

Quality Assurance Project Plan
for
U.S. Environmental Protection Agency
Region 5
And
Wisconsin Department of Natural Resources

PFAS Sampling
Oconomowoc Electroplating Company, Inc. Superfund Site
W2573 Oak Street, Ashippun, Dodge County, WI 53003

BRRTS#: 02-14-000905
WI FID: 114004220
US EPA ID: WID006100275

Prepared by:

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ENV-MAN-MIN4-0001, Pace Analytical, *Quality Manual*

ENV-SOP-MIN4-0008, Pace Analytical, *Sample Management*

ENV-SOP-MIN4-0184 v01_Data and Records Archival-30647-1

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Acronyms and Abbreviations

MNA	Monitored Natural Attenuation
O&M	Operation and Maintenance
PFOA	Perfluorooctanoic Acid
PFOS	Perfluorooctanesulfonic acid
PFAS	Per- and polyfluoroalkyl substances
PM	Project Manager
PRP	Potential Responsible Party
QA	Quality Assurance
QA/QC	Quality Assurance/Quality Control
QAPP	Quality Assurance Project Plan
QC	Quality Control
ROD	Record of Decision
RPD	Relative Percent Difference
SOP	Standard Operating Procedures
SOW	Statement of Work
TBD	To Be Determined
UFP-QAPP	Uniform Federal Policy for Quality Assurance Project Plans
US EPA	U.S. Environmental Protection Agency
WDNR	Wisconsin Department of Natural Resources

1. Project Description and Objectives

In 2022, the U. S. Environmental Protection Agency (US EPA) Region 5 awarded a grant to Wisconsin Department of Natural Resources (WDNR), in collaboration with Hyde Environmental, Inc. (Hyde), to perform sampling for Perfluoroalkylated substances. This effort will support Region 5's and WDNR's effort to characterize emerging contaminants at Wisconsin Superfund Sites. The Quality Assurance Project Plan (QAPP) was prepared as a project-wide overarching umbrella ("generic") QAPP as defined by, and in accordance with the *Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP), Manual VI*, March 2005 (US EPA, 2005a), and the *Uniform Federal Policy for Quality Assurance Project Plans, Part 2B, Quality Assurance/Quality Control Compendium: Minimum QA/QC Activities VI, March 2005* (US EPA, 2005b) and falls under WDNR's Quality Management Plan (WDNR 2019).

This QAPP has been prepared for sampling at the Oconomowoc Electroplating Company, Inc. (OECI) Superfund Site (herein referred to as the "Site") located in Ashippun, Dodge County, Wisconsin. The sampling will independently evaluate the presence/absence of Perfluorooctanoic Acid (PFOA) and Perfluorooctane Sulfonate (PFOS) compounds, collectively referred to as Per- and polyfluoroalkyl substances (PFAS), at the Site. Sampling includes all necessary aspects of sample collection and processing, including providing the necessary materials and labor at the site, packaging, labeling, procuring laboratory services from an US EPA- and WDNR-approved laboratory, and responsibility for shipping or transporting samples to the lab in accordance with handling procedures.

The purpose of the sampling is to:

- Independently evaluate the presence/absence of PFAS at the Site.

This QAPP, for the work to be performed by Hyde is structured in accordance with the Statement of Work (SOW) for the contract and with guidelines provided in US EPA QA/G-5, *Guidance for Quality Assurance Project Plans* (US EPA, 2002a) and US EPA QA/R-5, *EPA Requirements for Quality Assurance Project Plans* (US EPA, 2001). The latter documents identify a graded approach to developing the QAPP.

1.1 Site History

The Site is located at W2573 Oak Street, Town of Ashippun, Dodge County, WI.

The Primary Contaminants of concern in the Record of Decision (ROD) currently include Volatile organic compounds (VOCs), specifically trichloroethene (TCE), cis-1,2-dichloroethene (cis-1,2-DCE), and vinyl chloride (VC).

The Site is located approximately 11 miles east of Watertown, seven (7) miles north of Oconomowoc, WI, and 28 miles west-northwest of the state's largest city, Milwaukee. The Site is in the Northeast Quarter (NE ¼) of the Southeast Quarter (SE ¼) of Section 30, Township 9 North, Range 17 East, Dodge County, WI. The Site includes approximately four (4) acres of the former electroplating facility and an additional 6.5 acres of wetland, including part of Davy Creek, a tributary of the nearby Rock River. The former OECI facility is bounded by Oak St. to the northeast; Eva St. to the northwest; Elm St. to the southwest; and a Town garage and park to the southeast. The former electroplating area is vacant, devoid of any structures, and generally grass and tree covered. The Site is in a mixed-use

neighborhood with commercial operations and railroad tracks to the northeast of the Site; single-family residents to the northeast; Village-owned buildings and the Ashippun Community Park to the southeast; and wetland, farmland, and a wastewater treatment plant to the southwest.

OECI operated an electroplating facility at the Site from 1957 until February 1991. Electroplating and finishing operations at the facility used nickel, chromium, zinc, copper brass, cadmium, and tin. Wastewater discharged from the Site contained cyanide, chromium, and acid and alkaline solutions. Degreasing at the Site resulted in the discharge of 1,1-dichloroethane (1,1-DCA), chloroform, 1,2-dichloroethane (1,2-DCA), and TCE. Between 1957 and 1972, untreated wastewater was discharged directly to the wetland south of the Site. Two (2) unlined settling lagoons were constructed on-Site in 1972, prior to the construction of an on-Site wastewater treatment plant, which was completed in 1973. The lagoons measured approximately 60 feet long by 40 wide and were approximately 5 feet deep. Concrete lined two (2) of the walls, and sloped gravel lined the others. The untreated plating sludge was known to have overflowed the lagoon banks, accumulating wastes in the adjacent wetlands between Davy Creek and the OECI facility.

In 1980, OECI contracted to remove approximately one million pounds (500 tons) of plating sludge from the lagoons. This amount only represented approximately two-thirds of the volume present in the lagoons at the time. OECI refused to remove the remainder of the wastes. In the late 1980s, US EPA investigations of the Site revealed approximately 75,000 sq. ft. of wetland near the OECI facility was contaminated with metals and cyanide.

By 1990, the US EPA completed a Remedial Investigation (RI) and Feasibility Study (FS). A ROD was first signed in September 1990 (later amended [AROD] in September 1991 with the addition of OU Five) and contained five (5) separate discrete actions or operable units (OUs). In general, these include the following:

- OU 1 – Surface water, metal hydroxide sludge, and contaminated soil associated with the two lagoons.
- OU 2 – All other contaminated soil around the OECI facility not associated with the lagoons.
- OU 3 – Contaminated groundwater associated with the Site.
- OU 4 – The most highly contaminated sediments in Davy Creek and the wetlands.
- OU 5 – Dismantle the abandoned facility and dispose of associated debris.

In the early 1990s, all OECI assets were removed, including a main process building, wastewater treatment building, waste lagoons, and other miscellaneous equipment, along with 650 cubic yards of lagoon sediments, 700 yards of contaminated soil, and approximately 6,000 cubic yards of contaminated sediments from the adjacent wetlands around Davy Creek. A groundwater pump and treat system, including a building combined with five (5) groundwater recovery wells, was installed at the Site. The system operated between 1997 and 2004. At the request of the US EPA, groundwater treatment was discontinued because the system was deemed no longer effective. The recovery wells were abandoned in 2009 and the treatment building removed in early 2017.

In 2011, the ROD was amended to change the selected remedy for OU3 from groundwater extraction and treatment to either source area removal or in situ treatment, followed by MNA. Soil treatment of

the area beneath the former OECl process building was completed with a zero valent iron product in 2013. The Site has been reviewed for treatment effectiveness by the US EPA over five (5) 5-Year Reviews. The remedial actions for OUs 1, 2, 4, and 5 have been completed. Groundwater has been consistently monitored since 2004, with the last monitoring event completed in November 2021.

1.2 Development of this QAPP

This QAPP has been developed in accordance with the Uniform Federal Policy QAPP Guidance (US EPA, 2005a), which is composed of a series of 37 worksheets, along with a series of attachments. The worksheets (Appendix A of this QAPP) provide information on project management, project objectives, measurement and data acquisition, project assessment and oversight, and data review. In accordance with US EPA guidance, cross-referencing is utilized where applicable, in order to streamline the document, including references to other documents that are companion documents to this QAPP.

Quality Assurance/Quality Control (QA/QC) procedures performed under this QAPP will be performed in accordance with applicable professional technical standards, the previously mentioned US EPA, WDNR requirements and guidelines (or their successors), and the specific project goals and requirements.

2. References

- US EPA, 2021. Policy and Program Requirements for the Mandatory Agency-Wide Quality System. CIO 2105.1. Available at: <https://www.epa.gov/quality/agency-wide-quality-program-documents> .
- US EPA, 2001. *EPA Requirements for Quality Assurance Project Plans (EPA QA/R-5)*. EPA/240/B-01/003. March (Reissued May 2006). Available at: https://www.epa.gov/sites/production/files/2016-06/documents/r5-final_0.pdf.
- US EPA, 2002a. *Guidance for Quality Assurance Project Plans (EPA QA/G-5)*, EPA/240/R-02/009. December. Available at: <https://www.epa.gov/sites/production/files/2015-06/documents/g5-final.pdf>.
- US EPA, 2002b. *Guidance on Choosing a Sampling Design for Environmental Data Collection for Use in Developing a Quality Assurance Project Plan (EPA QA/G-5S)*, EPA/240/R-02-005. December. Available at: <https://www.epa.gov/sites/production/files/2015-06/documents/g5s-final.pdf>.
- US EPA, 2005a. Uniform Federal Policy for Quality Assurance Project Plans. Evaluating, Assessing, and Documenting Environmental Data Collection and Use Programs. *Part 1: UFP-QAPP Manual* (Version 1). Pub. Nos., EPA: EPA-505-B-04-900A, DoD: DTIC ADA 427785. March 2005. Available at: https://www.epa.gov/sites/production/files/documents/ufp_qapp_v1_0305.pdf.
- US EPA, 2005b. *Uniform Federal Policy for Quality Assurance Project Plans*. Part 2B, Quality Assurance/Quality Control Compendium: Minimum QA/QC Activities (Version 1). EPA: EPA-505-B-04-900B, DoD: DTIC ADA 426957. March. Available at: https://www.epa.gov/sites/default/files/documents/qaqc_v1_0305.pdf
- US EPA, 2006. Guidance on Systematic Planning Using the Data Quality Objectives Process: EPA QA/G-4. February 2006. Available at: <https://www.epa.gov/quality/guidance-systematic-planning-using-data-quality-objectives-process-epa-qag-4>
- US EPA, 2012. Handbook for Developing Quality Assurance Project Plans. December 2012.
- US EPA, 2016c. Drinking Water Health Advisory for Perfluorooctane Sulfonate Acid (PFOS). EPA 822-R-16-004. May 2016. 88 pp.
- US EPA, 2016d. Fact Sheet, PFOA & PFOS Drinking Water Health Advisories. EPA 800-F-16-003. 5 pp.
- US EPA, 2018. Data Review and Validation Guidelines for Perfluoroalkyl Substances (PFASs) Analyzed Using EPA Method 537. EPA 910-R-18-001, November 2018, 47 pp. Available at: <https://nepis.epa.gov/Exe/ZyPURL.cgi?Dockey=P100VW12.txt>
- US EPA 2021. Groundwater Sampling SOP. https://US.EPA.sharepoint.com/sites/R5_Work/fieldops/controleddocs/CrossProgram/SFD/R5%20SEM/D%20Groundwater%20Sampling%20Standard%20Operating%20Procedure.pdf
- WDNR, 2019. Quality Management Plan.
- WDNR, 2019. *Wisconsin PFAS Aqueous (Non-Potable Water) and Non-Aqueous Matrices Method Expectations, Version 12.16.2019, Per- and Polyfluorinated Alkyl Substances (PFAS) Analysis Using Isotope Dilution by LC/MS/MS*. Available at: https://dnr.wisconsin.gov/sites/default/files/topic/LabCert/EA-19-0001-C_WI_PFAS_Method_Expectations_12.16.19.pdf

WDNR PFAS Updates 3.1.21. Available at:

<https://dnr.wisconsin.gov/sites/default/files/topic/PFAS/LabUpdate20210301.pdf>

Appendix A _____

QAPP Worksheets UFP-QAPP, (US EPA, 2005a)

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

- 1. Project Identifying Information
 - a. Site name/project name: Oconomowoc Electroplating Company, Inc. (OECI)
 - b. Site location/number: W2573 Oak Street, Town of Ashippun, Dodge County, WI, EPA ID: WID006100275, BRRTS#: 02-14-000905, WI FID: 114004220
 - c. Contract/Work assignment number: 370004-O22-P033-RR1668-01

2. Contract/Field Sampling Organization:

Jim Lindemann, Hyde Environmental, Inc.
Project Manager


Signature 3/6/23
date

Robert Thomson, Hyde Environmental, Inc.
QA Manager


Signature 3/6/23
date


3. Federal Regulatory Agency:

U.S. Environmental Protection Agency, Region 5

Kelly Rodibaugh
Science and Quality Assurance Branch (SQAB)
Laboratory Services and Applied Science Division (LSASD)


Signature April 4, 2023
date

William Murray
Superfund & Emergency Management Division
U.S. Environmental Protection Agency, Region 5


Signature 4 April 2023
date

Lead Organization/State Regulatory Agency:

Gwen Salieres
WDNR Project Manager

Gwen Salieres
Signature

3/17/2023
date

Erin Endsley
WDNR QA Manager

Erin Endsley
Signature

3/17/2023
date

- 4. Other Stakeholders (as needed): Not applicable
- 5. List plans and reports from previous investigations relevant to this project: Not applicable

Site Name/Project Name:	Oconomowoc Electroplating Company, Inc. (OECI) Superfund Site
Contractor's Name:	Hyde Environmental, Inc., Pace Analytical Services
Contract Title:	RR Oconomowoc Electroplating PFAS Sampling
Identify guidance used to prepare QAPP:	Uniform Federal Policy for Quality Assurance Project Plans, Manual VI (US EPA, 2005a), Quality Policy CIO 2105 (US EPA, 2000a) as explained in the American National Standard ASQ/ANSI E4:2014: Quality management systems for environmental information and technology programs- Requirements with guidance for use. EPA Requirements for Quality Assurance Projects, EPA QA/R-5 (US EPA, 2001a), Guidance for the Quality Assurance Project Management Plans, EPA QA/G-5 (US EPA, 2002a), Guidance on Choosing a Sampling Design for Environmental Data Collection, EPA QA/G-5S (US EPA 2002b), EPA Region 5 Analytical Services Branch, PFAS Sampling Fact Sheet V4
Identify approval entity:	US EPA Region 5
Indicate whether the QAPP is a generic or a project-specific QAPP:	This QAPP is project-specific and has been prepared to cover PFAS sampling at the Oconomowoc Electroplating Company, Inc. (OECI) Site.
List dates of scoping sessions that were held:	June 22, 2022 (see Worksheet #9)
List dates and titles of QAPP documents written for previous site work, if applicable:	QAPP, Hyde, November 4, 2021 – Groundwater monitoring associated with OU 3
List data users:	WDNR, US EPA Region 5

QAPP Worksheet #2: Identifying Information
(UFP-QAPP Manual Section 2.2.4)

Required QAPP Element(s) and Corresponding QAPP Section(s) (US EPA, 2005a)	Required Information	Crosswalk to Related Information and Documents
Project Management and Objectives		
2.1 Title and Approval Page	Title and Approval Page	Worksheet #1
2.2 Document Format and Table of Contents	Table of Contents	
2.2.1 Document Control Format	QAPP Identifying Information	Worksheet #2
2.2.2 Document Control Numbering System		
2.2.3 Table of Contents		
2.2.4 QAPP Identifying Information		
2.3 Distribution List and Project Personnel Sign-Off Sheet		
2.3.1 Distribution List	Distribution List	Worksheet #3
2.3.2 Project Personnel Sign-Off Sheet	Project Personnel Sign-Off Sheet	Worksheet #4
2.4 Project Organization		
2.4.1 Project Organization Chart	Project Organizational Chart	Worksheet #5
2.4.2 Communication Pathways	Communication Pathways	Worksheet #6
2.4.3 Personnel Responsibilities and Qualifications	Personnel Responsibilities and Qualifications Table	Worksheet #7
2.4.4 Special Training Requirements and Certification		Worksheet #8
2.5 Project Planning/Problem Definition		
2.5.1 Project Planning (Scoping)	Project Planning Session Documentation, Project Scoping Session Participants Sheet	Worksheet #9
2.5.2 Problem Definition, Site History, and Background	Problem Definition, Site History, and Background Site Maps (historical and current)	Worksheet #10 Appendix D
2.6 Project Quality Objectives and Measurement Performance Criteria		

QAPP Worksheet #2: Identifying Information
(UFP-QAPP Manual Section 2.2.4)

Required QAPP Element(s) and Corresponding QAPP Section(s) (US EPA, 2005a)	Required Information	Crosswalk to Related Information and Documents
2.6.1 Development of Project Quality Objectives Using the Systematic Planning Process	Site-Specific Project Quality Objectives	Worksheet #11
2.6.2 Measurement Performance Criteria	Measurement Performance Criteria Table	Worksheet #12
2.7 Secondary Data Evaluation		
2.8 Project Overview and Schedule		
2.8.1 Project Overview	Summary of Project Tasks Reference Limits and Evaluation Table	Worksheet #14 Worksheet #15
2.8.2 Project Schedule	Project Schedule/Timeline	Worksheet #16 Appendix D
Measurement/Data Acquisition		
3.1 Sampling Tasks		
3.1.1 Sampling Process Design and Rationale	Sampling Design and Rationale Sampling Locations and Methods Table	Worksheet #17 Worksheet #18
3.1.2 Sampling Procedures and Requirements	Analytical SOP Requirements Table	Worksheet #19
3.1.2.1 Sampling Collection Procedures	Field Quality Control Sample Summary Project Sampling SOP References Table	Worksheet #20 Worksheet #21
3.1.2.2 Sample Containers, Volume, and Preservation	Analytical Methods/SOP Requirements Table	Worksheet #19 Worksheet #23
3.1.2.3 Equipment/Sample Containers Cleaning and Decontamination Procedures	Project Sampling SOP References Table	Worksheet #21

QAPP Worksheet #2: Identifying Information
(UFP-QAPP Manual Section 2.2.4)

Required QAPP Element(s) and Corresponding QAPP Section(s) (US EPA, 2005a)	Required Information	Crosswalk to Related Information and Documents
3.1.2.4 Field Equipment Calibration, Maintenance, Testing, and Inspection Procedures	Field Equipment Calibration, Maintenance, Testing, and Inspection Table	Worksheet #22
3.1.2.5 Supply Inspection and Acceptance Procedures	Project Sampling SOP References Table	Worksheet #21
3.1.2.6 Field Documentation Procedures	Field Documentation Procedures	Worksheet #22
3.2 Analytical Tasks		
3.2.1 Analytical SOPs	Analytical SOP References Table	Worksheet #23
3.2.2 Analytical Instrument Calibration Procedures	Analytical Instrument Calibration Table	Worksheet #24
3.2.3 Analytical Instrument and Equipment Maintenance, Testing and Inspection Procedures	Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	Worksheet #25
3.2.4 Analytical Supply Inspection and Acceptance Procedures		
3.3 Sample Collection Documentation, Handling, Tracking, and Custody Procedures	Groundwater sampling SOP, Site-Specific Field Sampling Plan	Appendix D
3.3.1 Sample Collection Documentation	Sample Container Identification	Worksheet #26 Worksheet #27
3.3.2 Sample Handling and Tracking System	Sample Handling Flow Diagram	Worksheet #26 Worksheet #27
3.3.3 Sample Custody	Example Chain of Custody Form and Seal	Worksheet #26 Worksheet #27
3.4 Quality Control Samples		
3.4.1 Sample Quality Control Samples		Worksheet #28 Appendix B
3.4.2 Analytical Quality Control Samples		Worksheet #28 Appendix B

QAPP Worksheet #2: Identifying Information
(UFP-QAPP Manual Section 2.2.4)

Required QAPP Element(s) and Corresponding QAPP Section(s) (US EPA, 2005a)	Required Information	Crosswalk to Related Information and Documents
3.5 Data Management Tasks		
3.5.1 Project Documentation and Records	Project Documents and Records Table	Worksheet #29
3.5.2 Data Package Deliverables	Analytical Services Table	Worksheet #30
3.5.3 Data Reporting Formats	Analytical SOPs Analytical SOP References Table	Worksheet #23 Appendix B
3.5.4 Data Handling and Management	Analytical SOPs Analytical SOP References Table	Worksheet #23 Appendix B
3.5.5 Data Tracking and Control	Analytical SOPs Analytical SOP References Table	Worksheet #23 Appendix B
Assessment/Oversite		
4.1 Assessments and Response Actions	Assessments and Response Actions	
4.4.1 Planned Assessments	Planned Project Assessments Table	Worksheet #31
4.1.2 Assessment Findings and Corrective Action Responses	Assessment Findings and Corrective Action Responses Table	Worksheet #32
4.2 QA Management Reports	QA Management Reports Table	Worksheet #33
4.3 Final Project Report		TBD
Data Review		
5.1 Overview		
5.2 Data Review Steps		
5.2.1 Step I: Verification	Verification (Step I) Process Table	Worksheet #34
5.2.2 Step II: Validation	Validation (Step II) Process Table	Worksheet #35
5.2.2.1 Step IIa Validation Activities	Validation (Steps IIa and IIb) Process Table	Worksheet #35





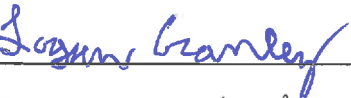
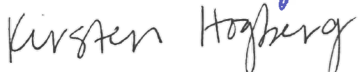
QAPP Worksheet #2: Identifying Information
(UFP-QAPP Manual Section 2.2.4)

Required QAPP Element(s) and Corresponding QAPP Section(s) (US EPA, 2005a)	Required Information	Crosswalk to Related Information and Documents
5.2.2.2 Step IIb Validation Activities	Validation (Steps IIa and IIb) Summary Table	Worksheet #36
5.2.3 Step III: Usability Assessment		
5.2.3.1 Data Limitations and Actions for Usability Assessment	Usability Assessment	Worksheet #37
5.2.3.2 Activities		
5.3 Streamlining Data Review		Not Applicable
5.3.1 Data Review Steps To Be Streamlined		
5.3.2 Criteria for Streamlining Data Review		
5.3.3 Amounts and Types of Data Appropriate for Streamlining		

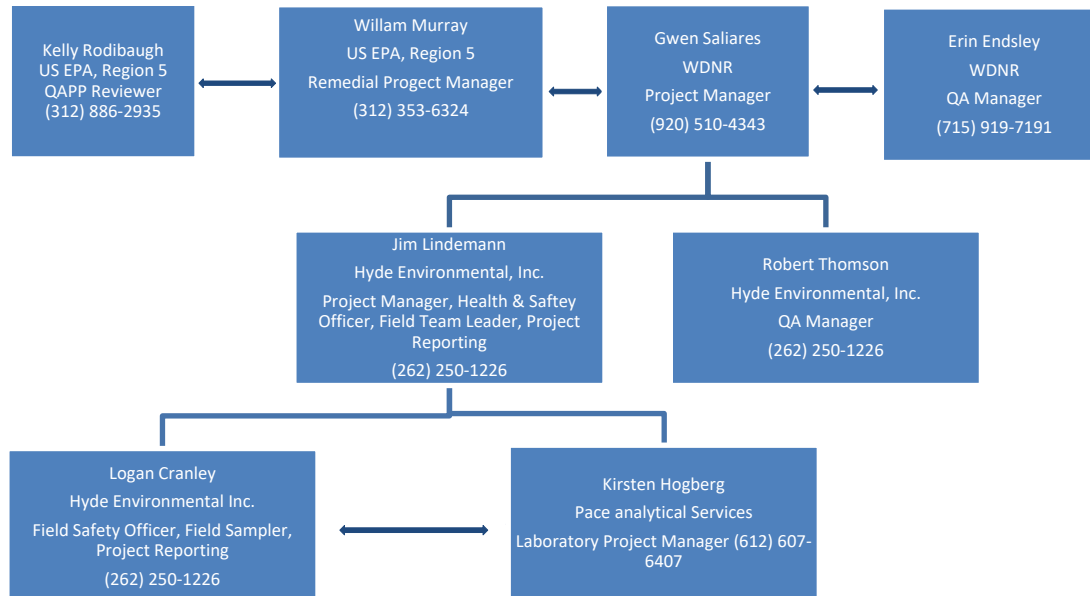
QAPP Worksheet #3: Distribution List
(UFP-QAPP Manual Section 2.3.1)

