Charlotte	pH Meter #19-1121	EQLIMS
TRC Meter	Hach	Pocket Colorimeter II	40700019648	2002	Unknown	Field Services - Charlotte	TRC Meter #8	EQLIMS
TRC Meter	Hach	Pocket Colorimeter II	12070E203555	10/09/2012	Unknown	Field Services - Charlotte	TRC Meter #TRCM-1210A	EQLIMS
TRC Meter	Hach	Pocket Colorimeter II	12070E203545	10/09/2012	Unknown	Field Services - Charlotte	TRC Meter #TRCM-1210B	EQLIMS

7.5.3 PAS-WCOL [Greenville Service Center]

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Composite Sampler	Isco	3710	216A00118	02/2016	New	Field Services - Greenville	Composite Sampler #16-0201	EQLIMS
Composite Sampler	Isco	3710	218B00157	Unknown	Unknown	Field Services - Greenville	Composite Sampler #18-1115R	EQLIMS

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Composite Sampler	Isco	3710	206D00712	Unknown	Unknown	Field Services - Greenville	Composite Sampler #18-1115S	EQLIMS
Composite Sampler	Isco	3710	214H00829	Unknown	Unknown	Field Services - Greenville	Composite Sampler #18-1115T	EQLIMS
Composite Sampler	Isco	3710	218B00159	Unknown	Unknown	Field Services - Greenville	Composite Sampler #18-1115U	EQLIMS
Composite Sampler	Isco	3710	218M00078	01/09/2019	New	Field Services - Greenville	Composite Sampler #19-0109A	EQLIMS
DO Meter	YSI	Pro 20	148104784	Unknown	Unknown	Field Services - Greenville	DO Meter #3	EQLIMS
pH Meter	ThermoFisher Scientific	Orion Star A221	K07028	12/02/2015	New	Field Services - Greenville	pH Meter #0215	EQLIMS
pH Meter	ThermoFisher Scientific	Orion Star A321	G09500	10/12/2017	New	Field Services - Greenville	pH Meter #103	EQLIMS
pH Meter	ThermoFisher Scientific	STARA3210	G11062	12/21/2018	New	Field Services - Greenville	pH Meter #18-1221A	EQLIMS
TRC Meter	Hach	Pocket Colorimeter II	15050E272953	01/07/2015	Unknown	Field Services - Greenville	TRC Meter #TRCM-0615	EQLIMS

8.0 ADDENDUM: PROGRAM REQUIREMENTS

Program specific information provided in this addendum supplements the main body of this manual. Each subsection is stand-alone, meaning the requirements for the quality management system in each subsection only apply to the program referenced. Additionally, only program requirements for the quality management system that are more stringent than the content of the main body of the manual are included.

8.1 DoD/DOE

PAS-WCOL maintains accreditation for DoD/DoE Environmental Laboratory Approval Program (ELAP)

This addendum outlines additional policies and processes established by this laboratory to maintain compliance with DoD/DOE program specific requirements as outlined in the DoD/DOE Consolidated Quality Systems Manual (QSM) for Environmental Laboratories. The QSM incorporates ISO/IEC 17025 and the TNI Standard and includes additional program-specific requirements for laboratories that perform analytical testing services for DoD and DoE and which must be followed for DoD / DoE projects.

Section 4.2.5: Supporting Documents

Technical SOPs used for DoD/DoE testing must also include instructions for equipment and instrument maintenance, computer software/hardware, and troubleshooting.

The review frequency for technical SOPs used for DoD/DoE testing is annual, instead of every 2 years.

Section 4.4: Review of Analytical Service Requests

If the DoD/DoE customer requests a statement of conformity, the standard used for the decision rule must be communicated to and agreed on with the customer and identified in the final test report.

Laboratory requests to deviate from the requirements specified in the DoD/DoE QSM must be requested on a project-basis and include technical justifications for the deviation. These requests are submitted to and approved by the DoD/DoE project chemist or contractor, however name, in addition to the PAS client.

For DoD / DoE projects, will also seek clarification from the customer when the customer has requested an incorrect, obsolete or improper method for the intended use of data; the laboratory needs to depart from its test method SOP in order to meet project-specific data quality objectives; information in project planning documents is missing or is unclear,

Section 4.5: Subcontracting

In addition to written client approval of any subcontractor for testing, the customer is notified of the laboratory's intent to use a subcontractor for any management system element (such as data review, data processing, project management or IT support) and consent for subcontracting is obtained approved in writing by the DoD/DoE customer and record of consent kept in the project record.

Section 4.6: Purchasing and Supplies

The laboratory procedure for records of receipt of materials and supplies used in testing also include a specification to record the date opened (DoE only).

Section 4.9.3: Nonconforming Work

The laboratory's procedure for client notification includes the 15-business day DoD /DOE timeframe for notification of the problem and the 30-business day timeframe for submission of the corrective action plan or corrective actions taken. This procedure also includes the DoD/DoE requirement for AB notification of discovery.

Section 4.13: Control of Records

Technical Records: The laboratory's procedure for logbooks includes measures to prevent the removal of or addition of pages to the logbook (applies to both hardcopy and electronic). Hardcopy logbooks are version controlled, pre-numbered and bound. Initials and entries and are signed or initialed and dated by the person making the entry and the entry is made at the time the activity is performed and in chronological order. Each page of the logbook must be closed by the last person making the entry on the page. Closure is recorded by the initial and date of the person making the last entry.

Section 5.4.5.3.3: Limit of Detection

For DoD/DOE the LOD is an estimate of the minimum amount of an analyte that can be reliably detected by an analytical process. For clarification, the LOD is the analyte concentration necessary to distinguish its presence from its absence. The LOD may be used as the lowest concentration for reliably reporting a non-detect (ND). The LOD is specific to each suite of analyte, matrix, and method including sample preparation.

After each DL determination, the laboratory establishes the LOD by spiking a quality system matrix at a concentration of least 2X but no greater than 4X the DL (i.e. 2X DL ≤ LOD Spike ≤ 4X DL). The spike concentration establishes the LOD and the concentration at which the LOD is verified.

The LOD is established during method validation and after major changes to the analytical system or procedure that affects sensitivity of analysis or how the procedure is performed.

An LOD study is not required for any component for which spiking solutions or quality control samples are not available. Additionally, an LOD study is not required if the laboratory does not report data below the LOQ.

The LOD must be verified on a quarterly basis. Each preparation method listed on the scope of accreditation must have quarterly LOD verifications; however, verification of all possible combinations of preparation and clean-up techniques is not required. Where LOD verifications are not performed on all combinations, the LOD verification is based on the worst-case combination (preparation method with all applicable cleanup steps).

The laboratory's procedure for LOD determination and verification is detailed in laboratory SOP *Method Validation* [QA Policy ME003BF].

Section 5.4.5.3.4: Limit of Quantitation

For DoD/DOE, the LOQ is established for each analyte-matrix-method combination, including surrogates. When an LOD is determined or verified by the laboratory, the LOQ must be above the LOD [DL<LOD<LOQ].

At a minimum, the LOQ must be verified quarterly; however, verification of all possible combinations of preparation and clean-up techniques is not required. Where LOQ verifications are not performed

on all combinations, the LOQ verification on the worst-case combination (preparation method with all applicable cleanup steps).

The laboratory's procedure for LOQ determination and verification is detailed in laboratory SOP INSERT LOCAL SOP REFERENCE.

Section 5.4.7: Control of Data

The laboratory will assure LIMS passwords are changed at least once per year.

An audit of the LIMS will be incorporated into the laboratory's annual internal audit schedule.

The laboratory will have procedures in place to notify DoD/DoE customers of changes to LIMS software or hardware configurations that may impact the customer's integrity of electronic data

Section 5.9.1: Quality Control

For DoD/DOE, storage blanks are essential QC to monitor the storage of samples for volatile organic analysis (VOA). The laboratory's SOP for storage of VOA samples must include a contamination monitoring program based on the performance of storage blanks. (See QSM 5.3.3)

Section 5.8.5: Sample Disposal

For DoE projects, the record of disposal must also include how the sample was disposed and the name of the person that performed the task.