Name	Title	Organizational Affiliation	Contact Information	QA Project Plan Receipt/ Control No.
William Murray	Remedial Project Manager	US EPA Region 5	77 W Jackson Blvd, Chicago, IL 60604 (312) 353-6324 murray.williamj@epa.gov	
Kelly Rodibaugh	QAPP Reviewer	US EPA Region 5	77 W Jackson Blvd, Chicago, IL 60604 (312) 353-6324 Rodibaugh.kelly@epa.gov	
Jim Lindemann	Project Manager, Health & Safety Officer, Field Team Lead, Project Reporting	Hyde Environmental, Inc.	W175N11163 Stonewood Drive, Suite 110 Germantown WI 53022-6501 (262) 250-1226 ext. 102 jlindemann@hyde-env.com	
Robert Thomson	QA Manager	Hyde Environmental, Inc.	W175N11163 Stonewood Drive, Suite 110 Germantown WI 53022-6501 (262) 250-1226 rthomson@hyde-env.com	
Erin Endsley	QA Manager	WDNR	Wisconsin Department of Natural Resources 1701 North 4th Street Superior, WI 54880 Phone: (715) 919-7191 erin.endsley@wisconsin.gov	
Gwen Saliars	Project Manager	WDNR	Wisconsin Department of Natural Resources 625 E County Rd Y, STE. 700 Oshkosh, WI 54901 Phone: (920) 510-4343 gwen.saliars@wisconsin.gov	
Logan Cranley	Field Sampler, Field Safety Officer, Project Reporting,	Hyde Environmental, Inc.	W175N11163 Stonewood Drive, Suite 110 Germantown WI 53022-6501 (262) 250-1226 Lcranley@hyde-env.com	

QAPP Worksheet #4: Project Personnel Sign-off Sheet
(UFP-QAPP Manual Section 2.3.3)

QAPP Recipient	Title/Role	Organization	Telephone Number	Signature	Date QAPP Read
Gwen Saliars	Project Manager – Lead Organization	WDNR	(920) 510-4343		3/17/2023
Erin Endsley	QA Manager – Lead Organization	WDNR	(715) 919-7191		3/17/2023
Jim Lindemann	Project Manager, Field Team Lead, Health & Safety Officer, Project Reporting - Contractor	Hyde Environmental, Inc.	(262) 250-1226		3/6/23
Robert Thomson	QA Manager, Field/analytical data QA/QC - Contractor	Hyde Environmental, Inc.	(262) 250-1226		3/6/23
Logan Cranley	Field Sampler, Field Safety Officer, Project Reporting, - Contractor	Hyde Environmental, Inc.	(262) 250-1226		3/6/23
Kirsten Hogberg	Laboratory Project Manager - Subcontractor	Pace Analytical Services	(612) 607-6407		3/16/2023
William Murray	Remedial Project Manager	US EPA	(312) 353-6324		
Kelly Rodibaugh	QAPP Reviewer	US EPA	(312) 353-6324		

QAPP Worksheet #5: Project Organization (UFP-QAPP Manual Section 2.4.1)



QAPP Worksheet #6: Communication Pathways
(UFP-QAPP Manual Section 2.4.2)

Communication Drivers	Responsible Entity	Name	Phone Number and Email Address	Procedure (Timing, Pathways, etc.)
Point of Contact with US EPA	Remedial Project Manager	William Murray	(312) 353-6324, murray.williamj@epa.gov	All materials and information related to the project will be forwarded to RPM
Daily Progress Updates	Field Technician/Sampler	Logan Cranley Hyde Environmental, Inc.	(262) 250-1226, lcranley@hyde-env.com	Notify Hyde Project Manager/Field Team Leader of sampling progress via phone or e-mail on a daily basis
Reporting Laboratory Data Quality Issues with Project Samples (Upon Receipt or During Analysis)	Laboratory	Kirsten Hogberg Pace Analytical Services	(612) 607-6407, kirsten.hogberg@pacelabs.com	Report problems related to receipt/analysis of project field samples to Hyde Project Manager via phone or e-mail within 1 business day. These lab data quality issues will be discussed with Project Manager via phone or e-mail, and the need for corrective action determined.
Field and Analytical Corrective Actions	Project Manager or QA Manager	Jim Lindemann Hyde Environmental, Inc. Robert Thomson Hyde Environmental, Inc	(262) 250-1226, jlindemann@hyde-env.com (262) 250-1226, rthomson@hyde-env.com	Project Manager in consultation with QA Manager will determine the need for corrective action. US EPA's RPM will be notified of field or laboratory correction actions by Project Manager or QA Manager
Release of Analytical Data	QA Manager	Robert Thomson Hyde Environmental, Inc.	(262) 250-1226, rthomson@hyde-env.com	Analytical data will be transmitted by QA Manager to US EPA's RPM following verification and validation
Safety	Health & Safety Officer	Jim Lindemann Hyde Environmental, Inc.	(262) 250-1226, jlindemann@hyde-env.com	Safety plans and issues will be communicated to and from field staff.

QAPP Worksheet #7: Personnel Responsibilities and Qualification Table
(UFP-QAPP Manual Section 2.4.3)

Name	Project Title/Role	Organization	Responsibilities	Education and Experience Qualifications
Prime Contractor				
Jim Lindemann	Project Manager, Health & Safety Officer, Field Team Leader, Project Reporting	Hyde Environmental, Inc.	Overall project management – contractor, Sampling/Health & Safety oversight, report preparation (results discussion)	M.S. Hydrogeology, B.S. Water Chemistry, PG, PH, CHMM
Robert Thomson	Quality Assurance Manager	Hyde Environmental, Inc.	Independent field/analytical data QA/QC	B.S. Geology, PG
Logan Cranley	Field Safety Officer, Field Sampler ¹ , Project Reporting	Hyde Environmental, Inc.	Field sampling services, report preparation	B.A. Environmental Science
Laboratory - Subcontractor				
Kirsten Hogberg	Laboratory Project Manager	Pace Analytical Services	Laboratory services, laboratory QA/QC	B.S. Environment and Sustainability

1. Field staff is subject to change depending on sampling schedule.

QAPP Worksheet #8: Special Personnel Training Requirements Table
(UFP-QAPP Manual Section 2.4.4)

No specialized training is required for this project outside of the standard HAZWOPER and First-aid training. HAZWOPER and first-aid certifications are provided to staff through a combination of in-person and online training courses. Documentation of staff HAZWOPER and first-aid certifications and records of training completions are maintained by Hyde Environmental's safety officer at W175N11163 Stonewood Drive, Suite 110, Germantown, WI 53022-6501.

QAPP Worksheet #9: Project Scoping Session Participants
(UFP-QAPP Manual Section 2.5.1)

Name	Project Title/Role	Organization	Phone Number	E-mail Address
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US EPA met with WDNR and Hyde Environmental, Inc. on June 22, 2022 to discuss the QAPP and analytical services. The decisions made during this meeting included discussing the contents of the QAPP and the inclusion of the performance evaluation (PE) sample. The meeting also included general discussion of the site and site background.

QAPP Worksheet #10: Problem Definition
(UFP-QAPP Manual Section 2.5.2)

Hyde Environmental, Inc. has been retained by WDNR to provide sampling at the Oconomowoc Electroplating Company, Inc. (OECD) Superfund Site located in Ashippun, Dodge County, Wisconsin (Figure 1). The sampling will independently evaluate the presence/absence of Perfluorooctanoic Acid (PFOA) and Perfluorooctane Sulfonate (PFOS) compounds, collectively referred to as Per- and polyfluoroalkyl substances (PFAS), at the Site. Sampling includes all necessary aspects of sample collection and processing, including providing the necessary materials and labor at the Site, packaging, labeling, procuring laboratory services from an US EPA-approved laboratory, and responsibility for shipping or transporting samples to the commercial lab in accordance with handling procedures.

The purpose of the sampling is to: investigate the emerging contaminant, PFAS, in groundwater at the Site and prepare and submit a “PFAS Groundwater Investigation Report”.

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

The project quality objectives are defined below:

1. Statement of Problem.

Independently evaluate the presence/absence of PFAS at the Site based on the historical use of the Site.

2. Identification of the Goals of the Study.

The overall goal of the *Groundwater Monitoring* is to:

- prevent or minimize the potential for human exposure to contaminated groundwater so that health-based allowable exposure limits are not exceeded.
- prevent or minimize the potential for future off-site migration of contaminants.
- provide the WDNR with valid data of known and documented quality that will be compared to Department of Health Services recommended Preventative Action Limits (PALs).

3. Identify Information Inputs

PFAS groundwater analytical data.

4. Definition of the Study Boundaries.

The Site is located at W2573 Oak Street, Town of Ashippun, Dodge County, WI. The Site includes approximately four (4) acres of the former electroplating facility and an additional 6.5 acres of wetland, including part of Davy Creek, a tributary of the nearby Rock River. The former OEI facility is bounded by Oak St. to the northeast; Eva St. to the northwest; Elm St. to the southwest; and a Town garage and park to the southeast.

5. Analytic Approach.

Sampling monitoring wells and private potable wells for PFAS.

6. Specify Performance or Acceptance Criteria.

The sampling is being conducted to determine the presence or absence of PFAS contamination exceeding concentrations allowed by the standards in either drinking water or regulated discharges to surface water. If it is determined that there are exceedances, steps to reduce the contamination may be required.

7. Develop the Detailed Plan for Obtaining Data.

See December 2022 Field Sampling Plan and Worksheet #17 for details. For further details. Worksheets #19, 20, 24-28, and 30 will specify analysis design requirements.

QAPP Worksheet #12: Measurement Performance Criteria Table
(UFP-QAPP Manual Section 2.6.2)

Matrix	Groundwater				
Analytical Group	PFAS				
Concentration Level	Low				
Sampling Procedure	Analytical Method/SOP ¹	Data Indications (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance ^{1,2}	QC Sample Assess Error for Sampling (S), Analytical (A) or Both (S&A)
001	LC/MS/MS (Isotope Dilution)	Accuracy/Bias - Sensitivity	Current Lab Control Limits ¹	Laboratory Control Sample	A
001	LC/MS/MS (Isotope Dilution)	Accuracy/Bias - Contamination	No target PFAS ≥ QL, except common lab contaminants (3x QL) ¹	Method Blanks	A
001	LC/MS/MS (Isotope Dilution)	Accuracy/Bias - Contamination	No target PFAS ≥ QL ¹	Trip and Field Blanks	S&A
001	LC/MS/MS (Isotope Dilution)	Precision (RPD)	< 30% RPD ¹	Field Duplicates	S&A
001	LC/MS/MS (Isotope Dilution)	Precision (RPD) Accuracy/Bias (%RSD)	Current Lab Control Limits ¹	Duplicate Matrix Spikes	S&A
001	LC/MS/MS (Isotope Dilution)	Accuracy/Bias	Current Lab Control Limits ¹	Surrogate Compound Spikes	A
001	LC/MS/MS (Isotope Dilution) ¹	Completeness	≥ 85% Samples Collected; ≥ 95% Lab Measurements ¹	Data Completeness Check	S&A
001	LC/MS/MS (Isotope Dilution)	Comparability	≤ 30% RPD ¹	Duplicates	S&A
001	LC/MS/MS (Isotope Dilution)	Representativeness	80% samples collected vs. planned;	Reports, COCs	S
001	LC/MS/MS (Isotope Dilution)	Accuracy and comparability of data sets associated with samples for emerging contaminants	≥90% pass rate (acceptable analytes) ¹	Performance Evaluation sample ²	A
001	LC/MS/MS (Isotope Dilution)	Sensitivity	MDLs and RLs ¹	Calibration and Continuing Calibration Verification (CCV) Standards	A

1. See Appendix B of this document. Test method standard operating procedure, Selected 36 Per- and Polyfluoroalkyl Substances (PFAS) by LC/MS/MS Isotope Dilution, Pace ENV – Minneapolis – MIN4, Appendix B: QC Summary.

2. US EPA will provide a performance evaluation (PE) sample that will be involved in the project. Hyde will include it in the coolers sent to the lab

for analysis, request additional containers from the lab to accommodate, not alert the lab to its presence, and include results in sample data reported to US EPA. US EPA would share scoring results with WDNR. The purpose of the PE sample is to serve as a data quality indicator for accuracy and comparability of data sets associated with samples for emerging contaminants.

QAPP Worksheet #12: Measurement Performance Criteria Table (continued)
(UFP-QAPP Manual Section 2.6.2)

Field parameters (oxidation-reduction potential [ORP], dissolved oxygen [DO], pH, conductivity, temperature, and turbidity) will be measured prior to sample collection. Sample collection will only occur after these field parameters have stabilized. Stabilization criteria are defined in the December 2022 Field Sampling Plan.

Potential outliers will be identified as those samples that have a > 30% RPD¹. Missing data will be identified by comparison of the availability of sampling results for the planned locations.

Below are sample formulas for calculating applicable quality control (QC) statistics:

Percent Relative Standard Deviation (%RSD):	$\%RSD = \frac{SD}{\bar{x}} \times 100$
Standard Deviation (SD):	$SD = \sqrt{\frac{\sum_{i=1}^n (x - \bar{x})^2}{n - 1}}$
Relative Percent Difference (RPD):	$RPD = \left(\frac{x_1 - x_2}{x_1 + x_2 / 2} \right) \times 100$

QAPP Worksheet #13: Secondary Data Criteria and Limitations Table
(UFP-QAPP Manual Section 2.7)

Not Applicable

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

Sampling Tasks: All samples collected by Hyde are expected to be samples, field duplicates, equipment blanks, or a performance evaluation (PE) sample. Hyde will provide the equipment and tools necessary for the sample collection; Pace Analytical Services will provide the appropriate sample containers for sample collection. US EPA will provide the PE sample for Hyde to include with the field samples and shipped to the analytical laboratory.

1. Collect groundwater samples from the Site,
2. Collect field duplicates, equipment blank sample, and MS/MSD samples,
3. Submit PE sample and trip blanks, (the PE sample will serve as a data quality indicator for accuracy and comparability of data sets associated with samples for emerging contaminants), along with collected samples,
4. Log oversight activities and tasks in field logbook; record visual/olfactory observations, photo-document sampling activities.
5. Prepare sample documentation such as chain-of-custody, sample labels, and custody seals. Ship samples to appropriate laboratory via overnight courier.

Analysis Tasks: Pace Analytical Services (laboratory) to analyze environmental media for PFAS.

Quality Control Tasks: The following QC samples will be collected and analyzed during the sampling event: matrix spike (MS), and matrix duplicate (MSD), field duplicates (DUP), equipment blank, and trip blank.

Secondary Data: See Worksheet #13

Time and Resource Constraints: Time and resource constraints for this sampling event include, but are not limited to: (1) availability of Hyde’s field staff, (2) ability for Hyde to conform with the sampling schedule, (3) capability to ship samples to the laboratory in a timely manner to satisfy sample holding times, and, (4) restrictions for field sampling staff posed by COVID-19 protocols.

Data Management Tools: Hyde will maintain a specific project file, which will be uploaded to a secure SharePoint™ folder owned by Hyde. The Sharepoint™ folder will act as a repository for all documents collected/generated as part of the project. The complete project file will include both hardcopies, stored at Hyde’s office, and electronic data. SharePoint™ is a web-based collaborative platform that is primarily used as a secure document management and storage system.

Documentation and Records: All field activity for which Hyde has been tasked to provide oversight and observations will be documented in a

project-specific logbook in black ink. Regarding samples, the following information will be recorded in the logbook for each sample to be sent for laboratory analysis: time of collection, water quality parameters (if collected), water-levels (if collected), weather conditions, identification number, sampling location, field observation, Hyde’s sampler’s name, and analyses. Each page of the logbook will be dated, numbered, and signed by Hyde personnel. Field data records will be maintained at Hyde’s Germantown, WI office.

Assessment/Audit Tasks: See Worksheets #31 and #32

Data Review Tasks: The Hyde QA manager or designee will review the case narrative and will detail any analytical problems that may potentially affect data quality in “Data Summary Report”.

The samples will be used to evaluate the usability of the data and to monitor for potential emerging contaminants. A review of the data usability and sampling oversight will be summarized in a Report.

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

Matrix: Groundwater

Analytical Group: PFAS

Concentration Level: Low

Analyte	CAS Number	Project Quantitation Limit Goal * (ng/L)	Laboratory-specific quantitation limit ₁ (ng/L)	Laboratory-specific detection limit (ng/L)
PFAS by WDNR Guidance SOP – 33 Compounds / Determination of Selected 36 Per- and Polyfluoroalkyl Substances (PFAS) by LC/MS/MS (Isotope Dilution)				
Perfluorobutanoic acid [C4] (FC 23, Fluorad FC 23)	375-22-4	2,000	2	0.441
Perfluoropentanoic acid [C5]	2706-90-3	--	2	0.438
Perfluorohexanoic acid [C6]	307-24-4	30,000	2	0.438
Perfluoroheptanoic acid [C7]	375-85-9	--	2	0.550
Perfluorooctanoic acid [C8]	335-67-1	2	2	0.585
Perfluorononanoic acid [C9]	375-95-1	3	2	0.740
Perfluorodecanoic acid [C10]	335-76-2	60	2	0.564
Perfluoroundecanoic acid [C11]	2058-94-8	600	2	0.540
Perfluorododecanoic acid [C12]	307-55-1	100	2	0.483
Perfluorotridecanoic acid [C13]	72629-94-8	--	2	0.622
Perfluorotetradecanoic acid [C14]	376-06-7	2,000	2	0.476
Perfluorobutanesulfonic acid [C4] (FC-98)	375-73-5	90,000	1.77	0.473
Perfluoropentanesulfonic acid [C5]	2706-91-4	--	1.88	0.475
Perfluorohexanesulfonic acid [C6]	355-46-4	4	1.82	0.508
Perfluoroheptanesulfonic acid [C7]	375-92-8	--	1.90	0.411
Perfluorooctanesulfonic acid [C8] (FC 95, Fluorad FC 95)	1763-23-1	2	1.85	0.548

Analyte	CAS Number	Project Quantitation Limit Goal * (ng/L)	Laboratory-specific quantitation limit ₁ (ng/L)	Laboratory-specific detection limit (ng/L)
Perfluorononanesulfonic acid [C9]	68259-12-1	--	1.92	0.446
Perfluorodecanesulfonic acid [C10]	335-77-3	--	1.93	0.450
Perfluorododecanesulfonic acid [C12]	79780-39-5	--	1.94	0.460
4:2 fluorotelomersulfonic acid [C6]	757124-72-4	--	1.87	0.558
6:2 fluorotelomersulfonic acid [C8]	27619-97-2	--	1.90	0.645
8:2 fluorotelomersulfonic acid [C10]	39108-34-4	--	1.93	0.653
Perfluorooctanesulfonamide [C8]	754-91-6	2	2	0.818
N-Methylperfluorooctanesulfonamide [C9] (<i>Fluorad FX 12</i>)	31506-32-8	--	2	0.511
N-Ethylperfluorooctanesulfonamide [C10] (<i>Alstar, Finitron, Fluramin, FX 12, Mirex S, Sulfluramid, Volcano</i>)	4151-50-2	2	2	0.608
N-Methylperfluorooctanesulfonamidoacetic acid [C11]	2355-31-9	--	2	0.434
N-Ethylperfluorooctanesulfonamidoacetic acid [C12]	2991-50-6	2	2	0.555
N-Methylperfluorooctanesulfonamidoethanol [C11]	24448-09-7	--	2	0.329
N-Ethylperfluorooctanesulfonamidoethanol [C12] (<i>FC-10, Fluorad FC 10</i>)	1691-99-2	2	2	0.497
Hexafluoropropylene oxide dimer acid [C6] (<i>FRD-903, GenX</i>)	13252-13-6	30	2	0.529
4,8-dioxa-3H-perfluorononanoic acid [C7]	919005-14-4	600	1.89	0.514
9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid [C8]	756426-58-1	--	1.86	0.305
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid [C10]	763051-92-9	--	1.88	0.436

* Department of Health Services recommended Preventative Action Limit (PAL)

-- Not established

QAPP Worksheet #16: Project Schedule/Timeline Table
(UFP-QAPP Manual Section 2.8.2)

Activities	Organization	Dates (MM/DD/YYYY)		Deliverable	Deliverable Due Date
		Anticipated Date of Initiation	Anticipated Date of Completion		
Field Sampling	Hyde	TBD	TBD	Field data	30 days after sampling event
Laboratory Analysis	Pace Analytical Services	TBD	TBD	Level IV Laboratory Package	40 days after receipt of samples (Current laboratory TAT on PFAS samples is 40 days.)
Evaluation of Sample Usability	Hyde	TBD	TBD	Data Summary and Usability Report	30 days after receipt of data

QAPP Worksheet #17: Sampling Design and Rationale
(UFP-QAPP Manual Section 3.1.1)

The purpose of the sampling is to independently evaluate the presence/absence of Perfluorooctanoic Acid (PFOA) and Perfluorooctane Sulfonate (PFOS) compounds, collectively referred to as Per- and polyfluoroalkyl substances (PFAS), at the Site. Hyde will follow the approved Field Sampling Plan and applicable SOPs when collecting the groundwater samples. Locations were selected based on accessible sampling points within the OEI monitoring well and private potable well sampling network.

Hyde will collect groundwater samples according to accepted procedures for analysis. If a sampling location is inaccessible, it will be noted in the field notes and communicated to the project manager and RPM. The PM will decide if another location will be sampled in lieu of the inaccessible sampling location.

Groundwater samples will be analyzed for 33 PFAS Compounds.

Sample locations will be determined by the WDNR PM and Hyde will collect 100% of the total samples. Sampling locations are listed in Worksheet 18.

QC samples will be collected at the following frequencies:

- MS/MSD Samples: One per 10 environmental samples collected
 - Field Duplicate Samples: One per 10 environmental samples collected or one per day
 - Equipment Blanks: One per sampling event when equipment is used in the field to collect samples
-

QAPP Worksheet #18: Sampling Locations and Methods
(UFP-QAPP Manual Section 3.1.1)

Sampling Location	PFAS
MW-1S	X
MW-1D	X
MW-2D	X
MW-3D	X
MW-4S	X
MW-5D	X
OW-6	X
MW-9S	X
MW-105S	X
MW-105D	X
MW-105B	X
MW-12S	X
MW-12D	X
MW-12B	X
MW-13S	X
MW-13D	X
MW-14DR	X
MW-15S	X
MW-15D	X
MW-15B	X
MW-16S	X
MW-101S	X
MW-101B	X
MW-102S	X
MW-102D	X
MW-103S	X
MW-103D	X
TW-2021	X
Potable wells	
PW-03	X
PW-04	X
PW-05	X
PW-07	X
PW-08	X
PW-09	X
PW-10	X
PW-11	X

Figure 1: Sampling Location Map (attached)

QAPP Worksheet #19: Analytical SOP Requirements Table
(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference¹	Sample Volume	Containers (number, size and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/analysis)	Data Turnaround Time
Groundwater	PFAS	Low	LC/MS/MS (Isotope Dilution) ¹	250 ml	Two 250-ml polypropylene bottles	Cool to ≤ 6 °C	28 days for preparation and analysis	40 days (Current laboratory TAT on PFAS samples is 40 days.)