Appendix E: Support Equipment Calibration

Mechanical Volumetric Pipette: In addition to the quarterly verification check, pipettes used for DoD/DoE projects are checked daily before use using the same procedure and criteria specified for the quarterly check.

Water Purification System: The performance of the water purification system is checked daily prior to use in accordance with laboratory SOP *Deionized Water System and Storage Blank Testing* [QA SOP ME0012S].

Radiological Survey Equipment: The performance of the radiological survey equipment is checked daily prior to use in accordance with laboratory SOP *Sample Receiving* [AD SOP ME0013H].

Additional: (DoE): Section 6.0 of the QSM outlines additional management system requirements for the management of hazardous and radioactive materials management and health and safety practices. The laboratory, if approved for DoE, will work with the PAS Health and Safety Director to establish plans, policies and procedures that conform to these comprehensive specifications and incorporate these documents into the quality management system.

8.2 US EPA Contract Laboratory Program (US EPA CLP)

PAS-WCOL holds active CLP contracts for the analysis of multi-media, multi-concentration organic, metals, and inorganic non-metals compounds in water and solid samples by SOM, ISM, and SFAM methodologies. The purpose of this analytical service is to provide analytical data for use by the US EPA in support of the investigation and clean-up activities under the Comprehensive Environmental Response, Compensation, and Liability Action of 980 (CERCLA) and the Superfund Amendments and Reauthorization act of 1986 (SARA). Other EPA Program Offices, as well as customers outside of the EPA, may use this service where similar analytical data needs are required.

This addendum outlines additional policies and processes established by this laboratory to maintain compliance with US EPA CLP specific requirements as outlined in the *EPA Contract Laboratory Program Statement of Work for Organic Superfund Methods* (SOW SOM) and *Inorganic Superfund Methods* (SOW ISM), as well as *Superfund Analytical Methods* (SFAM SOW). The EPA SOW provides a contractual framework for laboratories to perform analytical services and applies EPA CLP analytical methods for the isolation, detection, and quantitative measurement of target analytes in aqueous/water, soil/sediment, waste, and wipe samples. The analytical service contract provides the methods to be used and the specific contractual requirements by which the EPA will evaluate data. Specific technical requirements are outlined in individual method/analytical SOPs.

SOW Exhibit E Section 3.2 (A): Organization and Personnel

Please contact the laboratory for resumes of key personnel.

SOW Exhibit E Section 3.3.2: Revision Submissions (QM)

The Quality Manual must be amended and submitted to the recipient(s) identified in SOW Exhibit B – Reporting and Deliverables Requirements, Table 1 – Deliverable Schedule, within 14-days of the time when any one of the following circumstances occurs:

- The EPA modifies technical requirements of the Statement of Work or the contract;
- The EPA notifies the laboratory of deficiencies in the Quality Manual;
- The EPA notified the laboratory of deficiencies resulting from the EPA's review of the laboratory's performance;
- The laboratory identified changes in the organization, personnel, facility, equipment, policy, or procedures; or
- The laboratory identified deficiencies resulting from the internal review of their organization, personnel, facility, equipment, policy, procedure, or Quality Manual.

All changes must be clearly marked and the amended section pages must have the date on which the changes were implemented.

SOW Exhibit E Section 4.4.2: Revision Submissions (SOP)

SOPs must be amended and submitted to the recipient(s) identified in SOW Exhibit B – Reporting and Deliverables Requirements, Table 1 – Deliverable Schedule when any one of the following circumstances occurs:

- The EPA modifies the technical requirements of the SOW or the contract;
- The EPA notifies the laboratory of deficiencies in their SOP documentation;
- The EPA notifies the laboratory of deficiencies resulting from the EPA's review of the laboratory's performance;
- The laboratory's procedures change;
- The laboratory identifies deficiencies resulting from the internal review of SOP documentation; or
- The laboratory identifies deficiencies resulting from the internal review of procedures.

The laboratory shall amend and submitted revised or write and submit new SOPs within 14 days of when the circumstances listed above result in a discrepancy between what was previously described

in the SOP and what is presently occurring at the laboratory. All changes must be clearly marked and the amended/new SOP shall have the date on which the changes were implemented. Documentation of the reason(s) for the changes must be submitted along with the SOP.