1. See Appendix B of this document. Test method standard operating procedure, Selected 36 Per- and Polyfluoroalkyl Substances (PFAS) by LC/MS/MS Isotope Dilution, Pace ENV – Minneapolis – MIN4, Appendix B: QC Summary.

Notes: Additional sample bottles will be required for MS/MSD, equipment blank, and field duplicate samples. Sample bottles will be obtained from Pace Analytical Services.

QAPP Worksheet #20: Field Quality Control (QC) Summary
(UFP-QAPP Manual Section 3.1.1)

The field QC methods are defined in the accepted procedures attached to this QAPP. The sampling event will include samples from 28 monitoring wells and 8 private/potable wells. Hyde will collect samples from all accessible monitoring wells and private/potable wells sampled during this event.

QC samples will be collected at the following frequencies:

- MS/MSD Samples: One per 10 environmental samples collected
- Field Duplicate Samples: One per 10 environmental samples collected or one per day
- Equipment Blanks:
One per sampling event when equipment is used in the field to collect samples

QAPP Worksheet #21: Project Sampling SOP Reference Table
(UFP-QAPP Manual Section 3.1.2)

SOP or Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified from Project Work? (Y/N)	Comments
EQASOP-GW4	US EPA Low Stress (Low Flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells (September 19, 2017)	US EPA	Logbook, Water-level indicator, Peristaltic pump/tubing, Multi-parameter meter or similar with flow-through cell ¹	N	None
SASDPROC-202-R4	US EPA Region 4 Management of Investigated Derived Waste (May 8, 2020),	US EPA	5-gallon pail, 55-gallon steel drum	N	None
Field Sampling Plan	Field Sampling Plan – PFAS Sampling, Oconomowoc Electroplating Company, Inc Superfund Site W2573 Oak Street, Ashippun, Dodge County, Wisconsin 53003	Hyde	Water-level indicator, Peristaltic pump/tubing, Multi-parameter meter or similar with flow-through cell 5-gallon pail, 55-gallon steel drum	N	None
	EPA Region 5 Analytical Services Branch PFAS Sampling Fact Sheet	US EPA	Not Applicable	N	None
SOP1001 – Low Flow Groundwater Sampling	Low Flow Groundwater Sampling Activities (November 2022)	Hyde	Water-level indicator, Peristaltic pump/tubing, Multi-parameter meter or similar with flow-through cell	N	None

SOP1003- Investigation Derived Waste	Investigation Derived Waste Handling (November 2022)	Hyde	5-gallon pail, 55-gallon steel drum	N	None
SOP1002- Data Verification and Validation	Groundwater Sampling Data Verification and Validation (November 2022)	Hyde	Not Applicable	N	None

1. Deviations from accepted procedures will be noted in field records and communicated to the Project Manager and the EPA RPM. Necessary corrective actions will be determined by the Field Sampling Manager, Project Manager, or EPA RPM.

**QAPP Worksheet #22: Field Equipment Calibration, Maintenance, Testing, and Inspection Table
(UFP-QAPP Manual Section 3.1.2.4)**

Field Equipment	Activity	Reference	Title or position of responsible person	Frequency	Acceptance Criteria	Corrective Action
Multi-parameter meter (Aqua-Troll 500)	Maintenance, Testing, Calibration	Operation manual ¹	Field sampling technician	Prior to each daily sampling event	Per operation manual ¹	Re-calibration
Water-level meter (Heron Dipper T Keck 3/8", 200')	Maintenance, Testing, Calibration	Operation manual ¹	Field sampling technician	Prior to each daily sampling event	Per operation manual ¹	Change battery
Water-level meter (Heron Dipper T Keck 3/8", 200')	Decontamination	Operation manual ¹ , Hyde SOP1001	Field sampling technician	Prior to each sampling location	N/A	N/A

1 The operation manuals for the multi-parameter meter and water-level meter are included as an attachment to the December 2022 Field Sampling Plan which is included as Appendix D of this QAPP.

QAPP Worksheet #23: Analytical SOP References Table
(UFP-QAPP Manual Section 3.2.1)

Reference Number	Title, Revision, Date and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?
10LCMS01 (internal to Pace)	ENV-SOP-MIN4-0178, Revision 3, December 13, 2021 ¹	Definitive	PFAS	LC/MS-MS	Pace Analytical Services	No
NELAP Accreditation, MN300001	Environmental Laboratory Accreditation Program Certificate through the State of Oregon, a NELAP Recognized Accreditation Body, analysis of PFAS in non-potable water ²	Definitive	PFAS	LC/MS-MS	Pace Analytical Services	No
WDNR Accreditation, Laboratory FID: 999407970	Accreditation under NR 149, Pace Analytical Services, LLC, Minneapolis, MN (expires August 31, 2023) ²	Definitive	PFAS	LC/MS-MS	Pace Analytical Services	No

1. See Appendix B of this document. Test method standard operating procedure, Selected 36 Per- and Polyfluoroalkyl Substances (PFAS) by LC/MS/MS Isotope Dilution, Pace ENV – Minneapolis – MIN4, Appendix B: QC Summary.
2. See Appendix C of this document. NELAP and WDNR Accreditations, Pace Analytical Services, LLC, Minneapolis, MN

QAPP Worksheet #24: Analytical Instrument Calibration Table
(UFP-QAPP Manual Section 3.2.2)

Analytical instrument calibration methods are defined in the laboratory's SOP, ENV-SOP-MIN4-0178 (Appendix B).

**QAPP Worksheet #25: Analytical Instrument and Equipment Maintenance, Testing and
Inspection Table
(UFP-QAPP Manual Section 3.2.2)**

Analytical instrument and laboratory equipment maintenance and testing methods are defined in the laboratory's SOP, ENV-MAN-MIN4-0001 (Appendix B of this document). Field equipment maintenance and testing methods are defined in the Worksheet #22 and in the December 2022 Field Sampling Plan (Appendix D of this document).

**QAPP Worksheet #26: Sample Handling System
(UFP-QAPP Manual Appendix A)**

SAMPLE COLLECTION, PACKAGING AND SHIPMENT
Sample Collection (Personnel/Organization): Logan Cranley, Hyde
Sample Packaging (Personnel/Organization): Logan Cranley, Hyde
Coordination of Shipment (Personnel/Organization): Logan Cranley, Hyde and Pace Analytical Services
Type of Shipment/Carrier: FedEx or Commercial Courier contracted by Hyde or Pace Analytical Services. Samples will be sent to the laboratory via overnight shipping at the end of each field day.
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): TBD by Pace Analytical Services PM
Sample Custody and Storage (Personnel/Organization): TBD by Pace Analytical Services PM
Sample Preparation (Personnel/Organization): TBD by Pace Analytical Services PM
Sample Determinative Analysis (Personnel/Organization): TBD by Pace Analytical Services PM
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): 21 days after sample analysis (Pace Analytical Services' ENV-SOP-MIN4-0008, Sample Management, Section 6.0) (see Appendix B of this document)
Sample Extract/Digestate Storage (No. of days from extraction/digestion): 21 days after sample analysis (Pace Analytical Services' ENV-SOP-MIN4-0008, Sample Management, Section 6.0) (see Appendix B of this document)
Biological Sample Storage (No. of days from sample collection): N/A
SAMPLE DISPOSAL
Personnel/Organization: TBD by Pace Analytical Services' Personnel (see Appendix B of this document)
Number of Days from Analysis: 21 days after sample analysis (Pace Analytical Services' ENV-SOP-MIN4-0008, Sample Management, Section 6.0) (see Appendix B of this document)

**QAPP Worksheet #27: Sample Handling System
(UFP-QAPP Manual Appendix A)**

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory): Field personnel are required to keep written records of field activities in a logbook or on standard field forms. All field measurements obtained, and samples collected will be recorded in the logbook or on standard field forms. Samples will be collected following the procedures documented in applicable SOPs and in the December 2022 Field Sampling Plan (Appendix D of this document).

All sample containers will be identified by using sample labels that include the unique sample identification number/name, sampler's initials, the date and time of collection, and requested analysis. Sample labels will be pre-printed or will be hand-written for each sample using PFAS free indelible ink. Sample naming conventions will follow existing OECl Site location names.

Immediately after the samples are collected and labeled, the containers will be placed in shipping coolers containing bagged, cubed ice to maintain the proper temperature range for shipment (0-6 °C). The sample IDs, number of containers, and required analyses will be recorded with other project-specific information on the chain-of-custody (COC) form. All information recorded on the COC form will be in ink, signed, and dated with no erasures. If an incorrect entry is made, the incorrect information will be crossed out with a single strike mark that is initialed and dated by the person making the erroneous entry. The correct information will be entered on the COC form adjacent to the original entry. Containers of groundwater samples will be packaged in bubble wrap bags and placed in an upright position in the cooler. The field sampler is responsible for the care and custody of the samples until they are relinquished for shipment to the laboratory. If transferring the possession of samples prior to shipping to the laboratory is necessary, the individuals relinquishing and receiving custody of the samples will sign and record the date and time on the chain-of-custody form. When sample collection activities are completed or the shipping cooler has reached its capacity, additional bubble wrap or other suitable packing material will be added to fill the cooler, the completed COC form will be placed in a zipper-seal bag, and the cooler will be secured with packing tape and custody seals for shipment. It is noted that commercial couriers (e.g., FedEx) are not required to sign the chain-of-custody form provided the form is sealed inside the shipping cooler and the custody tape/seal remains intact. Samples will be shipped for delivery to the laboratory with sufficient frequency to ensure that sample analyses with short holding time periods will be completed within the required time period.

Laboratory Sample Custody Procedures (receipt of samples, archiving, disposal): Pace Analytical Services' SOP - *ENV-SOP-MIN4-0008 v03_Sample Management-33421-2* (Appendix B of this document)

Sample Identification Procedures: The sample naming convention that will be used to uniquely identify the groundwater samples collected is provided in the **Field Sample Custody Procedures** above and in the December 2022 Field Sampling Plan (Appendix D of this document). Sample naming conventions will follow existing OECl Site location names. The laboratory's sample identification procedures are included in

Appendix B of this document.

Chain-of-custody Procedures: Field chain-of-custody procedures are provided in the first section, above and the December 2022 Field Sampling Plan (Appendix D of this document). Laboratory chain-of-custody procedures are included in Pace Analytical Services' SOP - *ENV-SOP-MIN4-0008 v03_ Sample Management-33421-2* (Appendix B of this document)

QAPP Worksheet #28: Analytical Quality Control and Corrective Action
(UFP-QAPP Manual Section 3.4)

The laboratory quality control (QC) system is defined in the Pace Analytical Services' SOP, *ENV-MAN-MIN4-0001* (Appendix B of this document).

QAPP Worksheet #29: Project Documents and Records Table
(UFP-QAPP Manual Section 3.4)

Document	Where Maintained
Field notes/logbook	Project file (field data), Hyde's office, Sharepoint™ folder, SEMS Superfund Enterprise Management System
Chain of custody forms	Project file (laboratory data), Hyde's office, Sharepoint™ folder, SEMS Superfund Enterprise Management System, data will be uploaded to the EPA Region 5 Superfund Site: https://www.epa.gov/superfund/region-5-superfund-electronic-data-submission
Laboratory raw data package (Level IV)	Project file (laboratory data), Hyde's office, Sharepoint™ folder, Pace Analytical Services' project file (laboratory data), SEMS Superfund Enterprise Management System; data will be uploaded to the EPA Region 5 Superfund Site: https://www.epa.gov/superfund/region-5-superfund-electronic-data-submission
Laboratory equipment calibration logs	Project file (laboratory data), Hyde's office, Sharepoint™ folder, Pace Analytical Services' project file; SEMS Superfund Enterprise Management System; project file (laboratory data); data will be uploaded to the EPA Region 5 Superfund Site: https://www.epa.gov/superfund/region-5-superfund-electronic-data-submission
Validated data	Project file (laboratory data), Hyde's office, Sharepoint™ folder, SEMS Superfund Enterprise Management System, data will be uploaded to the EPA Region 5 Superfund Site: https://www.epa.gov/superfund/region-5-superfund-electronic-data-submission
Daily activities summary reports	Project file (data and reports), Hyde's office, Sharepoint™ folder, SEMS Superfund Enterprise Management System

QAPP Worksheet #30: Analytical Services Table
(UFP-QAPP Manual Section 3.5.2.3)

Matrix	Analytical Group	Concentration Level	Sample Locations/ ID Numbers	Analytical SOP	Data Package Turnaround Time¹	Laboratory/ Organization (Name and Address, Contact Person and Telephone Number)	Backup Laboratory/Organization (Name and Address, Contact Person and Telephone Number)
Groundwater	PFAS	Low	All locations	<i>Pace WDNR SOP MN 062222 Determination by LC_MS_MS (Appendix B of this document)</i>	40 days	Pace Analytical Services 1800 Elm Street Southeast, Minneapolis, MN 55414 Kirsten Hogberg (612) 607-6407	Pace Analytical Services

1. Samples will be sent to the laboratory via overnight delivery at the end of each field day. Current laboratory TAT on PFAS samples is 40 days.

**QAPP Worksheet #31: Planned Project Assessments Table
(UFP-QAPP Manual Section 4.1.1)**

Assessment Type	Frequency	Internal of External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (Title and Organization)	Person(s) Responsible for Responding to Assessment Findings (Title and Organization)	Person(s) Responsible for Identifying and Implementing CA (Title and Organization)	Person(s) Responsible for Monitoring Effectiveness of CA (Title and Organization)
Field Sampling Technical Systems Audit (TSA)	At the direction of the Hyde PM, or QA Manager	Internal	EPA	EPA QA Officer	Hyde PM	Hyde Field Team Leader	Hyde Field Team Leader
Field Assessment	At the direction of US EPA if requested	Internal	Hyde	Hyde QA Manager or Field Sampling Lead	Hyde PM	Hyde Field Team Leader	Hyde Field Team Leader
Performance Evaluation	At initial Sampling	Internal	EPA	EPA QA Officer	EPA QA Officer	EPA QA Officer	EPA QA Officer

**QAPP Worksheet #32: Assessment Findings and Corrective Action Responses
(UFP-QAPP Manual Section 4.1.2)**

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (Name, Title and Organization)	Timeframe of Notification	Nature of CA Response Documentation	Individual(s) Receiving CA Response (Name, Title and Organization)	Timeframe for Response
Field Sampling Technical Systems Audit (TSA)	Technical Memorandum	Jim Lindemann, Hyde PM	24 hours	Technical Memorandum	Hyde PM	24 hours

US EPA will review the results of the PE sample. If greater than or equal to 90% of analytes pass per the vendor’s scoring report, no corrective action will be taken. If less than 90% of analytes pass per the vendors scoring report, the data will be investigated including field and analytical records to attempt to identify a root cause of the failure. If one can be determined, US EPA will assess potential corrective measures with appropriate parties and implement them with that party(ies) as may be possible. US EPA will monitor the corrective action as the situation warrants and in coordination with participating party(ies).

**QAPP Worksheet #33: QA Management Reports Table
(UFP-QAPP Manual Section 4.2)**

Type of Report	Frequency	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (Name, Title, Organization)	Report Recipient(s) (Title and Organization)
Weekly Activities Summary Report Letters ¹	Weekly (anticipated one week of field work)	TBD	Hyde QA Manager	US EPA RPM, US EPA QA Manager, WDNR PM

1. Weekly Activities Summary Report Letters will include any QA issues and how they are addressed

**QAPP Worksheet #34: Verification (Step I) Process Table
(UFP-QAPP Manual Section 5.2.1)**

Verification Input	Description	Internal/External	Person(s) Responsible for Verification (Name and Organization)
Chain-of-Custody forms	Chain-of-custody forms will be reviewed internally when they are completed and verified against the packed sample coolers they represent. The shipper's signature on the chain-of-custody should be initialed by the reviewer, a copy of the chain-of-custody form retained in the project file, and the original and remaining copies taped inside the cooler for shipment.	Internal	Jim Lindemann, Hyde Field Team Lead
Field notes/logbook	Field notes will be reviewed internally and placed in the project file. A copy of the field notes will be attached to the final report.	Internal	Jim Lindemann, Hyde Field Team Lead
Laboratory data	All laboratory data packages will be verified internally by the laboratory performing the work for completeness and technical accuracy prior to submittal.	Internal	Pace Analytical Services
	All received data packages will be verified externally according to the data validation procedures specified in Worksheet #35.	External	Robert Thomson, Hyde QA Manager

Hyde's Field Team Lead has the authority to stop work if adverse conditions that could impact the quality of the data are identified. Deficiencies observed in the field will be corrected immediately and noted in field sheets.

Verification Input	Description	Person(s) Responsible for Verification (Name and Organization)
Chain-of-Custody forms	Examine traceability of samples from sample collection to sample analysis	Robert Thomson, Hyde QA Manager
Holding time	Confirm that holding time requirements are met	Pace Analytical Services, Robert Thomson, Hyde QA Manager
Laboratory Deliverable	Review package for completeness. Review case narrative for discrepancies/findings and follow up where needed. Level IV laboratory reports will be requested from the laboratory.	Robert Thomson, Hyde QA Manager
Analytical Method	Confirm that analytical methods are specified in QAPP	Robert Thomson, Hyde QA Manager
Performance Criteria	Confirm that QC samples meet specified performance criteria; document any deviations in data evaluation summary report.	Robert Thomson, Hyde QA Manager

QAPP Worksheet #36: Validation (Step IIb) Process Table
(UFP-QAPP Manual Section 5.2.2)

Step IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria¹	Data Validator (Title and Organization)
IIb	Groundwater	PFAS	Low	(see below)	Environmental Services and Assistance Team (ESAT), contractors to US EPA Superfund

1 Data validation will conform with the following guidance documents and SOPs (including associated checklists):

- EPA 910-R-18-001, November 2018, Data Review and Validation guidelines for Perfluoroalkyl Substances (PFASs) Analyzed Using EPA Method 537;
- Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use, OSWER No. 9200.1-85, EPA 540-R-08-005, January 2009;
- Wisconsin guidance document - Wisconsin PFAS Aqueous (Non-Potable Water) and Non-Aqueous Matrices Method Expectations, Version 12.16.2019, Per- and Polyfluorinated Alkyl Substances (PFAS) Analysis Using Isotope Dilution by LC/MS/MS;
- Pace Analytical Services' SOP - Determination of Selected 36 Per- and Polyfluoroalkyl Substances (PFAS) by LC/MS/MS (Isotope Dilution);
- Hyde Environmental' s SOP - Groundwater Sampling Data Verification and Validation, SOP 1002.

Lab-Generated Data will be validated by ESAT/ICF Inc., independent from field collection activities.

Field-Generated Data will be verified by Hyde's QA Manager, independent from field collection activities.

Issues with data quality that are identified by the validator will be documented and conveyed to the Hyde PM and the US EPA RPM to determine an appropriate resolution.

QAPP Worksheet #37: Usability Assessment
(UFP-QAPP Manual Section 5.2.3)

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used: A team of Hyde personnel will perform the data usability assessment. Hyde's project manager will be responsible for information in the usability assessment. The project manager will also be responsible for assigning task work to the individual task members who will be supporting the data usability assessment. Note that the data usability assessment will be conducted on validated data. The results of the data usability assessment will be presented in the final project report.

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

Precision – Results of laboratory and field duplicates will be presented separately in tabular format. For each duplicate pair, the RPD will be calculated for each analyte whose original and duplicate values are both greater than or equal to the QL. The RPDs will be checked against the measurement performance criteria presented in Worksheet #12. The RPDs exceeding criteria will be identified in the tables. A discussion will follow summarizing the laboratory precision results. Any conclusions about the precision of the analyses will be drawn, and any limitations on the use of the data will be described.

Accuracy/Bias – The results for each blank will be checked against the measurement performance criteria presented in Worksheet #12. A discussion will follow summarizing the laboratory accuracy/bias results. Any conclusions about the accuracy/bias of the analyses based on contamination will be drawn, and any limitations on the use of the data will be described.

Overall Accuracy/Bias – These results will be compared to the requirements listed in Worksheet #12. A discussion will follow summarizing overall accuracy/bias results. Any conclusions about the overall accuracy/bias of the analyses will be drawn, and any limitations on the use of the data will be described.

Sensitivity – The results for each analyte will be checked against the measurement performance criteria presented in Worksheet #12 and crosschecked against the quantitation limits (QLs) presented in Worksheet #15. A discussion will follow summarizing the laboratory sensitivity results. Any conclusions about the sensitivity of the analyses will be drawn, and any limitations on the use of the data will be described.

Representativeness – The samples collected are considered representative of Site conditions, as long as completeness criteria in Worksheet #12 are met.

Comparability – The results of this study will be used as a benchmark for determining comparability for data collected during any potential future sampling events using the same or similar sampling and analytical SOPs.

Completeness – A completeness check will be performed on all data generated by the laboratory. Completeness criteria are presented in Worksheet #12. Completeness will be calculated as the number of data points for each analyte and individual matrix that meet the measurement performance criteria for precision, accuracy/bias, and sensitivity, divided by the total number of data points for each analyte.

QAPP Worksheet #37: Usability Assessment

(UFP-QAPP Manual Section 5.2.3)

A discussion will follow summarizing the calculation of data completeness. Any conclusions about the completeness of the data for each analyte will be drawn, and any limitations on the use of the data will be described.

Identify the personnel responsible for performing the usability assessment: Hyde's QA manager will review analytical data to assess the usability of the data. Hyde's Project Manager will review the RPDs for samples and assess the overall usability of the data set in close consultation with the US EPA and WDNR.

Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies: The usability assessment will be documented in the data validation report, which will be generated 30 days after final analytical results are received from the laboratory.

Appendix B _____

**ENV-SOP-MIN4-0178, Determination of Selected
PFAS by LC/MS/MS**


**ENV-MAN-MIN4-0001, Pace Analytical, *Quality
Manual***

**ENV-SOP-MIN4-0008, Pace Analytical, *Sample
Management***

**ENV-SOP-MIN4-0184 v01_Data and Records
Archival-30647-1**

(Lab SOPs and Chain of Custody)

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	ENV-SOP-MIN4-0178 v04_Determination of Per- and Polyfluoroalkyl Substances (PFAS) by LC/MS/MS
	Effective Date: 12/28/2022

Management Approval:

Keith Sturgeon Approved on 12/28/2022 4:58:03 PM

Aileen Stacks Approved on 12/27/2022 2:05:04 PM

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes the laboratory procedure for the determination of per- and polyfluoroalkyl substances (PFAS) by LC/MS/MS technology, per Table B-15 of the Department of Defense Quality Systems Manual (DoD QSM v5.3, Table B-15); per the “Wisconsin PFAS Aqueous (Non-Potable Water) and Non-Aqueous Matrices Method Expectations” by the Wisconsin Department of Natural Resources (WIDNR); and per the “Guidance for Per- and Polyfluoroalkyl Substances” by the Minnesota Pollution Control Agency (MPCA); and USEPA Method 537.1.

1.1 Target Analyte List and Limits of Quantitation (LOQ)

The target analytes that can be determined by this SOP and the associated LOQ is provided in Table 1, Appendix A.

1.2 Applicable Matrices

This SOP is applicable to non-potable waters, leachate, solid (e.g., soil, sediment, and wipe), tissue matrices, and “articles of commerce” (Elastomers, agglomerates, extruded and molded plastics, etc., requiring Cryo-mill preparation).

2.0 SUMMARY OF METHOD

A 250-mL water sample is fortified with a known quantity of isotope dilution extracted internal standards (EIS) and then passed through a solid phase extraction (SPE) cartridge (e.g., Strata™ PFAS, WAX/GCB sorbent, weak anion exchange mixed-mode) to extract the method analytes and EIS. The analytes and EIS are eluted from the cartridge with a small amount of ammonia/methanol solution.

The method for the analysis of PFAS in solid materials extracts 5 g of material with a total of 9-mL aliquot of 0.4% ammonia/methanol. The extract is treated with 50 mg ENVI-Carb™ and filtered prior to nitrogen concentration. For tissue samples, 2 g of material is extracted with 7 mL of 0.6% ammonia acetonitrile for 16 hours. The extract is treated with ENVI-Carb™ and filtered prior to SPE cleanup.


The water or solid sample extract is concentrated to ~0.5 mL while the tissue extract is concentrated to ~0.2 mL with nitrogen and then brought to 1 mL with 96:4% (vol/vol) methanol:H₂O solution prior to LC/MS/MS analysis. A 3 µL injection is made into a Liquid Chromatography (LC) System equipped with a C18 column that is interfaced to a tandem mass spectrometer (MS/MS). The concentration of each analyte is determined by using the isotope dilution and internal standard techniques, depending on target analyte. EIS is added to all calibration standards, field samples, blanks and QC samples to monitor the extraction efficiency of the method analytes.

3.0 INTERFERENCES

- All glassware must be meticulously cleaned. Wash glassware with non-phosphate alkaline detergent and deionized (DI) water, rinse with DI water and reagent water, followed by a methanol rinse. Non-volumetric glassware can be heated in a muffle furnace at 400°C for 2 h or solvent rinsed. Volumetric glassware should be solvent rinsed and not be heated in an oven above 120 °C. Store clean glassware inverted or capped.

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Do not cover with aluminum foil because PFAS can be potentially transferred from the aluminum foil to the glassware.

NOTE: PFAS standards, extracts and samples should not come into contact with any glass containers or pipettes as these analytes can potentially adsorb to glass surfaces. PFAS analytes and EIS commercially purchased in glass ampoules are acceptable; however, all subsequent transfers or dilutions performed by the analyst must be prepared and stored in polypropylene or equivalent containers.

- Method interferences may be caused by contaminants in solvents, reagents (including reagent water), sample bottles and caps, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the chromatograms. The method analytes in this method can also be found in many common laboratory supplies and equipment, such as PTFE (polytetrafluoroethylene) products, LC solvent lines, methanol, aluminum foil, SPE sample transfer lines, etc. All items such as these must be routinely demonstrated to be free from interferences (less than 1/2 the RL) under the conditions of the analysis by analyzing method blanks.

Subtracting blank values from sample results is not permitted.

- 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 water. Humic and/or fulvic material can be co-extracted during SPE and high levels can cause enhancement and/or suppression in the electrospray ionization source or low recoveries on the SPE sorbent. Total organic carbon (TOC) is a good indicator of humic content of the sample. Under the LC conditions used during method development, matrix effects due to TOC were not observed.
- SPE cartridges can be a source of interferences. The analysis of field and method blanks can provide important information regarding the presence or absence of such interferences. Brands and lots of SPE devices should be tested to ensure that contamination does not preclude analyte identification and quantitation.


4.0 DEFINITIONS

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

- **Confirmation Ion:** One of the product ions used to help qualitatively confirm presence of the analytes. The product ion chosen is typically one of the remaining ions with high sensitivity and minimum interferences after the quantitation ion has been chosen. Not all precursor ions provide confirmation ions.
- **Extraction Internal Dilution standards (EIS):** Isotopically labeled internal standards that undergo the same extraction and analysis as the other analytes in the sample. The EIS are added to the sample at the beginning of the procedure before extraction, centrifugation, filtering, or phase separation. Ideally, there are exact isotopically labeled analogs of the native analytes so that identical behavior can be assumed. The recoveries of these standards are used to adjust the native analyte results.
- **Internal Standard Quantitation:** Measurement of native analytes using an alternate analog isotope (one that has the same chemical behavior and is close in retention time to the native analyte), thus providing a close approximation of matrix effects and losses that can occur during the preparation and analysis. The native analyte concentration is adjusted for the recovery of the alternate analog isotope. An alternate analog isotope is typically used when an exact analog

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isotope is not available.

- **Isotope Dilution Quantification:** Measurement of native analytes using an exact analog isotope of the native analyte. The native analyte concentration is adjusted for the recovery of the exact analog isotope that has been included in the preparatory and analytical procedure.
- **Precursor Ion:** For the purpose of this method, the precursor ion is the deprotonated molecule ($[M-H]^-$) of the method analyte. In MS/MS, the precursor ion is mass selected and fragmented by collisionally activated dissociation to produce distinctive product ions of smaller m/z .
- **Product Ion:** For the purpose of this method, a product ion is one of the fragment ions produced in MS/MS by collisional activated dissociation of the precursor ion.
- **Primary Dilution Standard (PDS) solution** – A solution containing the analytes prepared in the laboratory from stock standard solutions and diluted as needed to prepare calibration solutions and other needed analyte solutions.
- **Preparation Batch:** A group of up to 20 field samples (not including QC samples) extracted together by the same person(s) during a workday (24 hours) using the same lot of SPE devices, solvents, surrogate, internal standard and fortifying solutions. Required QC samples include MB, LCS, MS, and MSD.
- **Quantitation Ion:** One of the product ions used to quantitate analyte concentrations. The product ion chosen is typically one of high sensitivity and minimum interference.

5.0 HEALTH AND SAFETY


Contact your supervisor or local safety coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure

The following sections provide general health and safety information about chemicals and materials that may be present in the laboratory.

- 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 (EHS) policies and procedures specified in this SOP and in the Pace® Chemical Hygiene / Safety Manual (COR-MAN-0001).
- 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. For procedures that require use of acids, use acids in a fume hood whenever possible with PPE designed for handling 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. For procedures that that emit large volumes of solvents (evaporation/concentration processes), these activities must be performed in a fume hood or apparatus that reduces exposure.

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6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME & STORAGE

The laboratory provides containers for the collection of samples upon client request. Refer to laboratory SOP ENV-SOP-MIN4-0009 *Bottle Preparation* (current version or equivalent replacement) for procedures related to preparation of bottle kits for the test method(s) associated with this SOP.

The laboratory does not perform sample collection or field measurements for this test method. Samples should be collected in accordance with a sampling plan and sampling procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

Container Type, Minimum Sample Amount, Preservation, and Holding Time Requirements:

Matrix	Container Size & Type	Required Sample Amount ¹	Preservation	Holding Time
Aqueous	HDPE bottle fitted with polyethylene screw-cap lid	250 mL	Thermal: <6 °C but >0 °C Chemical: N/A	Collection to Prep: 28 Days Prep to Analysis: 28 Days
Solid	HDPE bottle fitted with polyethylene screw-cap lid	5 g	Thermal: <6 °C but >0 °C Chemical: N/A	Collection to Prep: 28 Days Prep to Analysis: 28 Days
Tissue	HDPE bottle fitted with polyethylene screw-cap lid	2 g	Thermal: Frozen Chemical: N/A	Collection to Prep: 1 year Prep to Analysis: 30 Days
Articles of Commerce	No specific requirements	5 g	Thermal: N/A Chemical: N/A	Collection to Prep: 1 year Prep to Analysis: 30 Days

¹ Amount of sample required for each discrete test.

Thermal preservation is checked and recorded on receipt in accordance with laboratory SOP ENV-SOP-MIN4-0008 *Sample Management* (current version or equivalent replacement). Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are stored at 0-6°C until sample preparation. Prepared samples (extracts, digestates, distillates, other) are stored at 0-6°C until sample analysis.

After analysis, samples are retained as stated in the Pace® standard terms and conditions, unless otherwise specified in the analytical services contract. Samples are then disposed of in accordance with Federal, State, and Local regulations.


7.0 EQUIPMENT & SUPPLIES

7.1 Equipment

Equipment	Description	Vendor / Item #
Extraction manifold	Phenomenex Strata™ PFAS, WAX/GCB, 200 mg/50 mg 6 mL	Phenomenex, Cat# CS0-9207 or equivalent
Analytical column	An automatic/robotic sample preparation system designed for use with SPE cartridges	Supelco Cat# 57030 and 57275 or equivalent
HPLC	1100/1290 infinity series/NexeraXR	Agilent/Shimadzu
MS	API 4000/ quadrupole/Agilent 6495 TQ	Sciex/Agilent

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Equipment	Description	Vendor / Item #
Analyst®	Data acquisition software	Version 1.6.3
MassHunter Data Acquisition	Data acquisition software	Version 10.1
Multiquant™	Data processing software	Version 3.0.2
MassHunter Quantitative Analysis	Data acquisition software	Version 10.2
Avalon	Data reporting software	See master list for current version
Horizon LIMS	Data reporting software	Version 11.2.0, or equivalent replacement
Nitrogen evaporator	N-EVAP™ 112 nitrogen evaporator equivalent nitrogen evaporator/heated waterbath capable of heating 25-60°C	Oasys Heating system (Berlin, MA, USA)
Balance	Electronic, capable of weighing to 0.001 g or equivalent.	NA
Syringe pump	Model # NE-300 or equivalent system capable of delivering variable flow rates.	New Era Pump Systems, Inc
Ultrasonicator	Branson ultrasonicator	Branson Model 8510
Adjustable auto-pipettors	Ranges 10-100 µL, 100-1000 µL, and 1000-5000 µL. Laboratory or aspirator vacuum system	NA
Vacuum extraction manifold	A manual vacuum manifold with Visiprep volume sampler (Supelco Cat# 57030 and 57275 or equivalent) for extraction, or an automatic/robotic sample preparation system designed for use with SPE cartridges, may be used if all QC requirements are met.	Supelco Cat# 57250-U and 57275 or equivalent


7.2 Supplies

Supply	Description	Vendor / Item #
SPE cartridge	Phenomenex Strata™ PFAS, WAX/GCB, 200 mg/50 mg 6 mL	Phenomenex, Cat# CS0-9207 or equivalent
Sample container	High density polyethylene (HDPE) or polypropylene, 250 mL, wide mouth, with screw top	C&C Container, Cat# 183277
Centrifuge tube and cap	15-mL and 50-mL conical polypropylene tubes with polypropylene screw caps for collection and storage of the extracts	BD Falcon, P/N 352096 and P/N352070
Polypropylene bottles	4-mL narrow-mouth polypropylene bottles	Thermo Cat# 2006-9125
Polypropylene bottles	15-mL narrow-mouth polypropylene bottles	Thermo Cat# 2002-9050
Autosampler vials	Polypropylene 0.3-mL autosampler vials with polypropylene caps	Phenomenex Cat# AR0-9995-12-C
ENVI-Carb	Supelclean™ ENVI-Carb™ SPE Bulk Packing	Sigma Aldrich, Cat# 57210-U

8.0 REAGENTS & STANDARDS

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8.1 Reagents

LC/MS grade or equivalent is used (Fisher equivalent is Optima). Other grades may be used, provided it is first determined that the reagent is of sufficiently high purity to permit its use without lessening the quality of the determination. Fisher solvents are preferred for mobile phases (water, methanol, acetonitrile) as the one liter bottles can be directly loaded on the instrument removing a transfer step with the inherent low level contamination.

Reagent	Description / Concentration	Vendor / Item # / Requirements
Ammonium Acetate (NH ₄ C ₂ H ₃ O ₂ , CAS# 631-61-8)	Optima LC/MS grade, demonstrated to be free of analytes and interferences	Fisher or equivalent
Acetonitrile (CH ₃ CN, CAS# 75-05-8)	Optima HPLC grade	Fisher or equivalent Cat# A955-4
Ammonium Hydroxide (NH ₄ OH, 28-30% in water)	Certified ACS Plus grade demonstrated to be free of analytes and interferences	Fisher or equivalent
Ammonia/Methanol Solution	Optima HPLC grade, w = 0.4% mass fraction. Mix 1.44 mL of 28-30% ammonia solution with 99.28 mL of methanol	Fisher or equivalent
Glacial Acetic Acid (C ₂ H ₃ CO ₂ H, CAS# 64-19-7)	HPLC grade. Demonstrated to be free of analytes and interferences	VWR Analytical Cat# BDH20108
Sodium hydroxide (NaOH, 1310-73-2)	Certified ACS. High purity demonstrated to be free of analytes and interferences	Fisher, or equivalent
Methanol (CH ₃ OH, CAS# 67-56-1)	Optima HPLC grade, demonstrated to be free of analytes and interferences	Fisher, or equivalent
Reagent water (H ₂ O, CAS# 7732-18-5)	Optima HPLC grade, demonstrated to be free of analytes and interferences	Fisher or equivalent Cat# W7-4
Aqueous Mobile Phase (20 mM Ammonium acetate)	To prepare 1 L, add 1.54 g ammonium acetate to 1 L of reagent water.	Fisher or equivalent
Acetate Buffer (25 mM, pH 4)	Mix 0.5 mL of acetic acid with 349.5 mL of water. Dissolve 0.116 g of ammonium acetate in 60 mL of water. Mix 200 mL of the diluted acetic acid with 50 mL of the ammonium acetate solution	Fisher or Equivalent
PPG Tuning Solutions	Instrument tuning compound. Using standards chemical kit with low/high concentration is recommend, however solution can be prepared from a neat material.	Sciex P/N 4406127
Ottawa Sand	To prepare method blank, LCSs, for the extraction of soil samples	EMD or equivalent Cat# SX0075-3
Nitrogen (N ₂)	Nitrogen aids in aerosol generation of the ESI liquid spray and is used as collision gas in some MS/MS instruments. The nitrogen (Ultra High Purity or equivalent) used should meet or exceed instrument manufacturer's specifications	Ultra-High Purity or equivalent
Canola Oil	Canola oil, or equivalent, for Oil quality control sample matrix,	Local grocery store

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Reagent	Description / Concentration	Vendor / Item # / Requirements
Lake Michigan Fish Tissue	Standard reference materials (SRM) for tissue analysis	NIST, 1947

8.2 Standards

Non-neat standards purchased from vendors that are used for the preparation of working standards. Standards containing both branched and linear isomers must be used when commercially available. If not available, the total response of the analyte must be integrated, (i.e., accounting for peaks that are identified as linear and branched isomers) and quantitated using a calibration curve which includes the linear isomer only for that analyte, i.e. PFOA.

If no expiration date is assigned by the vendor, expiration date is 1 year from the date of receipt. For open stock standards, the expiration date is 1 year for the open date.

PFBS, PFPeS, PFHxS, PFHpS, PFOS, PFNS, PFDS, 4:2FTS, 6:2FTS, 8:2FTS, DONA, 9Cl-PF3ONS and 11Cl-PF3OUdS are not available as the acid form, but rather as their corresponding salts, such as Na⁺ and K⁺. These salts are acceptable for use as stock standards as long as the weight is corrected for the salt (see calculation in section 10.2.5).

NOTE: All standards purchased are greater than or equal to 98% purity, therefore the weight can be used without correction to calculate the concentration of stock standards. Primary stock standards are stored at $\leq 4^{\circ}\text{C} \pm 2^{\circ}\text{C}$. Stock solution is brought to room temperature before using. PFAS may be purchased in glass ampoules, however all further solutions and storage is in polypropylene or equivalent containers.

Standard	Description / Concentration	Vendor / Item # / Requirements
PFAC-30PAR	Various- see Attachment I	Wellington Laboratories
PFAC-8Native	~50 µg/mL - see Attachment I	Wellington Laboratories
PFAC-24PAR	Various- see Attachment I	Wellington Laboratories
PFAC-12Nativev	Various- see Attachment I	Wellington Laboratories
MPFAC-24ES	Various- see Attachment I	Wellington Laboratories
MPFAC-6ES	50 µg/mL	Wellington Laboratories
T-PFOA	50 µg/mL	Wellington Laboratories

Store standards at room temperature. Expires 180 days from prep.

NOTE: Stock standards are stored at $\leq 4^{\circ}\text{C}$, $\pm 2^{\circ}\text{C}$. Primary dilution standards are stored at room temperature to prevent adsorption of the method analytes onto the container surfaces that may occur when refrigerated. Storing the standards at room temperature will also minimize daily imprecision due to the potential of inadequate room temperature stabilization.

8.3 Formulations

8.3.1 537 Mix

Mix 40 mL of Optima grade Water with 960 mL of Optima grade Methanol.


NOTE: Expires 1 year from prep.

8.3.2 PFAC_EIS – PFAC (Extracted Internal Standards) (0.05 µg/mL, 25 EIS)

1) Dissolve 40 µL of each MPFAC-6ES standard in 537 Mix.

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- 2) Dilute to 2 mL.
- 3) Dissolve each 1 mL of MPFAC-24ES and 10.3.2.1 mix solution in 537 Mix.
- 4) Dilute to 20 mL.
- 5) Add 100 µL to each field sample, standard, blanks and QC samples prior to extraction.

NOTE: Used for curve and sample prep. Store at room temperature. Expires 180 days from prep.

8.3.3 PFAC_Native Spike Solution

- 1) Dissolve 100 µL of each PFAC-8Native in 537 Mix.
- 2) Dilute to 5 mL.

8.3.4 PFAC_Native Spike 1 (~0.2 µg/mL)

- 1) Dissolve 1 mL of PFAC-30PAR and 1 mL of 9.2.1.16.1 mix solution to 537 Mix.
- 2) Dilute to 5 mL.

8.3.5 PFAC_Native Spike 2 (~0.050 µg/mL)

- 1) Dissolve 2.5 mL of PFAC_Native Spike 1 to 537 Mix.
- 2) Dilute to 10 mL.

NOTE: Used for curve prep and sample extraction. Store at room temperature. Expires 180 days from prep.

8.3.6 PFAC_ICV Spike Solution

- 1) Dissolve each of 100 µL PFAC-12Native in 537 Mix.
- 2) Dilute to 2.5 mL.

8.3.7 PFAC_ICV Spike 1 (~0.2 µg/mL)

- 1) Dissolve 1 mL of PFAC-24PAR and 1 mL of 9.2.1.17.1 mix solution to 537 Mix.
- 2) Dilute to 10 mL.

8.3.8 PFAC_ICV Spike 2 (~0.050 µg/mL)

- 1) Dissolve 40 µL of T-PFOA stock solution in 537 Mix.
- 2) Dilute to 1 mL.

NOTE: Expires 180 days from prep.


8.3.9 Isomer Check PDS – Isomer check Qualitative primary standard Spike

- 1) Dissolve 40 µL of T-PFOA stock solution in 537 Mix.
- 2) Dilute to 1 mL.

NOTE: Expires 180 days from prep.

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8.3.10 PFOA Qualitative Dilution Standard Spike

- 1) Dissolve 50 µL PFOA qualitative primary standard spike in 537 Mix.
- 2) Dilute to 2 mL.

NOTE: Expires 180 days from prep.

9.0 PROCEDURE

9.1 Equipment Preparation

9.1.1 Support Equipment

For additional information regarding support equipment requirements, including documentation and calibration verifications, see laboratory SOP ENV-SOP-MIN4-0161 *Support Equipment* (current version or equivalent replacement).

9.1.2 Instrument Set Up

9.1.2.1 LC System

Liquid chromatography (LC) system with binary pump, autosampler, column heater. All solvent lines were replaced with (polyether ether ketone) PEEK tubing. PFAS isolator column (Phenomenex Luna® 30 × 3 mm 5 µm C18 reverse phase LC column, Cat# 00A-4252-Y0) and stainless-steel tubing installed between the mixing chamber and injection port. Other equivalent automated LC system capable of reproducibly injecting up to 5-µL aliquots and performing binary linear gradients at a constant flow rate near the flow rate used for development of this method (0.5 mL/min) may be used.

9.1.2.2 LC/MS/MS


The LC/MS/MS must be capable of negative ion electrospray ionization (ESI) near the suggested LC flow rate of 0.6 mL/min. The system must be capable of performing MS/MS to produce unique product ions for the method analytes within specified retention time segments. A minimum of 10 scans across the chromatographic peak is required to ensure adequate precision.

9.1.2.3 Routine Instrument Operating Conditions

Injector	Syringe Size	100 µL			
	Sample Loop vol.	40 µL			
	Injection Volume:	3 µL			
	Needle Wash 1	100% Methanol			
Pump	Flow rate	400 µL/min			
	Flow method	Gradient			
	Mobile Phase 1	20 mM Ammonium Acetate H2O			
	Mobile Phase 2	LCMS Acetonitrile			
	Gradient Program	Time	% Mobile Phase 1	% Mobile Phase 2	
		Initial	90	10	
		0.5	90	10	
8.0		20	80		

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		10.0	20	80
		10.1	5	95
		12.0	5	95
		12.1	90	10
		15.0	90	10
Column	Type:	Phenomenex Gemini® (or equivalents)		
	Part Number:	00D-4439-Y0		
	Running temp	40 °C		
	Length:	100 mm		
	Diameter:	3 mm		
	Particle Size	3.0 µm		
Nominal Tune Values	Collision Gas	10 psi		
	Curtain Gas	25 psi		
	Ion Source Gas 1	40 psi		
	Ion Source Gas 2	50 psi		
	IonSpray Voltage	-4500 v		
	Temperature	450 °C		
	ESI polarity	Negative		
	Decustering Potential	Optimized for each analyte (See Appendix C for reference)		
	Collision Energy			
	Collision Cell Exit Potential			

9.1.3 Routine Instrument Maintenance

Routine instrument maintenance is critical to achieve optimum method sensitivity. All laboratory materials must be determined to be free of contamination to ensure potential background interferences are minimized.


Please refer to the instrument manual for maintenance procedures performed by the lab.

All maintenance activities are listed in maintenance logs that are assigned to each separate instrument.

- **LC Maintenance** – LC system components, as well as the mobile phase constituents, contain many of the method analytes in this method. Thus, these PFAS will build up on the head of the LC column during mobile phase equilibration.
 - **Column Equilibrate** – To minimize the background PFAS peaks and to keep background levels constant, the time the LC column sits at initial conditions must be kept constant and as short as possible (while ensuring reproducible retention times).
 - **Column Flush** – In addition, prior to daily use, flush the column with 95% methanol for at least 15 min before initiating a sequence. It may be necessary on some systems to flush other LC components such as wash syringes, sample needles or any other system components before daily use.
- **MS Maintenance** – Please refer to the instrument manual for maintenance procedures performed by the lab. Common maintenance procedures are listed below
 - **Source Cleaning** – Clean the ion source parts which include curtain plate, orifice plate or skimmer, Q0, and etc. with reagent water and methanol. Tuning or

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optimizing the instrument followed the instrument manual. Refer to the Operating Instruction – Tune and Calibrate, the ion source operator guide, or the Analyst® software Help system

- **Pump Oil** – Check pump oil level and color periodically. Add or change pump oil when necessary, following the manual instruction.


9.1.4 Troubleshooting

Any deviations from the norm encountered while conducting this analysis must be noted and brought to the attention of the section supervisor. This section contains basic information for troubleshooting basic system issues. Certain activities may be carried out by the Agilent and AB SCIEX trained Qualified Maintenance Person (QMP) in the laboratory. For advanced troubleshooting, contact field service agents of the instruments.

- **LC Troubleshooting** – Please refer to the instrument manual for troubleshooting procedures performed by the lab. Common LC issues are listed below.
 - **Pressure Issue** – Large pressure variation could cause by the presence of air bubble in the system, blockage of the system, column contamination, system leaking, and etc. High pressure issue could be solved through system solvent purge, column rinse, clean or change of column inlet frit, injection valve, needle seat, and etc. Low pressure issue could usually be fixed by tighten or replace the capillary connection or other parts such as pump seals.
 - **Peak Shape Issue** – Split peaks, peak tailing, poor efficiency, and inconsistent response are usually associated with issues like column contamination, partially plugged frit, column void, injection solvent effects, or sample overload effects. Rinsing or changing the column, preparing fresh mobile solvent, reducing sample injection volume could usually
 - **Retention Time** – Deviation of retention time from originally values could cause by column aging or contamination, insufficient system equilibration, mobile phase variation, change in column temperature, or other instrument issues. Cleaning the HPLC system and column could Ammonium acetate is volatile and may cause the shift of retention time over certain period of time. Prepare fresh mobile phase solvent when necessary.
 - **Background Contamination** – After multiple injections or long period of operation, background interference may accumulate at the gradient proportional valve, needle seat, or other instrument parts. Rinsing and cleaning corresponding parts with reagent water, methanol or stronger solvents to remove the interference.
- **MS Troubleshooting** – Please refer to the instrument manual for troubleshooting procedures performed by the lab. Common MS issues are listed below:
 - **Sensitivity Loss** – The possible causes for intensity decrease could be contamination of TurboV ion spray, or the instrument requires tuning and optimization. Clean the ion source including curtain plate, orifice plate or skimmer, Q0. Tune or optimize the instrument following the instrument manual. Refer to the Operating Instruction – Tune and Calibrate, the ion source operator guide, or the Analyst® software Help system.

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- **Low Vacuum Pressure** – Low pump oil level could cause the vacuum pressure issue. Check the pump oil level and add oil if necessary.

9.2 Calibration

9.2.1 Calibration Frequency

See Appendix B.

9.2.2 Calibration Levels

Different volumes of PFAC_Native Spike solutions at various concentrations are added to 1 mL 537 Mix. See Table 1 below:

Table 1

Calibration Standard Point	Native std. Soln added (µL)	Native std. Soln conc. (µg/mL)	Extracted IS Soln added (µL)	Extracted IS Soln conc. (µg/mL)	Injection IS Soln added (µL)	Injection IS Soln conc. (µg/mL)
CS-1	10	0.05	100	0.05	100	0.05
CS-2	20	0.05	100	0.05	100	0.05
CS-3	40	0.05	100	0.05	100	0.05
CS-4	100	0.05	100	0.05	100	0.05
CS-5	200	0.05	100	0.05	100	0.05
CS-6	100	0.20	100	0.05	100	0.05
CS-7	250	0.20	100	0.05	100	0.05
ICV	100	0.05	100	0.05	100	0.05
T-PFOA (Qualitative Calibration)	100	0.05	100	0.05	100	0.05


A known amount of EIS is added into each calibration point. The corresponding concentration in 1 mL final solvent (concentrations of each analyte in 1 mL—final solvent in µg/L). See Table 2 below:

Table 2

Analyte	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6	CS-7	ICV
PFBA	0.50	1.00	2.00	5.00	10.00	20.00	50.00	5.00
PFPeA	0.50	1.00	2.00	5.00	10.00	20.00	50.00	5.00
PFHxA	0.50	1.00	2.00	5.00	10.00	20.00	50.00	5.00
PFHpA	0.50	1.00	2.00	5.00	10.00	20.00	50.00	5.00
PFOA	0.50	1.00	2.00	5.00	10.00	20.00	50.00	5.00
PFNA	0.50	1.00	2.00	5.00	10.00	20.00	50.00	5.00
PFDA	0.50	1.00	2.00	5.00	10.00	20.00	50.00	5.00
PFUdA	0.50	1.00	2.00	5.00	10.00	20.00	50.00	5.00
PFDoA	0.50	1.00	2.00	5.00	10.00	20.00	50.00	5.00
PFTTrDA	0.50	1.00	2.00	5.00	10.00	20.00	50.00	5.00
PFTeDA	0.50	1.00	2.00	5.00	10.00	20.00	50.00	5.00
PFOSA	0.50	1.00	2.00	5.00	10.00	20.00	50.00	5.00
N-EtFOSAA ¹	0.50	1.00	2.00	5.00	10.00	20.00	50.00	5.00
N-MeFOSAA ¹	0.50	1.00	2.00	5.00	10.00	20.00	50.00	5.00
PFBS	0.44	0.89	1.77	4.43	8.85	17.70	44.25	4.43

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Analyte	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6	CS-7	ICV
PFPeS	0.47	0.94	1.88	4.70	9.40	18.80	47.00	4.70
PFHxS ¹	0.46	0.91	1.82	4.55	9.10	18.20	45.50	4.55
PFHpS	0.48	0.95	1.90	4.75	9.50	19.00	47.50	4.75
PFOS ¹	0.47	0.93	1.86	4.65	9.30	18.60	46.50	4.65
PFNS	0.48	0.96	1.92	4.80	9.60	19.20	48.00	4.80
PFDS	0.48	0.97	1.93	4.83	9.65	19.30	48.25	4.83
4:2FTS	0.47	0.94	1.87	4.68	9.35	18.70	46.75	4.68
6:2FTS	0.48	0.95	1.90	4.75	9.50	19.00	47.50	4.75
8:2FTS	0.48	0.96	1.92	4.80	9.60	19.20	48.00	4.80
10:2FTS	0.48	0.97	1.93	4.83	9.65	19.30	48.25	4.83
HFPO-DA	0.50	1.00	2.00	5.00	10.00	20.00	50.00	5.00
DONA	0.47	0.95	1.89	4.73	9.45	18.90	47.25	4.73
N-MeFOSA	0.50	1.00	2.00	5.00	10.00	20.00	50.00	5.00
N-EtFOSA	0.50	1.00	2.00	5.00	10.00	20.00	50.00	5.00
N-MeFOSE	0.50	1.00	2.00	5.00	10.00	20.00	50.00	5.00
N-EtFOSE	0.50	1.00	2.00	5.00	10.00	20.00	50.00	5.00
9Cl-PF3ONS	0.47	0.93	1.86	4.66	9.32	18.64	46.60	4.66
11Cl-PF3OUdS	0.47	0.94	1.88	4.71	9.42	18.84	47.10	4.71
PFDoS	0.48	0.97	1.94	4.84	9.68	19.36	48.40	4.84
PFHxDA	0.50	1.00	2.00	5.00	10.00	20.00	50.00	5.00
PFODA	0.50	1.00	2.00	5.00	10.00	20.00	50.00	5.00

¹ Target analytes with isomer branch


- 1) Prepare a set of seven CAL standards (see Table 1 above for example). Analyze each standard level with the same acquisition method used to analyze samples, changes to retention times or other analytical parameters are saved as part of the local method generated with each analytical sequence, these parameters can be adjusted mid-sequence so long as they are applied to all data.
- 2) The LC/MS/MS system is calibrated using the isotope dilution and internal standard technique. Use the LC/MS/MS data system software to generate a linear regression calibration curve for each of the relevant analytes. This curve may be concentration weighted, if necessary.
- 3) A calibration meets criteria when the recovery for each calibration point reads back at $\pm 30\%$ for all calibration points.

NOTE: For Wisconsin samples, re-quantitated concentrations for all target compounds at all concentration levels must be within the range 70-130% of their actual concentrations, except for the lowest calibration concentration level, which must be within the range of 50-150% of actual concentration.
- 4) Provided a minimum of five calibration points are still being used, a point at the top or bottom of the calibration curve may be dropped to achieve recovery requirements across the remaining points. Dropping high concentration points lowers the PQL of the calibration and may require that more dilutions are performed. Dropping low calibration points can potentially elevate the RL for this sequence.

9.2.3 Calibration Sequence

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- 1) Tuning should occur at least once annually using PPGs for tuning. Tune the system when ICAL won't pass, when the peak shape is significantly off (indicating an MS problem), when major maintenance is performed, or instrument is moved.

NOTE: The instrument tuning with PPGs has its own manufacturing criteria- see the documentation. After PPGs passes, calibration must be verified to be +/- 0.5 amu of true values by acquiring a full scan continuum mass spectrum of a PFAS stock standard.

- (i) Load a 500 µL syringe filled with PPGs tuning solution in the syringe pump and connect it directly to the probe. Hold syringe pump power button for a few seconds to purge the line. Use the appropriate instrument control software (dependent on the manufacturer) to adjust the parameters under the Tune and Calibrate tab to a relative signal maxima for peaks 44.998, 585.385, 933.636, 1223.845, 1572.097, 1863.306, 2037.431, 2211.557 in negative mode and 59.050, 175.133, 616.464, 906.673, 1254.925, 1545.134, 2010.469, 2242.637 in positive mode.
- (ii) Mass assignment of tuning standard within 0.5 amu of true value.
- (iii) When done, run the Compound Optimization or Manual Tuning under the Tune and Calibrate tab to optimize response and peak shape.

- 2) Optimize the precursor ion and product ion for each target analyte by infusing a standard mix from calibration curve to MS. The MS parameters (voltages, temperatures, gas flows, etc.) and the MS/MS parameters (collision energy, declustering potential, collision cell exit potential, etc.) are determined to achieve optimal analyte responses.


NOTE: There have been reports that not all product ions in the linear PFOS are produced in all branched PFOS isomers. (This phenomenon may exist for many of the PFAs.) Thus, to reduce PFOS, PFBS and PFHxS bias, it is required that the precursor $m/z \rightarrow m/z$ 80 transition be used as the quantitation transition. Some MS/MS instruments may not be able to scan a product ion with such a wide mass difference from the precursor ion; therefore, if the MS/MS cannot measure the precursor $m/z \rightarrow m/z$ 80 transition they may not be used for this method if PFOS, PFBS, or PFHxS analysis is to be conducted.

- 3) Establish LC operating parameters that optimize resolution and peak shape.
- 4) Inject a mid-level CAL standard under optimized LC/MS/MS conditions to ensure that each target analyte is observed in its retention time window and that there are at least 10 scans across the peak for optimum precision.
- 5) Prepare at least five calibration point standards for linear fit (see Table 1 in section 9.2.2 for example). Analyze each standard level with the same acquisition method used to analyze samples, changes to retention times or other analytical parameters are saved as part of the local method generated with each analytical sequence, these parameters can be adjusted mid-sequence so long as they are applied to all data. Use the LC/MS/MS data system software to generate a linear regression curve for each of the relevant analytes. This curve may be concentration weighted, if necessary. Forcing zero is not allowed for this analysis.

Calibration points at the top or the bottom of the curve may be dropped to achieve

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recovery requirements across the remaining points provided the minimum number of calibration points are still being used based on the curve fit. Dropping high concentration points lowers the upper QL of the calibration and may require that more dilutions are performed. Dropping low calibration points may elevate the reporting limit for samples associated with this calibration. The RL must be met without exception.

- 6) Analyte quantification uses the isotope dilution technique for the analytes having commercially available isotopically labeled analogs. The internal standard technique is used when a labeled analog is not commercially available for the target analyte. For specific relationships in quantification refer to Appendix D.

9.2.4 Calibration Evaluation

9.2.4.1 Curve Fit

Average response factor: calculate the relative standard deviation (RSD) of the RF values of the initial calibration standards for each native compound. The RSD must be $\leq 20\%$ for all analytes.

For each calibration standard, reprocess the target (native) analyte against the chosen calibration function. The reprocessed recoveries are expected to be within $\pm 30\%$ of true value. For some data applications the lowest concentration standard reprocessed recoveries are expected to be within $\pm 50\%$ of true value.

The lowest concentration ICAL standard must be \leq reporting level (RL).

S/N Ratio: $\geq 10: 1$ for all quantification ions and S/N Ratio of $\geq 3:1$ for confirmation ions.

9.2.4.2 Relative Error (%RSE)

Percent error between the calculated and expected amounts of an analyte should be $\leq 30\%$ for all standards. For some data applications $\leq 50\%$ may be acceptable for the lowest calibration point.

9.2.4.3 Tune

9.2.4.4 Initial Calibration Verification (ICV)

Analyze an ICV sample from a source different from the source of the CAL standards with each new ICAL before sample analysis. If a second vendor is not available, then a different lot of the standard should be used. The ICV should be prepared and analyzed just like a CCV. Acceptance criteria for the ICV are identical to the CCV; the calculated amount for each analyte must be $\pm 30\%$ of the expected value. If measured analyte concentrations are not of acceptable accuracy, check the entire analytical procedure to locate and correct the problem.


9.2.4.5 Continuing Calibration Verification (CCV)

CCVs are run at the beginning, end, and bracketing every 10 field samples. Blanks, rinses, and spiked QC (LCS/LCSD/MS/MSD) are not considered field samples, and so can be run in addition to 10 field samples in a CCV window.

The opening CCV for any batch must be below or at the RL (CS-1), all further CCVs cycle between mid and high-level calibration point.

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Calculate the concentration of each analyte in the CCV. The calculated amount for each analyte must be within $\pm 30\%$ of the true value. Determine that the absolute areas of the quantitation ions of the EIS are within $\pm 50\%$ from the mid-point measured during initial calibration. On days when ICAL is not performed, the peak areas must be within $\pm 50\%$ of the peak area measured in daily initial CCV. If any of the EIS areas has changed by more than these amounts, adjustments must be made to restore the system.

NOTE: For Wisconsin samples, the calculated amount for each analyte must be within $\pm 30\%$ of the true value except for the lowest ICAL point, for which the calculated amount for each analyte must be within $\pm 50\%$ of the true value.

If the CCV fails high for a particular analyte, and the field sample shows no detection for that analyte, samples may be reported without re-analysis.

9.2.4.6 EIS Recovery

The EIS is fortified into all samples, CCVs, MBs, LCSs, MSs, MSDs, and FD prior to extraction. It is also added to the CAL standards. The EIS is a means of assessing method performance from extraction to final chromatographic measurement.

A minimal signal to noise ratio of 10:1 is expected for each EIS. Do not report results with a qualifier if this minimum is not achieved.

EIS recovery must be in $\pm 50\%$ of the mid-point ICAL when the day the ICAL was performed, except d3-MeFOSA, d5-EtFOSA, d7-MeFOSE, and d9-EtFOSE, which must recover within the range 10-150%. When EIS recovery from a sample, blank, or CCV failed the criteria, check 1) calculations to locate possible errors, 2) standard solutions for degradation, 3) contamination, and 4) instrument performance. Correct the problem and re-analyze the extract.

NOTE: For Wisconsin samples, all EIS compounds must recover within the range 25-150%, except 13C8-PFOA, d3-MeFOSA, d5-EtFOSA, d7-MeFOSE, and d9-EtFOSE, which must recover within the range 10-150%. Recovery will be based on area counts.


- If the EIS recoveries in a chromatographic run do not meet these criteria, inject a second aliquot of that extract from a new capped auto-sampler vial.
- If the reinjected aliquot produces an acceptable EIS recoveries, report results for that aliquot.
- If recoveries are acceptable for QC samples, but not field samples, the field samples must be re-prepped and reanalyzed (greater dilution may be needed). If recoveries are unacceptable for QC samples, correct problem, and reanalyze all associated failed field samples.

Apply qualifier and discuss in the Case Narrative only if reanalysis confirms failures in exactly the same manner.

- If the extract re-analysis meets the EIS recovery criterion, report only data for the re-analyzed extract.

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- If the extract re-analysis fails the criterion, the analyst should check the calibration by injecting the last CAL standard that passed. If the CAL standard fails the criteria, re-calibration is in order. If the CAL standard is acceptable, extraction of the sample should be repeated provided the sample is still within the holding time. If the re-extracted sample also fails the recovery criterion, report all data for that sample as suspect recovery to inform the data user that the results are suspect due to EIS recovery. Alternatively, collect a new sample and re-analyze.

9.3 Sample Preparation

9.3.1 Homogenization & Subsampling

See laboratory SOP ENV-SOP-MIN4-0172 *Sub-Sampling, Sample Homogenization, and Sample Composting* (current version or equivalent replacement) for additional sample homogenization and subsampling procedures.

9.3.2 Solid Samples

- 1) Homogenize the entire solid sample received in the sample container in which it was collected by stirring the solid with a clean spatula or other implement.
- 2) 5 g of solid sample is weighed in a tared 50-mL polypropylene bottle.

NOTE: Spiking solutions will be spiked directly onto solid samples and must be allowed to evaporate prior to the addition of any solution (at least 15 minutes).


- 3) Add a 100 µL aliquot of the PFAC_EIS to all field and QC samples at the very beginning of the procedure.
- 4) QC samples for each batch include a MB, LCS and MS/MSD which are extracted along with each prep batch.
 - (i) MB is required for each prep batch. Each batch contains a LCS and a pair of MS/MSD. LCS/MS/MSD spike at concentrations \geq LOQ and \leq the mid-level calibration concentration. If insufficient sample is available for a pair of MS/MSD, an MS, Dup, and LCSD at the same level of LCS may be used.

NOTE: WI samples must be spiked at 2x the MRL.

- (ii) The LCS/LCSD/MS/MSD is spiked with 20 µL of the PFAC_Native Spike 2.
- 5) 5 mL of 0.4% ammonia/methanol is added to all samples and QC, bottles are sealed and put on an ultrasonicator for 20 minutes and then shake for one hour.
 - 6) Centrifuge the samples and QC for 5 minutes after shake.
 - 7) Decant the supernatant layer in a 50-mL polypropylene bottle with 50 mg of ENVI-Carb powder.
 - 8) Repeat Centrifuge steps with 4 mL of 0.4% ammonia/methanol and centrifuge. All supernatant are collected and combined.
 - 9) The combined supernatant is shaken for one hour and then centrifuge for 5 minutes after shake.

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- 10) Clean the filter with 10 mL of 0.4% ammonia/methanol.
- 11) Condition the pre-cleaned filter with 10 mL methanol. Pass the combined supernatant through the filter. Rinse the filter with an additional 1 mL 0.4% ammonia/methanol. Collect the filtrate and turn on the vacuum for 10 minutes.

9.3.3 Aqueous Samples


- 1) Sample volume is determined gravimetrically. The full sample bottle is weighed, and the empty bottle is weighed after extraction. The sample volume is the difference between the full and empty bottle weights. Sample density is assumed at 1 g/mL. When the sample has significant solids, the laboratory should account for the weight or volume displaced by the solid in the initial sample volume determination and include this information in the report.
- 2) pH is taken using strips in the lab. This is accomplished via the use of common laboratory grade pH strips (Whatman Indicator Paper pH 0-14 Type CF Cat. No. 2613-991). Adjust the pH to ~5 with acetic acid or 10 mM sodium hydroxide solution when necessary.
- 3) Add a 100 µL aliquot of the PFAC_EIS to all field and QC samples at the very beginning of the procedure, before extraction, centrifuging, filtering, or phase separation takes place. Cap and invert and mix.
- 4) Ideally, whole samples will pass through the cartridge as received. If the percentage of particulates in the sample is greater than one percent, centrifuge the sample and take the liquid phase through the SPE after spiking the PFAC_EIS.

NOTE: For Wisconsin Samples, if particulates in the sample must be removed before using SPE, centrifuge the sample and take the liquid phase through the SPE. Samples should only be centrifuged when the suspended solids content visually appears to be high enough, by chemist inspection, that it would cause the SPE cartridge to clog

- If aqueous samples with a solid phase are centrifuged, the solid phase of the sample is expected to be a plug at the bottom of the container. It is expected that the solid phase remains in the container when rinsing the container walls with the polar elution solvent. Rinsing the container walls would therefore also include rinsing of the solids. If the polar elution solvent disrupts the solid phase significantly, the container can be centrifuged again before removing the solvent for use during the elution step of the SPE procedure.
- If a total sample concentration is needed and there are significant solids in the sample, the initial spike of EIS into the sample container is sufficient for both phases. There is no need to respire the solid phase with EIS if it is being extracted separately
- Using filters to separate the solid phase from the liquid phase is discouraged unless there is data to demonstrate that the filters used do not result in contamination greater than one-half the MRL.
- In the cases where a filter is used to separate the solid phase from the liquid phase, it is expected that the filter would also be rinsed to remove any potentially adsorbed PFAS. The filtrate is then added to the SPE cartridge during the elution step.

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- The data quality objectives from the data user should determine whether the solid phase of the sample has to be extracted or not. Not analyzing the solid phase may lead to a low bias in total sample concentration. Analyzing the liquid phase only would provide a liquid sample concentration result. It is expected that the laboratory would make it clear to the data user whether the reported concentrations are a total or liquid concentration sample result.
- 5) QC samples for each batch include a MB, LCS and MS/MSD which are extracted along with each prep batch.
 - MB is required for each prep batch. Method blanks must be rotated through each sample port on the SPE manifold.
 - Each batch contains a LCS and a pair of MS/MSD. LCS/MS/MSD spike at concentrations \geq LOQ and \leq the mid-level calibration concentration. If insufficient sample is available for a pair of MS/MSD, an MS, Dup, and LCSD at the same level of LCS may be used.
 - The LCS/LCSD/MS/MSD is spiked with 20 μ L of the PFAC_Native Spike 2.
 - 6) Proceed with SPE procedure in section 9.3.5.

9.3.4 Tissue Samples


NOTE: Sample homogenization for tissue samples are performed by Pace-Green Bay WI laboratory in accordance with laboratory SOP ENV-SOP-GBAY-0129 *Sample Homogenization, Compositing and Sub-Sampling* (current version or equivalent replacement).

- 1) 2 g of tissue sample is weighed in a tared 50-mL HDPE bottle.

NOTE: Spiking solutions will be spiked directly onto tissue samples and must be allowed to evaporate prior to the addition of any solution (at least 15 minutes).
- 2) Add a 100 μ L aliquot of the PFAC_EIS to all field and QC samples (canola oil and SRM) at the very beginning of the procedure.
- 3) QC samples for each batch include a MB, LCS, MS/MSD, and SRM which are extracted along with each prep batch.
 - (i) MB is required for each prep batch. Each batch contains a LCS and a pair of MS/MSD. LCS/MS/MSD spike at concentrations at concentrations \geq LOQ and \leq the mid-level calibration concentration. If insufficient sample is available for a pair of MS/MSD, an MS, Dup, and LCSD at the same level of LCS may be used.
 - (ii) The LCS/LCSD/MS/MSD is spiked with 40 μ L of the PFAC_Native Spike 2.
- 4) 7 mL of 0.6% ammonia/acetonitrile is added to all samples and QC, bottles are sealed and put on a shaker for 16 hours
- 5) Centrifuge the samples and QC for 5 minutes after shaking
- 6) Decant the supernatant layer in a 50-mL polypropylene bottle with 100 mg of ENVI-Carb powder and shake for 1 hour and then centrifuge for 5 minutes after shake

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- 7) Clean the 250 mg ENVI-Carb cartridge with 10 mL of 0.4% ammonia/methanol.
- 8) Condition the pre-cleaned cartridge with 10 mL methanol. Pass the supernatant through the cartridge. Rinse the filter with additional 0.4% ammonia/methanol.
- 9) Collect the filtrate and dilute the filtrate with 125 mL H₂O and adjust pH to ~5. Proceed with SPE procedure in 9.3.5.

9.3.5 Cartridge SPE Procedure

9.3.5.1 Cartridge Clean-Up

Rinse each cartridge with 10 mL of 0.4% ammonia/methanol solution.

9.3.5.2 Cartridge Conditioning

- 1) Condition each cartridge with 10 mL of 0.4% ammonia/methanol solution following with 10 mL of methanol.
- 2) Next, rinse each cartridge with 10 mL of reagent water following with 10 mL of acetate buffer, without allowing the water to drop below the top edge of the packing.

NOTE: If the cartridge goes dry during the conditioning phase, the conditioning must be started over.

- 3) Turn on the vacuum and begin adding the sample to the cartridge through the attached plastic sample transfer reservoir.

NOTE: Do NOT allow cartridge packing material to go dry during any of the conditioning steps.

9.3.5.3 Sample Extraction

- 1) Adjust the vacuum so that the approximate flow rate is 6-10 mL/min.

NOTE: DO NOT allow the cartridge to go dry before all the sample has passed through.

9.3.5.4 Sample Bottle and Cartridge Rinse

- 1) Rinse the sample bottles with two 5-mL aliquots of reagent water, then draw each aliquot through the plastic sample transfer reservoir and the cartridges.
- 2) Draw air through the cartridge for 25 min at high vacuum (10-15 in. Hg).


NOTE: If transfer tubes are used in place of the sample transfer tubes to pass the samples through the cartridges, these reservoirs must be treated like empty plastic sample transfer reservoirs. After the entire sample has passed through the cartridge, the tubes must be rinsed to waste with reagent water.

9.3.5.5 Sample Bottle and Cartridge Elution

- 1) Turn off and release the vacuum.
- 2) Lift the extraction manifold top and insert a rack with collection tubes into the extraction tank to collect the extracts as they are eluted from the cartridges.

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- 3) Use 3 mL of 0.4% ammonia/methanol for NPW and solid or 3 mL of 0.6% ammonia/acetonitrile for tissue twice to elute the analytes by pulling the two aliquots of organic solvent through the sample plastic reservoirs and the cartridges.
- 4) Rinse the sample bottle with an additional 3 mL of 0.4% ammonia/methanol or 3 mL of 0.6% ammonia/acetonitrile, pull the additional 3 mL organic solvent through the cartridge.
- 5) Turn the vacuum on for 20 minutes between each elution.

NOTE: The elution solvent must be swirled down the sides of the reservoirs while eluting the cartridge to ensure that any method analytes on the surface of the reservoirs are transferred to the extract.

NOTE: After centrifuging, it is expected that the solid phase remains in the bottom of the container when rinsing the container with elution solvent. If rinsing the container disrupts the solid phase significantly, the container can be centrifuged again before removing the solvent for use during the elution step.

9.3.6 Mechanical Sample Preparation (Cryomilling-Articles of Commerce)

9.3.6.1 Manual Cutting

If possible, manual cutting with shears or scissors is suitable for rough cutting and preparation of samples for further reduction by grinding, etc. Recommended maximum sample sizes is 10 x 10 mm, but will depend on the specification of the equipment used in the subsequent preparation processes:

9.3.6.2 Fine Grinding Cryogenic Milling

Some cryogenic mills are capable of handling materials < 10 mm and can perform both coarse grinding and fine grinding via use of interchangeable grinding wheels/balls and sieves. Fine grinding via cryogenic milling is suitable for further reducing samples to ~100 µm in diameter. Be careful not to allow the LN2 to come into direct contact with the sample to prevent spattering and sample loss.

Cryogenic milling will be described using a Retsch CryoMill (ball mill) with automatic LN2 feed, as described below:

Sample Type	Cycles	Frequency (Hz)	PCT ¹ (minutes)	MT ² (minutes)	ICT ³ (minutes)
Elastomers (FKM, FFKM, and FEPM) and Agglomerates)	1	30	Auto	2	2
Compacted Cake	5	30	Auto	2	2
Melt Extruded Pellets and Molded Articles	7	30	Auto	4	2

¹ PCT = Precooling time (automatic set)


² MT = Milling time

³ ICT = In-between cooling time

- 1) In the case of FFKM compounds and articles, care must be taken to avoid excessive energy input as it may lead to cryo-mechanical degradation of the polymer and the creation of mechanoradicals, which can inflate the amount of

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target substance detected. Filling levels will impact the energy applied in the grinding process and can inflate test results as well.

- 2) In the case of cured FFKM elastomeric articles, pulverizing the product in a 2-roll mill as commonly used for compounding of elastomers can be an alternate sample preparation method to avoid or reduce the risk of mechanical degradation. Alternative methods for FFKM compounds and articles that do not require cryo-grinding are being investigated.
- 3) Cryogenically mill the sample powder and sweep out the milled material to collect all the powder. The collected plastic-based sample material should be precision sieved to obtain a sufficiently homogeneous portion of known particle size range of 50 µm to 150 µm, by first sieving material through the 150 µm sieve, then the 50 µm sieve (keep contents captured on 50 µm sieve). Size range is 100 + 50 µm (i.e. ~ 100 µm).
- 4) The collected FKM-, FFKM- and FEPM-based samples are visually inspected for homogenous size, verification via light-scattering is beneficial. Sieving of the samples is not recommended due to materials' elastomeric nature and the potential for particles to re-agglomerate into larger segments over time.
- 5) Thorough cleaning of the mill components between samples is crucial to avoid cross-contamination of certain substances.

NOTE: Freshly cryomilled samples should either be extracted ASAP (same day) OR placed under frozen conditions (-10C) ASAP and held under frozen conditions until ready to extract.

- 6) Make sure to follow User Manual for installing and removing the grinding jar. This includes the two lock rings on left side of mechanism. Details follow.
 - (i) Make sure outer LOCK RINGS are loosened.
 - (ii) Screw in the grinding jar (from the right side of the mechanism). Tighten with black plastic tightening aid.
 - (iii) TIGHTEN TWO LOCK RINGS: Outside ring tightened first (hand-tighten only), inner ring tightened second (use tool; snug).
 - (iv) Perform grinding cycle.
 - (v) LOOSEN THOSE TWO LOCK RINGS: Inside ring loosened first, outer ring loosened second. To loosen either ring, the tool will be needed because of icing.
 - (vi) Unscrew grinding jar.
 - (vii) When cryomill is not in use, make sure to replace the cooling jacket seal.


9.3.6.3 Cleaning Procedure for Mill Components

Thorough cleaning of the mill components between samples is crucial to avoid cross-contamination of certain substances.

The following cleaning procedure is recommended:

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- 1) Remove residual polymer thoroughly with DI water and brush.
- 2) If necessary, wipe away last polymer residue with clean polish cloth or cotton ball/swab.
- 3) Rinse all parts with methanol, use an ultrasonic bath as needed.
- 4) Rinse all parts with DI Water, use an ultrasonic bath as needed.
- 5) Repeat steps 1-4 as needed.

9.3.7 Analysis Specifics for Cryomilled Samples

Freeze cryomilled sample until extraction.

Extraction:

- 1) 0.5 grams of cryomilled sample will be used for extraction.
- 2) Spike the sample with 100 µL EIS.
- 3) Add 4.5 mL of methanol to the spiked sample.
- 4) Shake 24 hours.
- 5) In cases where samples are fully or partially soluble in methanol, samples can be diluted 1:1 in HPLC grade water to crash the soluble polymer, avoiding precipitation and clogging the HPLC injector. After adding water, vortex mix the samples for 1 minute prior to proceeding to the next step .
- 6) Centrifuge sample for 10 minutes.
- 7) Transfer 8 mL of supernatant to clean tube.
- 8) Aliquot clarified supernatant to autosampler vial and cap for analysis.

9.3.8 Extract Concentration

- 1) Concentrate the extract to approximately 0.5 mL for water and solid extract and approximate 0.2 mL for tissue extract under a gentle stream of nitrogen without a heated water bath.
- 2) Add 100 µL of PFAC_IIS and fill the sample vial to 1 mL mark with 537 Mix.
- 3) Vortex for 5-10 seconds.
- 4) Transfer a ~100 µL to a 300 µL polypropylene autosampler vial with a plastic pipette.

NOTE: For Wisconsin sample, the remaining extract is stored at 0-6°C.


NOTE: For Wisconsin samples, thoroughly vortex autosampler vials including standards and sample vials before loading the autosampler to remove any PFAS that may have adsorbed to container wall.

9.4 Analysis

9.4.1 Preparation

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- 1) Sample bottles must be rinsed with the elution solvent. The bottle rinse is passed through the cartridge to elute the method analytes and is then collected.

NOTE: The SPE cartridges and sample bottles described in this section are designed as single use items and should be discarded after use. They may not be refurbished for reuse in subsequent analyses.

- 2) Establish operating conditions equivalent to those summarized in Appendix C.

NOTE: Standards and sample extracts should be vortexed before taking aliquots prior to injection.

- 3) Establish an appropriate retention time window for each analyte. This should be based on measurements of actual retention time variation for each method analyte in Calibration (CAL) standard solutions analyzed on the LC over the course of time. A value of plus or minus three times the standard deviation of the retention time obtained for each method analyte while establishing the initial calibration and completing the IDC can be used to calculate a suggested window size. However, the experience of the analyst should weigh heavily on the determination of the appropriate retention window size.

- 4) **Retention Time (RT) Acceptance** – RT of each analyte and EIS analyte must fall within 0.4 minutes (± 0.2 minutes) of the predicted retention times from the daily CCV or, on days when ICAL is performed, from the midpoint standard of the ICAL. Analytes must elute within 0.1 minute of the associated EIS. This criterion applies only to analyte and labeled analog pairs.

- 5) Calibrate the system by either the analysis of a calibration curve or by confirming the initial calibration is still valid by analyzing a CCV. If establishing an initial calibration, complete the IDC.

- 6) Begin analyzing field samples, including QC samples, at their appropriate frequency by injecting 3 μ L of final sample extractant, under the same conditions used to analyze the ICAL standards.

- 7) At the conclusion of data acquisition, use the same software that was used in the calibration procedure to identify peaks of interest in predetermined retention time windows. Use the data system software to examine the ion abundances of the peaks in the chromatogram. Identify an analyte by comparison of its retention time with that of the corresponding method analyte peak in a reference standard. Comparison of the MS/MS mass spectra is not particularly useful given the limited ± 0.5 amu mass range around a single product ion for each method analyte.


- 8) **Dilution** – When the concentrations of target analytes exceed the highest concentration of ICAL, dilution analyses are required.

An appropriate dilution should be in the upper half of the calibration range, or close to the CCV. The diluted extract must maintain the same methanol/water ratio as the original extract.

NOTE: If an analyte concentration exceeds the range of the initial calibration curve, the extract is diluted with 537 Mix. Re-inject the diluted extract. Incorporate the dilution factor into the final concentration calculations. The resulting data is documented as a

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dilution, with an increased LOQ.

- 9) In validating this method, concentrations were calculated by measuring the product ions listed in Appendix C. Two transitions and the ion transition ratio per analyte shall be monitored and documented with the exception of PFBA and PFPeA. In order to avoid biasing results high due to known interferences for some transitions, the following transitions must be used for the quantification of the following analytes: PFOA: 413 → 369, PFOS: 499 → 80, PFHxS: 399 → 80, PFBS: 299 → 80, 4:2FTS: 327 → 307, 6:2FTS: 427 → 407 8:2FTS: 527 → 507, N-EtFOSAA: 584 → 419, N-MeFOSAA: 570 → 419. If these transitions are not used, the reason must be technically justified and documented (e.g., alternate transition was used due to observed interferences).
- 10) Calculate analyte concentrations using multipoint calibration. Do not use daily calibration verification data to quantitate analytes in samples. Adjust final analyte concentrations to reflect the actual sample volume.
- 11) Prior to reporting the data, the chromatogram is reviewed for any incorrect peak identification or poor integration. Modify if necessary.
- 12) Calculations must utilize all available digits of precision, but final reported concentrations are rounded to an appropriate number of significant figures (one digit of uncertainty), typically two, and not more than three significant figures.
- 13) For native analytes, the Signal to Noise (S/N) ratio should be $\geq 10:1$ for quantitation and $\geq 3:1$ for confirmation ions. If S/N is not achieved, the analyte would be reported as not detected.
- 14) **Ion Ratios** – For analytes with two ion transitions (quantitation and confirmation) are analyzed, the area ratio between the confirmation and quantitation transitions shall be monitored and documented. The ion ratio for all analytes in each injection should be within $\pm 50\%$ of the mid ICAL ion ratio for the same analyte in the ICAL. On days ICAL is not performed, the ion ratio should be within $\pm 50\%$ of the initial CCV standard.

NOTE: Samples not meeting minimum ion ratio criteria must be qualified.

To calculate the ion ratio, see section 10.2.6.


- 15) Report results in the acid form.
- 16) Perform a moisture analysis on solid samples (on a subsample different than that used for extraction) and adjust the final concentration of solid sample for the percent moisture.

NOTE: A LCS is required with each extraction batch. See DoD acceptance criteria for LCS targets in Appendix E for aqueous and solid matrices. If the LCS results do not meet the criteria listed in Appendix E for all analytes, then all data for the problem analyte(s) must be considered invalid for all samples in the extraction batch. For target analytes not included in the DoD Limits for batch control table in Table B-15 per DoD QSM 5.3, limits of 70-140% recovery for water and soil, 60-140% recovery for tissue will be used as acceptance criteria.

NOTE: For Wisconsin samples, the recoveries are expected to be within 60-135%, except for the low range (1-2x RL) where the recoveries are expected to be within 50-150% in

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aqueous and solid batches. For tissue batches, spike the LCS at midrange. For tissue batches, the recoveries are expected to be within 60-135% with the following exceptions: for PFHxDA, PFODA, and NMeFOSA, the recoveries are expected to be within 50-135%; for PFDS, PFDoS, and 4:2 FTS, the recoveries are expected to be within 40-135%.

9.4.2 Example Analytical Sequence

ICB
ICAL
ICB
ICV
CCV
CCB
T-PFOA (Qualitative Standard for PFOA)
MB
LCS/LCSD
MS/MSD
Field Samples
CCV
CCB
Field Samples
CCV
CCB

10.0 DATA ANALYSIS & CALCULATIONS

10.1 Qualitative Identification

Complete chromatographic resolution is not necessary for accurate and precise measurements of analyte concentrations using MS/MS. In validating this method, concentrations were calculated by measuring the product ions listed in Appendix C. Other ions may be selected at the discretion of the analyst.


Prior to reporting the data, the chromatogram should be reviewed for any incorrect peak identification or poor integration.

- For PFHxS, PFOS, N-MeFOSAA and N-EtFOSAA, all the chromatographic peaks observed in the standard must be integrated and the areas summed. Chromatographic peaks in all Field Samples and QC samples must be integrated in the same way as the CAL standard for analytes with quantitative standards containing the branched and linear isomers.
- For PFOA, identify the branched isomers by analyzing a qualitative standard (T-PFOA) that includes both linear and branched isomers and compare retention times and tandem mass spectrometry transitions. Quantitate Field Samples and QC samples by integrating the total response (i.e., accounting for peaks that are identified as linear and branched isomers) and relying on the initial calibration with a linear isomer quantitative PFOA standard. This qualitative PFOA standard is not used for quantitation. This branched isomer identification check must be repeated any time changes occur that affect the analyte retention times.

Peaks that are consistent with branched isomers have been observed with other target analytes, in particular PFOA. Quantitate of PFOA by integrating the total response (i.e. accounting for peaks that are identified as linear and branched isomers) and relying on the initial calibration

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with the linear-isomer quantitative standard is acceptable.

All analytes are quantified using the isotope dilution or internal standard technique.

The native analytes are quantified by comparison of their responses to the mass-labelled internal standards. Relative response factors are calculated from analyses of standard mixtures containing native analytes at six concentration levels, and the concentration remains at a constant level for each internal standard. The target analytes response factors are calculated by comparing the response from the native ion mass monitored to the response from the ion mass of the corresponding isotopically labelled internal standard (See Appendix D for reference).

10.1.1 Tentatively Identified Compounds (TICs)

Not applicable to this SOP.

10.1.2 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 justified and proper all manual integrations must be performed, documented, reviewed, and approved in accordance with corporate SOP ENV-SOP-CORQ-0006 *Manual Integration*. Refer to this SOP for guidance on manual integration techniques and required procedures.

10.2 Calculations

10.2.1 Linear Calibration Using Average Response Factors

For each target analyte, calculate the response factor of each calibration level as follows:

$$RF_i = \frac{(A_a Q_s)}{A_s Q_a}$$

Where: RF_i = Response factor

A_a = Sum of integrated areas for analyte

Q_s = Quantity of labeled standard


A_s = Sum of integrated areas for labeled standard

Q_a = Quantity of analyte

10.2.2 Quantifying Native Analyte Levels

The levels of native analytes in the samples are quantified using the following equations:

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$$C = \frac{(A_n Q_{is})}{A_{is} \times W \times RF}$$

Where: RF = Response factor

A_n = Sum of integrated areas for target isomer

Q_{is} = Quantity of labeled internal standard added to the sample

A_{is} = Sum of integrated areas for labeled internal standard

W = Sample amount

C = Concentration of target isomer

$$\overline{RF} = \left(\frac{\sum_{i=1}^n RF_i}{n} \right)$$

Where: \overline{RF} = Average response factor

n = Number of calibration levels

RF_i = Response factor for the ith level

10.2.3 Relative Standard Deviation (%RSD)

$$\%RSD = \frac{SD}{\overline{RF}} \times 100\%$$

Where: \overline{RF} = Average response factor


SD = Standard deviation

Where SD is the standard deviation of the average RF, which is calculated as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^N (RF_i - \overline{RF})^2}{N - 1}}$$

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Where: \overline{RF} = Average response factor
 RF_i = Response factor
 N = Number of calibration levels

10.2.4 Weight Correction for Salt Content

$$Mass_{acid} = Measured\ Mass_{salt} \times \frac{MW_{acid}}{MW_{salt}}$$

Where: MW_{acid} = the molecular weight of PFAS
 MW_{salt} = the molecular weight of purchased salt

10.2.5 Ion Ratio Calculation

$$Ion\ Ratio = \frac{I_c}{I_q}$$

Where: I_c = Confirmation ion abundance
 I_q = Ion abundance quantitation

10.2.6 Reporting Wisconsin Samples

For Wisconsin samples, report sample results and all quality control blank results to the MDL and include the RL for each result reported. See example below:


	Laboratory	Report Result as
MDL	0.6	0.6
MRL	2.0	2.0
Sample Result	0.4	< 0.6

Quality results reported between the MDL and RL are estimated concentrations and reported with a J-flag. See example below:

	Laboratory	Report Result as
MDL	0.6	0.6
MRL	2.0	2.0

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	Laboratory	Report Result as
Sample Result	0.8	0.8 J

11.0 QUALITY CONTROL & METHOD PERFORMANCE

11.1 Quality Control

Prepare the following QC samples with each batch of samples. Refer to Appendix B for acceptance criteria and required corrective action(s).

QC Check	Acronym	Frequency
Method Blank	MB	1 per batch of 20 or fewer samples. If batch exceeds 20 samples, every 20 samples.
Laboratory Control Sample	LCS	1 per batch of 20 or fewer samples. If batch exceeds 20 samples, every 20 samples.
LCS Duplicate	LCSD	As Required.
Matrix Spike	MS	1 per batch of 20 or fewer samples. If batch exceeds 20 samples, every 20 samples.
Matrix Spike Duplicate	MSD	1 per batch of 20 or fewer samples. If batch exceeds 20 samples, every 20 samples.
Laboratory Field Duplicate	LFD	1 per batch of 20 or fewer samples. If batch exceeds, 20 samples, every 20.
Field Blank	TB	As Required.
Surrogate	SSTD	All samples and QC
Internal Standards	ISTD	All samples and QC
Standard Reference Material	SRM	1 per batch of 20 or fewer tissue samples. If batch exceeds, 20 samples, every 20.


11.2 Instrument QC

Perform the following checks to verify instrument performance. Refer to Appendix B for acceptance criteria and required corrective action.

Instrument Check	Acronym	Frequency
Tune (MS Only)		Every 6 months or when ICAL won't pass, the peak shape is significantly off, major maintenance is performed, or instrument is moved.
Initial Calibration	ICAL	At instrument set up, after major maintenance, after consecutive CCV failure, as otherwise indicated
Initial Calibration Verification	ICV	Once per calibration at mid-level of ICAL
Initial Calibration Blank	ICB	One following the highest standard analyzed and prior to ICV
Continuing Calibration Verification	CCV	At the beginning, end, and bracketing every 10 field samples
Continuing Calibration Blank	CCB	One following the highest standard analyzed and prior to ICV
Retention Time Windows	RTW	RT of each analyte and EIS analyte must fall within 0.4 minutes (± 0.2 minutes) of the predicted retention times from the daily CCV or, on days when ICAL is performed, from the midpoint standard of the ICAL.
Relative Retention Time	RRT	Analytes must elute within 0.1 minute of the associated EIS. This criterion applies only to analyte and labeled analog pairs

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11.3 Method Performance

11.3.1 Method Validation

Refer to corporate SOP ENV-SOP-CORQ-0011 for general requirements and procedures for method validation.

Establish detection limits (DL) and limits of quantitation (LOQ) at initial method set up and verify the DL and LOQ on an on-going basis thereafter. Refer to corporate policy and/or SOP for DL and LOQ requirements and procedures.

11.3.2 DoD Samples

For target analytes not included in the DoD Limits for batch control Table B-15 per DoD QSM 5.3, laboratory defined limits of recovery will be used as acceptance criteria. For tissue batches, the recoveries are expected to be within 60-140%. The RSD must be less than 30%. If any target analyte fails to meet this criterion, the source of the problem must be corrected, and the test repeated.

11.3.3 Wisconsin Samples

For Wisconsin samples, the mean accuracy is true value $\pm 35\%$. The RSD must be less than or equal to 30%. If any target analyte fails to meet this criterion, the source of the problem must be corrected, and the test repeated.

12.0 DATA REVIEW & CORRECTIVE ACTION

12.1 Data Review

The data review process of Pace® Analytical Services includes a series of checks performed at different stages of the process by different people to ensure that SOPs were followed, the analytical record is complete, and properly documented, QC criteria were met, proper corrective actions were taken for QC failure and other nonconformance(s), and test results are reported with proper qualification, when necessary.

The review and checks that are performed by the employee performing the task is called primary review.

All data and test results are also peer reviewed.


This process, known as 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, and approved in accordance with the Pace® Analytical Services SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

Lastly, a third-level review, called a completeness check, is performed by reporting or project management staff to verify the test report is complete.

Refer to laboratory SOP ENV-SOP-MIN4-0092 *Data Review Process* (current version or equivalent replacement) for specific instructions and requirements for each step of the data review process.

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12.2 Corrective Action

Corrective action is required when QC or sample results are not within acceptance criteria.

Refer to Appendix B for a complete summary of QC, acceptance criteria, and recommended corrective actions for QC associated with this test method.

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 & WASTE MANAGEMENT

Pace® proactively seeks ways to minimize waste generated during 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 practices comply with all applicable federal and state laws and regulations. Excess reagents, samples, and method process wastes are characterized and disposed of in an acceptable manner in accordance with the Pace® Chemical Hygiene Plan / Safety Manual. Refer to this manual for these procedures.


14.0 MODIFICATIONS

The procedures in this SOP have been modified from the reference test method as follows:

Modification	Test Method Procedure	Justification for Modification
Method EPA 537.1 was validated for drinking water matrix. Laboratory modified for non-potable matrices.	EPA 537.1	Isotope dilution is used for quantification. 537.1, Version 2.0 is only applicable to drinking water analysis and has been modified to include NPW, Solids and Tissue
MPCA recommends LCS in triplicate at 3 levels per analytical batch (low, medium, high). LCS must contain all project specific PFAS analytes in the same media as associated samples. The recovery acceptance for each	MPCA Guidance PFAS	Modifications to QC criteria

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Modification	Test Method Procedure	Justification for Modification
<p>method analyte is 100 + 30% and the percent relative standard deviation (RSD) of the recoveries < 30%.</p> <p>Laboratory process is 1 spike at greater than LOQ and less than the mid-level calibration 70-130% recovery, 30% RPD.</p>		
<p>MPCA recommends two MBs per batch of field samples, not to exceed 20 field samples. Same media as associated field samples and undergoes same sample prep. Each analyte must be < 1/3 the method reporting limit.</p> <p>Laboratory process is 1 MB rep 1/3 RL for Method blanks</p>	MPCA Guidance PFAS	Modifications to QC criteria
<p>When isotope dilution samples require a dilution, the volume of the diluent contains the same concentration of labeled isotope compounds as what was originally spiked into the sample. The isotope recovery results from the initial analysis should not be used to adjust the data from the secondary dilution analysis.</p> <p>Lab performs dilution If an analyte concentration exceeds the range of the initial calibration curve. The extract is diluted with 537 Mix. Re-inject the diluted extract. Incorporate the dilution factor into the final concentration calculations. The resulting data is documented as a dilution, with an increased LOQ</p>	MPCA Guidance PFAS	Modifications to dilution method.


When applicable, comparability and/or equivalency studies necessary to validate the modification as required per corporate SOP ENV-SOP-CORQ-0011 are retained by local quality personnel for historical reference.

15.0 RESPONSIBILITIES

- All employees of Pace® Analytical Services that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement (R&A) in their training file for the version(s) of the SOP that were in effect during the time the employee performed the activity.
- Local quality personnel are responsible for tracking the currency of the R&A on this SOP for employees at the locations they are assigned to and for notifying the General Manager (GM), however named, when R&A are overdue or outstanding. The GM and the employee's direct supervisor are responsible for ensuring the employee completes the R&A assignments as required.
- The supervisors and managers of Pace® Analytical Services, however named, are responsible for training employees on the procedures in this SOP, implementing the SOP in the work area, and monitoring on-going adherence to the SOP the work area(s) they oversee.

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- All employees of Pace® Analytical Services are responsible for following the procedures in this SOP. Unauthorized deviations or departures from this SOP are not allowed except with documented approval from the local Quality Manager and only when those deviations do not violate the Pace® Code of Ethics or Professional Conduct (COR-POL-0004) or associated policy and procedure(s). Hand-edits or manual change to the SOP are not permitted. If a change is desired or necessary, Pace® employees must follow the procedures for document revision specified in corporate SOPs ENV-SOP-CORQ-0015 *Document Management* and ENV-SOP-CORQ-0016 *SOP for Creation of SOP and SWI*.
- Local quality personnel are responsible for monitoring conformity to this SOP during routine internal audits of work areas that utilize this SOP and for communicating gaps and deviations found during monitoring to the work area supervisor, who is responsible for correction of the situation.

16.0 ATTACHMENTS


- Appendix A: Routine Analyte List and LOQ
- Appendix B: QC Summary & Corrective Action Table
- Appendix C: Typical MS/MS Method Conditions
- Appendix D: PFAS Analyte and Recommended Extracted Internal Standard Used for Quantification
- Appendix E: PFAS by LCMSMS Compliant with DoD QSM Batch Control Limits
- Appendix F: DoD QSM 5.3, Appendix B, Table B-15 - Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

17.0 REFERENCES

- ENV-SOP-CORQ-0006, *Manual Integration*, current version.
- ENV-SOP-CORQ-0011, *Method Validation*, current version.
- ENV-SOP-CORQ-0015, *Document Management*, current version.
- ENV-SOP-CORQ-0016, *SOP for SOP and SWI*, current version.
- ENV-TMP-CORQ-0007, *Quality Manual Template*, current version.
- COR-POL-0004, *Code of Ethics and Professional Conduct*, current version.
- COR-MAN-001, *Pace® Safety Manual*, current version.
- Laboratory Quality Manual, ENV-MAN-MIN4-0001, current version.
- TNI Standard, *Management and Technical Requirements for Laboratories Performing Environmental Analyses*, EL-V1-2016-Rev.2.1.
- Department of Defense Department of Energy Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.3, Appendix B, Table B-15, June 2019.
- Wisconsin Department of Natural Resources, Wisconsin PFAS Aqueous (Non-Potable Water) and Non-Aqueous Matrices Method Expectations, Document number EA-19-0001, December 2019

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- DoD Guidance for PFAS Analysis in Biota. April 2020.
- USEPA, Method 537.1, Version 2.0 “Determination of selected per- and polyfluorinated alkyl substances in drinking water by solid phase extraction and liquid chromatography/tandem mass spectrometry (LC/MS/MS)”; March 2020.
- USEPA, Technical Advisory, “Technical Advisory - Laboratory Analysis of Drinking Water Samples for Perfluorooctanoic Acid (PFOA) Using EPA Method 537 Rev. 1.1”; September 2016.
- JT Baker, Application Technical Support Group, Endothall extraction using BAKERBOND Speedisk SAX, PN-8058-06, 2006
- USEPA, “Manual for the Certification of Laboratories Analyzing Drinking Water”; Fifth Edition, January 2005.
- USEPA, “Supplement 1to the Fifth Edition of the Manual for the Certification of Laboratories Analyzing Drinking Water”; June 2008.
- 40 CFR Appendix B to Part 136, *Definition and Procedure for the Determination of the Method Detection Limit - Rev 2*, August 28, 2017.
- Minnesota Pollution Control Agency, *Guidance for Per- and Polyfluoroalkyl Substances: Analytical*, Document number p-eao2-28, October 2022.


18.0 REVISION HISTORY

Revisions Made from Prior Version

Section	Description of Change
ALL	Converted to new Corp test method SOP template.
Title	Updated from “Selected 36 Per- and Polyfluoroalkyl Substances (PFAS) by LC/MS/MS” to “Determination of Per- and Polyfluoroalkyl Substances (PFAS) by LC/MS/MS”
1.0	Updated to include MPCA Guidance and 537.1.
1.2	Added matrix, articles of commerce.
2.0	Deleted “and spiked with Injection Internal Standards (IIS)” in paragraph 3.
3.0	Deleted “IIS EIS” from the note section in bullet 1.
6.0	Added matrix, Articles of Commerce.
61	Deleted “Sample Collection” section.
6.2	Deleted “Sample Shipment” section.
6.3	Deleted “Sample Receipt and Storage” section
7.1	Deleted “Brand names and catalogue...may be substituted”; Updated the description for equipment, MS; Added equipment, MassHunter Data Acquisition; Added equipment, MassHunter Quantitative Analysis; and Added Horizon LIMS.
8.2	Deleted calculation in paragraph 3 and moved to section 10.2.4; and Updated standards table.
8.3.3	Deleted formulation for “PFAC_IIS – PFAC (Injection Internal Standards) (0.05 µg/mL)”; Shifted following sections up.
9.2.2	Added definition for footnote in Table 2.
9.2.3	Replaced “at least every six months” with “at least once annually”.
9.2.4.1	Replaced linear regression information with average response factor.

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Section	Description of Change
9.2.4.4	Updated entire section.
9.2.4.5	Deleted references to IIS.
9.2.4.7	Deleted "IIS Recovery" section.
9.3.4	Updated note for sample homogenization of tissue matrix by Pace-GB.
9.3.6	New section.
9.3.7	New section.
9.4.1	Step 1: Added new step; Step 8: Deleted "Acceptable injection internal standard (IIS) performance is determined from the undiluted sample extract" from the note section; Step 14: Deleted and moved ion ratio calculation to section 10.2.5; and Step 16: Updated "DoD acceptance...in Appendix E" to "A LCS is required...and soild matrices".
10.2.4	Deleted linear regression equation; and Shifted following sections up.
14.0	Added method modifications for variances between MPCA Guidance and laboratory method.
17.0	Updated citation for method 537.1 from version 1.0 to 2.0; and Added citation for MPCA Guidance.
Appendix B	Deleted QC item, Injection Internal Standard (IIS); Deleted linear regression from WIDNR Guidance for QC item, ICAL; Updated the WIDNR acceptance criteria for QC item, MS/MSD; and Added acceptance criteria column for MPCA Guidance.

Document Succession: This version replaces the following documents:

Document Number & Version	Document Title	Effective Date:
ENV-SOP-MIN4-0178 v03	Selected 36 Per- and Polyfluoroalkyl Substances (PFAS) by LC/MS/MS	12/13/2021

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Appendix A: Target Analyte List and LOQ

Table 1: Standard Analyte List and LOQ

Analyte	Acronym(s) ³	CAS #	LOQ ¹ Water (ng/L)	LOQ ¹ Solid (ng/kg)	LOQ ¹ Tissue (ng/kg)
Perfluorobutanoic acid	PFBA	375-22-4	2	100	250
Perfluoropentanoic acid	PFPeA	2706-90-3	2	100	250
Hexafluoropropylene oxide dimer acid	HFPO-DA ² PFPrOPrA	13252-13-6	2	100	NA
Perfluorohexanoic acid	PFHxA	307-24-4	2	100	250
Perfluoroheptanoic acid	PFHpA	375-85-9	2	100	250
Perfluorooctanoic acid	PFOA	335-67-1	2	100	250
Perfluorononanoic acid	PFNA	375-95-1	2	100	250
Perfluorooctanesulfonamide	PFOSAm PFOSA FOSA	754-91-6	2	100	250
N-methylperfluorooctane sulfonamide	MeFOSA ² N-MeFOSA NMeFOSA	31506-32-8	2	100	250
Perfluorodecanoic acid	PFDA	335-76-2	2	100	250
N-ethylperfluorooctane sulfonamide	EtFOSAm ² N-EtFOSA NEtFOSA	4151-50-2	2	100	250
Perfluoroundecanoic acid	PFUnDA PFUnA PFUdA	2058-94-8	2	100	250
N-methyl perfluorooctanesulfonamidoacetic acid	NMeFOSAA N-MeFOSAA	2355-31-9	2	100	250
N-ethyl perfluorooctanesulfonamidoacetic acid	NEtFOSAA N-EtFOSAA	2991-50-6	2	100	250
Perfluorododecanoic acid	PFDOA PFDoA PFDoDA	307-55-1	2	100	250
N-methylperfluorooctane sulfonamidoethanol	MeFOSE ² N-MeFOSE NMeFOSE	24448-09-7	2	100	250
N-ethylperfluorooctane sulfonamidoethanol	EtFOSE ² N-EtFOSE NEtFOSE	1691-99-2	2	100	250
Perfluorotridecanoic acid	PFTTrDA PFTTriA PFTTrA	72629-94-8	2	100	250
Perfluorotetradecanoic acid	PFTDA PFTeDA PFTA PFTeA	376-06-7	2	100	250
Perfluorohexadecanoic acid	PFHxDA ²	67905-19-5	2	100	250
Perfluorooctadecanoic acid	PFODA ²	16517-11-6	2	100	250
Perfluorobutanesulfonic acid	PFBS	375-73-5	1.77	88.5	221.3
Perfluoropentanesulfonic acid	PFPeS	2706-91-4	1.88	94	235
Perfluorohexanesulfonic acid	PFHxS	355-46-4	1.82	91	227.5

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Analyte	Acronym(s) ³	CAS #	LOQ ¹ Water (ng/L)	LOQ ¹ Solid (ng/kg)	LOQ ¹ Tissue (ng/kg)
Perfluoroheptanesulfonic acid	PFHpS	375-92-8	1.90	95	237.5
Perfluorooctanesulfonic acid	PFOS	1763-23-1	1.85	92.5	231.3
Perfluorononanesulfonic acid	PFNS	68259-12-1	1.92	96	240
Perfluorodecanesulfonic acid	PFDS	335-77-3	1.93	96.5	241.3
Perfluorododecanesulfonic acid	PFDoS ² PFDoDS	79780-39-5	1.94	97	242.5
1H, 1H, 2H, 2H-Perfluorohexanesulfonic acid	4:2 FTS 4:2 FTSA 4:2FTS	757124-72-4	1.87	93.5	233.8
1H, 1H, 2H, 2H-Perfluorooctanesulfonic acid	6:2 FTS 6:2 FTSA 6:2FTS	27619-97-2	1.90	95	480
1H, 1H, 2H, 2H-Perfluorodecanesulfonic acid	8:2 FTS 8:2 FTSA 8:2FTS	39108-34-4	1.93	96.5	241.3
1H, 1H, 2H, 2H-perfluorododecane sulfonic acid	10:2 FTS ² 10:2 FTSA	120226-60-0	1.93	96.5	241.3
4,8-Dioxa-3H-perfluorononanoic acid	DONA ² ADONA	919005-14-4	1.89	94.5	236.3
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9Cl-PF3ONS ² F-53B Major	756426-58-1	1.86	93	232.5
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS ² F-53B Minor	763051-92-9	1.88	94	235


¹ Values as of effective date of this SOP. LOQ are subject to change, contact quality personnel for most current information.

² DoD currently does not have guidance for the analyte in Table B-15 per DoD QSM 5.3, Appendix B, as of June 2019.

³ All possible acronym variations are listed as the acronym used and/or referenced may vary depending on the State data is being reported to.

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Appendix B: QC Summary and Corrective Action Table

QC Item	Frequency	Acceptance Criteria			Corrective Action	Qualification
		WIDNR Guidance	Table B-15 per DoD QSM v5.3	MPCA Guidance		
ICAL	At instrument set up, after CCV failure	Lowest ICAL \pm 50% Other points \pm 30% RSD \leq 20% For any curve fit other than Average RF (RSD), curve must also pass RSE test at the low and midpoint calibration standard.	All points \pm 30% of true value. RSD \leq 20% For any curve fit other than Average RF (RSD), curve must also pass RSE test at the low and midpoint calibration standard.	Lowest ICAL \pm 50% Other points \pm 30% The calibration curve should contain six, but preferably 8-10, nonzero calibration standards containing a consistent amount of stable isotope internal standards.	Identify and correct source of problem, repeat	None. Do not proceed with analysis
Curve Refitting	Whenever ICAL is performed	Must meet one of curve fit options presented in Section 9.2.4.1.	Must meet one of curve fit options presented in Section 9.2.4.1.	Select the simplest curve fit possible	Identify and correct source of problem, repeat	None. Do not proceed with analysis
ICV	1 after each ICAL	True value \pm 30%	True value \pm 30%	N/A	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.
RT Window Position (Daily)	Once per ICAL and at the beginning of the analytical window.	Position is set using the mid-point of the ICAL on the day ICAL is performed; otherwise mid-point of CCV is used	Position is set using the mid-point of the ICAL on the day ICAL is performed; otherwise mid-point of CCV is used	N/A	NA	N/A
RT Window Study	At method set-up and after major instrument maintenance	Window is \pm 0.2 minutes the daily CCV or, on days when ICAL is performed, from the midpoint standard of the ICAL. Analytes must elute within 0.1 minute of the associated EIS	Window is \pm 0.2 minutes the daily CCV or, on days when ICAL is performed, from the midpoint standard of the ICAL. Analytes must elute within 0.1 minute of the associated EIS	N/A	Correct problem and reanalyze samples	N/A
Continuing Calibration Curve (CCV)	Daily, before sample analysis, after every 10 field samples, and at end of analytical window.	CCV at low level: True value \pm 50% Other CCV: True value \pm 30%	True value \pm 30%	run prior to sample analysis, after every 10 field samples, and after the analytical sequence CCV at low level: True value \pm 50% Other CCV: True value \pm 30%	Perform necessary maintenance and demonstrate stability by analyzing an initial calibration before resuming sample analysis. Generally, samples between passing CCV and failing CCV should be re-analyzed.	Qualify analytes with CCV out of criteria.
Extracted Internal Standards (EIS)	Every field sample, standard and QC sample	For QC, 50-150% of the mid-point ICAL when the day the ICAL was performed. \pm 50% of the peak area measured in daily initial CCV. 20-150% for 13C8-PFOSA, d3-MeFOSA, d5-EtFOSA, d7-MeFOSE, and d9-EtFOSE For sample, 25-150% of the mid-point ICAL	\pm 50% of the mid-point ICAL when the day the ICAL was performed. \pm 50% of the peak area measured in daily initial CCV 10-150% for d3-MeFOSA, d5-EtFOSA, d7-MeFOSE, and d9-EtFOSE	Laboratories must include the isotope analog recoveries for each sample and analyte in data reports, including the calibration curve data.	Reanalyze samples, If the reinjected aliquot produces acceptable recoveries, report results from reanalysis, If the recovery fails again, the data may be reported with a qualifier.	Qualify outages and explain in case narrative.

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QC Item	Frequency	Acceptance Criteria			Corrective Action	Qualification
		WIDNR Guidance	Table B-15 per DoD QSM v5.3	MPCA Guidance		
		when the day the ICAL was performed. 10-150% for 13C8-PFOA, d3-MeFOA, d5-EtFOA, d7-MeFOE, and d9-EtFOE				
Dilution	As needed	N/A	N/A	When isotope dilution samples require a dilution, the volume of the diluent contains the same concentration of labeled isotope compounds as what was originally spiked into the sample. The isotope recovery results from the initial analysis should not be used to adjust the data from the secondary dilution analysis.		
Method Blank (MB)	1 per batch	Analytes $\leq 1/2$ the RL or $1/10^{\text{th}}$ the amount measured in any sample	Analytes $\leq 1/2$ the RL or $1/10^{\text{th}}$ the amount measured in any sample or $1/10^{\text{th}}$ the regulatory limit, whichever is greater	Two per batch of field samples, not to exceed 20 field samples. Must be $< 1/3$ the method reporting limit	1) If sample ND, report sample without qualification. 2) If sample result $> 10x$ MB detects and sample cannot be reanalyzed, report sample with appropriate qualifier indicating blank contamination. 3) If sample result $< 10x$ MB detects, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted to give authorization to report this data. 4) Analyte detection or failure of internal standard fails entire batch.	Qualify outages and explain in case narrative
LCS/ LCSD	1 spiked at a concentration \geq LOQ and \leq the mid-level calibration concentration	True Value $\leq 1-2x$ RL 50-150% True Value $> 2x$ RL 60-135% RPD $\leq 30\%$ NOTE: LCS must be spiked at 2x the MRL.	See Appendix E RPD $\leq 30\%$	In triplicate at 3 levels per analytical batch (low, medium, high). LCS must contain all project specific PFAS analytes in same media as associated samples. True value $\pm 30\%$ RPD $\leq 30\%$	Reanalyze and/or re-prepare batch of samples with new LCS. If LCS rec $>$ QC limits and these compounds are non-detect in the associated samples, the sample data may be reported with appropriate data qualifiers. If these criteria are not met, where extra samples are available, a re-extract is analyzed or else data is narrated.	Qualify outages and explain in case narrative
MS/MSD	1 pair/batch spiked at a concentration \geq LOQ and \leq the mid-level	70-130% RPD $\leq 30\%$	See Appendix E RPD $\leq 30\%$	70-130% RPD $\leq 30\%$	Failures are flagged but do not prevent reporting data if MB and LCS meet criteria.	Qualify outages and explain in case narrative

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QC Item	Frequency	Acceptance Criteria			Corrective Action	Qualification
		WIDNR Guidance	Table B-15 per DoD QSM v5.3	MPCA Guidance		
	calibration concentration					
Laboratory Field Duplicate (LFD)	1 per batch. A MSD may be substituted for a sample duplicate if sample is insufficient	N/A	N/A	1 per batch of 20 field samples or fewer	If these criteria are not met, results are labeled suspect due to matrix effects	Qualify outages and explain in case narrative
Tune Standard	Once or twice a year, when ICAL won't pass, when the peak shape is significantly off (indicating an MS problem), when major maintenance is performed, or instrument is moved	The instrument tuning with PPGs has its own manufacturing criteria-see the documentation	The instrument tuning with PPGs has its own manufacturing criteria-see the documentation	Mass calibration is done once or twice a year or as described by manufacturer	Refer to manufacturer criteria	N/A
Instrument Blank (ICB)	1 following the highest standard analyzed	< ½ RL	≤ ½ RL	≤ ½ RL	If acceptance criteria are not met after the highest calibration standard, calibration must be performed using a lower concentration for the highest standard until acceptance criteria is met.	Flagging is only appropriate in cases when the sample cannot be reanalyzed and when there is no more sample left.
CCB	1 following the CCV and prior to sample analysis	< ½ RL	N/A	N/A	If acceptance criteria are not met after the CCV. Clean the system and prepare new CCV if needed.	Flagging is only appropriate in cases when the sample cannot be reanalyzed and when there is no more sample left.
Equipment Blank	One per sampling event equipment is used in the field to collect samples	1/2 the MRL 1/10 the sample concentration	N/A	N/A		It is not necessary to qualify method blank detections between the MDL and one-half the MRL.
Field Blank	One per sampling event	1/2 the MRL 1/10 the sample concentration	N/A	N/A		It is not necessary to qualify method blank detections between the MDL and one-half the MRL.

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Appendix C: Typical MS/MS Method Conditions

Analyte	Precursor Ion (m/z)	Product Ion (m/z)	RT (min)	Declustering Potential (v)	Collision Energy (v)	Collision Cell Exit Potential (v)
PFBA	212.9	169	2.43	-45	-12	-11
PFPeA	262.9	219	3.33	-25	-12	-11
HFPO-DA	285	169	4.3	-70	-12	-11
HFPO-DA_2	285	185	4.29	-70	-24	-13
PFBS	298.9	80	4.27	-65	-58	-9
PFBS_2	298.9	99	4.27	-65	-40	-9
PFHxA	313	269	4.03	-25	-12	-19
PFHxA_2	313	119	4.02	-25	-28	-19
4:2FTS	327	307	3.75	-65	-28	-13
4:2FTS_2	327	81	3.75	-65	-56	-11
PFPeS	349	80	4.94	-45	-68	-9
PFPeS_2	349	99	4.94	-65	-40	-9
PFHpA	363	319	4.63	-50	-14	-15
PFHpA_2	363	169	4.63	-50	-24	-11
DONA	377	251	4.84	-50	-16	-11
DONA_2	377	85	4.84	-50	-36	-11
PFHxS	399	80	5.52	-55	-84	-9
PFHxS_2	399	99	5.52	-55	-68	-11
PFOA	413	369	5.17	-55	-14	-17
PFOA_2	413	169	5.17	-55	-24	-9
6:2FTS	427	407	4.89	-65	-32	-17
6:2FTS_2	427	81	4.89	-65	-68	-7
PFHpS	449	80	6.07	-105	-92	-9
PFHpS_2	449	99	6.07	-80	-80	-13
PFNA	463	419	5.7	-70	-16	-15
PFNA_2	463	169	5.7	-70	-26	-11
PFOSA	498	78	7.47	-130	-90	-11
PFOS	499	80	6.58	-65	-112	-9
PFOS_2	499	99	6.58	-65	-90	-11
N-MeFOSA	512	169	8.76	-55	-36	-11
N-MeFOSA_2	512	218.9	8.75	-60	-34	-19
PFDA	513	469	6.21	-80	-16	-19
PFDA_2	513	169	6.21	-80	-28	-13
N-EtFOSA	526	169	9.23	-40	-36	-13
N-EtFOSA_2	526	219.15	9.23	-15	-34	-9
8:2FTS	527	507	5.92	-70	-38	-21
8:2FTS_2	527	81	5.92	-70	-92	-9

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Analyte	Precursor Ion (m/z)	Product Ion (m/z)	RT (min)	Declustering Potential (v)	Collision Energy (v)	Collision Cell Exit Potential (v)
9CI-PF3ONS	530.9	351	6.97	-75	-36	-15
9CI-PF3ONS_2	530.9	83	6.97	-75	-70	-11
PFNS	549	80	7.08	-65	-118	-13
PFNS_2	549	99	7.08	-85	-96	-11
PFUdA	563	519	6.72	-30	-18	-21
PFUdA_2	563	169	6.71	-30	-32	-11
N-MeFOSAA	570	419	6.11	-125	-28	-33
N-MeFOSAA_2	570	483	6.11	-125	-22	-33
N-EtFOSAA	584	419	6.34	-125	-28	-33
N-EtFOSAA_2	584	526	6.33	-125	-28	-33
PFDS	599	80	7.57	-85	-122	-11
PFDS_2	599	99	7.57	-85	-100	-11
PFDaA	613	569	7.21	-25	-18	-23
PFDaA_2	613	169	7.21	-25	-34	-11
N-MeFOSE	616	59	8.58	-20	-76	-5
10:2FTS	627	607	6.92	-50	-44	-25
10:2FTS_2	627	81	6.91	-50	-108	-9
N-EtFOSE	630	59	9.03	-20	-58	-27
11CI-PF3OUdS	630.9	451	7.94	-90	-40	-19
11CI-PF3OUdS_2	630.9	99	7.94	-90	-92	-5
PFTrDA	663	619	7.69	-75	-20	-25
PFTrDA_2	663	169	7.69	-75	-34	-9
PFDoS	699	80	8.46	-30	-134	-9
PFDoS_2	699	99	8.46	-20	-132	-11
PFTeDA	713	669	8.17	-85	-20	-27
PFTeDA_2	713	169	8.17	-85	-36	-13
PFHxDA	813	769	9.23	-30	-22	-33
PFHxDA_2	813	169	9.23	-30	-38	-9
PFODA	913	869	9.72	-5	-22	-35
PFODA_2	913	169	9.72	-5	-42	-11

NOTE: Analyte_2 Ions used for confirmation purposes.

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Appendix D: PFAS Analyte and Recommended Extracted Internal Standard Used for Quantification

Analyte	EIS Name
PFBA	13C4_PFBFA
PFPeA	13C5_PFPeA
HFPO-DA	13C3_HFPO-DA
PFBS	13C3_PFBFS
PFHxA	13C5_PFHxA
4:2FTS	13C2_4:2FTS
PFPeS	13C3_PFHxS
PFHpA	13C4_PFHpA
DONA	13C8_PFOA
PFHxS	13C3_PFHxS
PFOA	13C8_PFOA
6:2FTS	13C2_6:2FTS
PFHpS	13C3_PFOS
PFNA	13C9_PFNA
PFOSA	13C8_PFOSA
PFOS	13C8_PFOS
N-MeFOSA	d3-N-MeFOSA
PFDA	13C6_PFDA
N-EtFOSA	d5-N-EtFOSA
8:2FTS	13C2_8:2FTS
9Cl-PF3ONS	13C8_PFOS
PFNS	13C8_PFOS
PFUdA	13C7_PFUdA
N-MeFOSAA	d3-MeFOSAA
N-EtFOSAA	d5-EtFOSAA
PFDS	13C8_PFOS
PFDaA	13C2_PFDaA
N-MeFOSE	d7-N-MeFOSE
10:2FTS	13C2_8:2FTS
N-EtFOSE	d9-N-EtFOSE
11Cl-PF3OUdS	13C8_PFOS
PFTTrDA	13C2_PFDaA
PFDoS	13C8_PFOS
PFTeDA	13C2_PFTeDA
PFHxDA	13C2_PFHxDA
PFODA	13C2_PFHxDA

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Appendix E: PFAS by LCMSMS Compliant with DoD QSM Batch Control Limits

CAS#	Analyte Acronym	Aqueous Matrix		Solid Matrix		Tissue Matrix ³	
		Lower Control Limit (%REC)	Upper Control Limit (%REC)	Lower Control Limit (%REC)	Upper Control Limit (%REC)	Lower Control Limit (%REC)	Upper Control Limit (%REC)
2991-50-6	N-EtFOSAA	61	135	61	139	60	140
2355-31-9	N-MeFOSAA	65	136	63	144	60	140
757124-72-4	4:2 FTS	63	143	62	145	60	140
27619-97-2	6:2 FTS	64	140	64	140	60	140
39108-34-4	8:2 FTS	67	138	65	137	60	140
375-73-5	PFBS	72	130	72	128	60	140
375-22-4	PFBA	73	129	71	135	60	140
335-77-3	PFDS	53	142	59	134	60	140
335-76-2	PFDA	71	129	69	133	60	140
307-55-1	PFDoA	72	134	69	135	60	140
375-92-8	PFHpS	69	134	70	132	60	140
375-85-9	PFHpA	72	130	71	131	60	140
355-46-4	PFHxS	68	131	67	130	60	140
307-24-4	PFHxA	72	129	70	132	60	140
68259-12-1	PFNS	69	127	69	125	60	140
375-95-1	PFNA	69	130	72	129	60	140
754-91-6	PFOSA	67	137	67	137	60	140
1763-23-1	PFOS	65	140	68	136	60	140
335-67-1	PFOA	71	133	69	133	60	140
2706-91-4	PFPeS	71	127	73	123	60	140
2706-90-3	PFPeA	72	129	69	132	60	140
376-06-7	PFTeDA	71	132	69	133	60	140
72629-94-8	PFTrDA	65	144	66	139	60	140
2058-94-8	PFUdA	69	133	64	136	60	140
31506-32-8	N-MeFOSA ¹	68	141	70	140	60	140
4151-50-2	N-EtFOSA ²	70	140	70	140	60	140
120226-60-0	10:2FTS ²	70	140	70	140	60	140
13252-13-6	HFPO-DA ²	70	140	70	140	NA	NA
919005-14-4	DONA ¹	70	140	70	140	60	140
756426-58-1	9Cl-PF3ONS ²	70	140	70	140	60	140
763051-92-9	¹¹ Cl-PF3OUDS ²	70	140	70	140	60	140
24448-09-7	N-MeFOSE ²	70	140	70	140	60	140
1691-99-2	N-EtFOSE ²	70	140	70	140	60	140

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CAS#	Analyte Acronym	Aqueous Matrix		Solid Matrix		Tissue Matrix ³	
		Lower Control Limit (%REC)	Upper Control Limit (%REC)	Lower Control Limit (%REC)	Upper Control Limit (%REC)	Lower Control Limit (%REC)	Upper Control Limit (%REC)
67905-19-5	PFHxDA ²	70	140	70	140	60	140
16517-11-6	PFODA ²	60	140	60	140	60	140
79780-39-5	PFDoS ²	60	140	60	140	45	140


¹ DoD currently does not have guidance for the analyte in solid matrix in Table B-15 per DoD QSM 5.3, Appendix B, as of June 2019.

² DoD currently does not have guidance for the analyte in both aqueous and solid matrix in Table B-15 per DoD QSM 5.3, Appendix B, as of June 2019.

³ DoD currently does not have guidance for the analyte in tissue matrix in Table B-15 per DoD QSM 5.3, Appendix B, as of June 2019.

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Appendix F: DoD QSM 5.3, Appendix B, Table B-15 - Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Qualification Criteria	Comments
Aqueous Sample Preparation	Each sample and associated batch QC samples.	<p>Solid Phase Extraction (SPE) must be used unless samples are known to contain high PFAS concentrations (e.g., Aqueous Film Forming Foam (AFFF) formulations). Inline SPE is acceptable.</p> <p>Entire sample plus bottle rinsate must be extracted using SPE.</p> <p>Known high PFAS concentration samples require serial dilution be performed in duplicate.</p> <p>Documented project approval is needed for samples prepared by serial dilution as opposed to SPE.</p>	NA.	NA.	Samples with >1% solids may require centrifugation prior to SPE extraction. Pre-screening of separate aliquots of aqueous samples is recommended.
Solid Sample Preparation	Each sample and associated batch QC samples.	Entire sample received by the laboratory must be homogenized prior to subsampling.	NA.	NA.	NA.
Biota Sample Preparation	Each sample and associated batch QC samples.	Sample prepared as defined by the project (e.g., whole fish versus filleted fish).	NA.	NA.	NA.
AFFF and AFFF Mixture Samples Preparation	Each sample and associated batch QC samples.	<p>Each field sample must be prepared in duplicate (equivalent to matrix duplicate).</p> <p>Serial dilutions must be performed to achieve the lowest LOQ possible for each analyte.</p>	NA.	NA.	<p>Adsorption onto bottle is negligible compared to sample concentration so subsampling is allowed.</p> <p>Multiple dilutions will most likely have to be reported in order to achieve the lowest LOQ possible for each analyte.</p>
Sample Cleanup Procedure	<p>Each sample and associated batch QC samples.</p> <p>Not applicable to AFFF and AFFF Mixture Samples.</p>	ENVI-Carb™ or equivalent must be used on each sample and batch QC sample.	NA.	Flagging is not appropriate.	Cleanup should reduce bias from matrix interferences.
Mass Calibration	Instrument must	Calibrate the mass scale	If the mass	Flagging is not	Problem must be

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QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Qualification Criteria	Comments
	<p>have a valid mass calibration prior to any sample analysis.</p> <p>Mass calibration is verified after each mass calibration, prior to initial calibration (ICAL).</p>	<p>of the MS with calibration compounds and procedures described by the manufacturer.</p> <p>Mass calibration range must bracket the ion masses of interest. The most recent mass calibration must be used for every acquisition in an analytical run.</p> <p>Mass calibration must be verified to be ± 0.5 amu of the true value, by acquiring a full scan continuum mass spectrum of a PFAS stock standard.</p>	<p>calibration fails, then recalibrate. If it fails again, consult manufacturer instructions on corrective maintenance.</p>	<p>appropriate.</p>	<p>corrected. No samples may be analyzed under a failing mass calibration.</p> <p>The mass calibration is updated on an as-needed basis (e.g., QC failures, ion masses fall outside of the ± 0.5 amu of the true value, major instrument maintenance is performed, or the instrument is moved).</p>
Mass Spectral Acquisition Rate	Each analyte, Extracted Internal Standard (EIS) Analyte.	A minimum of 10 spectra scans are acquired across each chromatographic peak.	NA.	Flagging is not appropriate.	NA.
Calibration, Calibration Verification, and Spiking Standards	All analytes.	<p>Standards containing both branched and linear isomers must be used when commercially available.</p> <p>PFAS method analytes may consist of both branched and linear isomers, but quantitative standards that contain the linear and branched isomers do not exist for all method analytes.</p> <p>For PFAS that do not have a quantitative branched and linear standard, identify the branched isomers by analyzing a qualitative standard that includes both linear and branched isomers and determine retention times, transitions and transition ion ratios. Quantitate samples by integrating the total response (i.e., accounting for peaks that are identified as linear and branched isomers) and relying on the initial calibration that uses the linear isomer quantitative</p>	NA.	Flagging is not appropriate.	<p>Standards containing both branched and linear isomers are to be used during method validation and when reestablishing retention times, to ensure the total response is quantitated for that analyte.</p> <p>Technical grade standards cannot be used for quantitative analysis.</p>

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QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Qualification Criteria	Comments
Sample PFAS Identification	All analytes detected in a sample.	<p>standard.</p> <p>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 and PFPeA).</p> <p>Documentation of the primary and confirmation transitions and the ion ratio is required.</p> <p>In-house acceptance criteria for evaluation of ion ratios must be used and must not exceed 50-150%.</p> <p>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.</p> <p>Quant ion and confirmation ion must be present and must maximize simultaneously (± 2 seconds).</p>	NA.	<p>PFAS identified, with ion ratios that fail acceptance criteria, must be qualified in the final report.</p> <p>Any quantitation ion peak that does not meet the maximization criteria shall be included in the summed integration and the resulting data qualified as "estimated and biased high".</p>	<p>For example: Ion Ratio = (quant ion abundance/confirm ion abundance)</p> <p>Calculate the average ratio (A) and standard deviation (SD) using the ICAL standards. An acceptance range of ratio could be within $A \pm 3SD$ for confirmation of detection.</p>
Ion Transitions (Precursor-> Product)	Every field sample, standard, blank, and QC sample.	<p>In order to avoid biasing results high due to known interferences for some transitions, the following transitions must be used for the quantification of the following analytes:</p> <p>PFOA: 413 → 369 PFOS: 499 → 80 PFHxS: 399 → 80 PFBS: 299 → 80 4:2 FTS: 327 → 307 6:2 FTS: 427 → 407 8:2 FTS: 527 → 507 NEtFOSAA: 584 → 419 NMeFOSAA: 570 → 419</p> <p>If these transitions are not used, the reason must be technically</p>	NA.	Flagging is not appropriate	NA.

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QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Qualification Criteria	Comments
		justified and documented (e.g., alternate transition was used due to observed interferences).			
Initial Calibration (ICAL)	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	<p>The isotopically labeled analog of an analyte (Extracted Internal Standard Analyte) must be used for quantitation if commercially available (Isotope Dilution Quantitation).</p> <p>Commercial PFAS standards available as salts, are acceptable, providing the measured mass is corrected to the neutral acid concentration. Results shall be reported as the neutral acid with appropriate CAS number.</p> <p>If a labeled analog is not commercially available, the Extracted Internal Standard Analyte with the closest retention time or chemical similarity to the analyte must be used for quantitation. (Internal Standard Quantitation)</p> <p>Analytes must be within 70-130% of their true value for each calibration standard.</p> <p>ICAL must meet one of the two options below:</p> <p>Option 1: The RSD of the RFs for all analytes must be ≤20%.</p> <p>Option 2: Linear calibrations must have $r^2 \geq 0.99$ for each analyte.</p>	Correct problem, then repeat ICAL.	Flagging is not appropriate.	<p>No samples shall be analyzed until ICAL has passed.</p> <p>External Calibration is not allowed for any analyte.</p> <p>Calibration can be linear (minimum of 5 standards); weighting is allowed.</p>
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 EIS.
Retention Time (RT) window	Every field sample,	RT of each analyte and EIS analyte must fall	Correct problem and	NA.	Calculated for each analyte and EIS.

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QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Qualification Criteria	Comments
width	standard, blank, and QC sample.	<p>within 0.4 minutes of the predicted retention times from the daily calibration verification or on days when ICAL is performed, from the midpoint standard of the ICAL.</p> <p>Analytes must elute within 0.1 minutes of the associated EIS. This criterion applies only to analyte and labeled analog pairs.</p>	reanalyze samples.		
Instrument Sensitivity Check (ISC)	Prior to analysis and at least once every 12 hours.	Analyte concentrations must be at LOQ; concentrations must be within $\pm 30\%$ of their true values.	Correct problem, rerun ISC. If problem persists, repeat ICAL.	Flagging is not appropriate.	<p>No samples shall be analyzed until ISC has met acceptance criteria.</p> <p>ISC can serve as the initial daily CCV.</p>
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	Analyte concentrations must be within $\pm 30\%$ of their true value.	Correct problem, rerun ICV. If problem persists, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified.
Continuing Calibration Verification (CCV)	Prior to sample analysis, after every 10 field samples, and at the end of the analytical sequence.	<p>Concentration of analytes must range from the LOQ to the mid-level calibration concentration.</p> <p>Analyte concentrations must be within $\pm 30\%$ of their true value.</p>	<p>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.</p> <p>Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.</p>	<p>If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative.</p> <p>Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.</p>	<p>Results may not be reported without valid CCVs.</p> <p>Instrument Sensitivity Check (ISC) can serve as a bracketing CCV.</p>

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QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Qualification Criteria	Comments
Instrument Blanks	Immediately following the highest standard analyzed and daily prior to sample analysis.	<p>Concentration of each analyte must be $\leq \frac{1}{2}$ the LOQ.</p> <p>Instrument Blank must contain EIS to enable quantitation of contamination.</p>	<p>If acceptance criteria are not met after the highest calibration standard, calibration must be performed using a lower concentration for the highest standard until acceptance criteria is met.</p> <p>If sample concentrations exceed the highest allowed standard and the sample(s) following exceed this acceptance criteria ($>1/2$ LOQ), they must be reanalyzed.</p>	Flagging is only appropriate in cases when the sample cannot be reanalyzed and when there is no more sample left.	<p>No samples shall be analyzed until instrument blank has met acceptance criteria.</p> <p>Note: Successful analysis following the highest standard analyzed determines the highest concentration that carryover does not occur.</p> <p>When the highest standard analyzed is not part of the calibration curve, it cannot be used to extend out the calibration range, it is used only to document a higher concentration at which carry over still does not occur.</p>
Extracted Internal Standard (EIS) Analytes	Every field sample, standard, blank, and QC sample.	<p>Added to solid sample prior to extraction. Added to aqueous samples, into the original container, prior to extraction.</p> <p>For aqueous samples prepared by serial dilution instead of SPE, added to final dilution of samples prior to analysis.</p> <p>Extracted Internal Standard Analyte recoveries must be within 50% to 150% of ICAL midpoint standard area or area measured in the initial CCV on days when an ICAL is not performed.</p>	<p>Correct problem. If required, re-extract and reanalyze associated field and QC samples.</p> <p>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).</p> <p>Samples may be re-extracted and analyzed outside of hold times, as</p>	Apply Q-flag and discuss in the Case Narrative only if reanalysis confirms failures in exactly the same manner.	<p>Failing analytes shall be thoroughly documented in the Case Narrative.</p> <p>EIS should be 96% (or greater) purity. When the impurity consists of the unlabeled analyte, the EIS can result in a background artifact in every sample, standard and blank, if the EIS is fortified at excessive concentrations.</p>

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QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Qualification Criteria	Comments
			necessary for corrective action associated with QC failure.		
Method Blank (MB)	One per preparatory batch.	No analytes detected > $\frac{1}{2}$ LOQ or > $1/10^{\text{th}}$ the amount measured in any sample or $1/10^{\text{th}}$ the regulatory limit, whichever is greater.	<p>Correct problem. If required, re-extract and reanalyze MB and all QC samples and field samples processed with the contaminated blank.</p> <p>Samples may be re-extracted and analyzed outside of hold times, as necessary for corrective action associated with QC failure.</p> <p>Examine the project-specific requirements. Contact the client as to additional measures to be taken.</p>	<p>If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative.</p> <p>Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.</p>	<p>Results may not be reported without a valid MB.</p> <p>Flagging is only appropriate in cases where the samples cannot be reanalyzed.</p>
Laboratory Control Sample (LCS)	One per preparatory batch.	<p>Blank spiked with all analytes at a concentration \geq LOQ and \leq the mid-level calibration concentration.</p> <p>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.</p>	<p>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.</p> <p>Samples may be re-extracted and analyzed</p>	<p>If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative.</p> <p>Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.</p>	<p>Results may not be reported without a valid LCS.</p> <p>Flagging is only appropriate in cases where the samples cannot be reanalyzed.</p>

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QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Qualification Criteria	Comments
			<p>outside of hold times, as necessary for corrective action associated with QC failure.</p> <p>Examine the project-specific requirements. Contact the client as to additional measures to be taken.</p>		
Matrix Spike (MS)	<p>One per preparatory batch.</p> <p>Not required for aqueous samples prepared by serial dilution instead of SPE.</p>	<p>Sample spiked with all analytes at a concentration \geq LOQ and \leq the mid-level calibration concentration.</p> <p>A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified.</p> <p>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.</p>	<p>Examine the project-specific requirements. Contact the client as to additional measures to be taken.</p>	<p>For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.</p>	<p>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).</p>
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	<p>For MSD: One per preparatory batch.</p> <p>For MD: Each aqueous sample prepared by serial dilution instead of SPE.</p>	<p>For MSD: Sample spiked with all analytes at a concentration \geq LOQ and \leq the mid-level calibration concentration.</p> <p>A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified.</p> <p>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.</p> <p>RPD \leq 30% (between MS and MSD or sample and MD).</p>	<p>Examine the project-specific requirements. Contact the client as to additional measures to be taken. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.</p>	<p>For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.</p>	<p>The data shall be evaluated to determine the source of difference.</p> <p>For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is \geqLOQ.</p> <p>The MD is a second aliquot of the field sample that has been prepared by serial dilution.</p>
Post Spike Sample	<p>Only applies to aqueous samples prepared by serial dilution instead of SPE</p>	<p>Spike all analytes reported as $<$LOQ into the dilution that the result for that analyte is reported from. The spike must be at the LOQ</p>	<p>When analyte concentrations are calculated as $<$LOQ, and the spike recovery</p>	<p>Flagging is not appropriate.</p>	<p>When analyte concentrations are calculated as $<$LOQ, results may not be reported without acceptable post spike</p>

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QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Qualification Criteria	Comments
	that have reported value of <LOQ for analyte(s).	concentration to be reported for this sample as <LOQ. When analyte concentrations are calculated as <LOQ, the post spike for that analyte must recover within 70-130% of its true value.	does not meet the acceptance criteria, the sample, sample duplicate, and post spike sample must be reanalyzed at consecutively higher dilutions until the criteria is met.		recoveries.

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Title Page

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Signatory Attestation: I attest the application of my electronic signature on this title page affirms my management commitment and responsibility to uphold the requirements of the PAS Quality Management System (QMS) described in this Quality Manual (manual) at each location for which this manual is prepared.

Refer to the Quality Manual Signatory Page to view the job title and physical address for each signatory.



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Quality Manual Approval Signatories

The following individuals represent the PAS corporate and local management team responsible for implementing the PAS Quality Management System (QMS) and upholding the requirements of this manual at the location(s) for which this manual was prepared, at the time this version of the manual was made effective, and that correlate with the electronic signatures shown on the title page of this manual.

If these persons(s) change positions, leave the company, or are on extended leave of absence, the approval of this manual automatically transfers to the person replacing the signatory or to the signatory's primary or alternate deputy until the manager is replaced and/or the manager returns to work. The individual replacing the signatory automatically accepts the responsibilities associated with the original signatory's attestation. Refer to Section 4.1.5.1.1 of this manual for the deputies assigned to key personnel job titles.

The manual is not revised and released under an updated version for the sole purpose of updating personnel change(s). Personnel information is updated when the next revision of the manual is released. See manual Sections 1.2.1 and 1.2.2 for more information about how this manual is maintained.

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1.0 PURPOSE AND SCOPE

1.1 Purpose

This quality manual (manual) outlines the quality management system (QMS) and management structure of Pace[®] Analytical Services, LLC. Pace[®] Analytical Services, LLC is referred to by brand name Pace[®] Analytical Services and by the acronyms PAS or ENV. The acronyms PAS and ENV are interchangeable.

The PAS QMS is also referred to as the quality program throughout this manual and other PAS documents. The phrases “quality management system” and “quality program” are synonymous and are referred to by the acronym QMS.

The QMS is the collection of policies and processes established by the senior leaders of PAS (top management) to ensure the service and products provided by PAS consistently meet relevant requirements and achieves the goal of Pace[®] to provide customers with high quality, cost-effective, analytical measurements, and services.

The QMS is also planned to establish conformance¹ and compliance with the current published 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*

¹The 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 QMS is planned to achieve regulatory compliance with the various federal and state programs for which PAS locations provide compliance testing and/or holds certification or accreditation. Federal or state requirements that do not apply to all PAS locations, 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 location associated with this manual is provided in Appendix A.
- A list of analytical testing capabilities offered by each location associated with this manual is provided in Appendix B.

1.2 Scope and Application

This manual applies to each location listed on the Title Page of this manual, including PAS laboratories, satellite laboratories, service centers, and supporting business functions.

For purposes of the PAS QMS:

- The term “location” used in this manual refers to laboratories and/or service centers.
- The term “laboratory” refers to any PAS location, however named by Pace[®] that provides testing, collects samples (sampling), or conducts field measurement services in a fixed building, mobile unit, or in-situ (field).

- The phrase “service center” refers to any PAS location, however named by Pace[®] that does not perform any testing, sampling, or field measurements.
- The phrase “satellite laboratory” refers to a limited-service laboratory affiliated to a larger business unit or location. Some PAS business groups, such as accounting, may refer to a satellite laboratory as a “service center.” Irrespective of internal jargon or reference by any group, any PAS location that generates a test result for external use is a “laboratory” and must comply with the requirements specified in this manual for all analytical testing services.

PAS locations are defined by physical address. Laboratories are defined by physical address and certification/accreditation ID except mobile units which may be defined by the address of the location to which they are assigned, by VIN (vehicle identification number), or by certification/accreditation ID. Laboratories that provide sampling and field testing are defined by the physical address of the PAS location to which they are affiliated and that manages these activities.

1.2.1 Quality Manual Template

This manual was prepared using the PAS Quality Manual Template (template) created by the PAS Corporate Quality Director (CQD).

The template, known as document ID ENV-TMP-CORQ-0007, specifies the minimum requirements that every PAS location must abide by, regardless of scope of services or number of personnel, to maintain a quality program that achieves the objectives of the PAS Quality Policy (see Section 4.2.2).

The template is the mechanism used by top management to communicate to PAS personnel their commitment to continuously develop and improve the QMS for effectiveness, to meet customer expectations, and to comply with any statutory and regulatory requirements. Their signature of approval on this template is the mechanism used to document this responsibility.

“Top Management” is the phrase used by the TNI Standard to refer to the leaders of an organization that develop and/or release the PAS Quality Policy Statement and QMS under their authority

For PAS, these managers include the Chief Executive Officer (CEO) and Chief Compliance Officer (CCO) of Pace[®] and the President, CQD, Senior Vice President of Operations (Sr. VPO), and the Chief Technical Officer (CTO) of PAS.

The template and instructions for use of the template are released by corporate quality personnel to local quality managers responsible for each location (Local QM). The local QM uses the template to prepare the location manual by following the instructions provided to them. The local QM 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 unique to each location are provided in addendum or in documents that supplement the manual.

The template is reviewed by corporate quality personnel annually and updated, if needed. More frequent review and revision may occur to manage change, to maintain conformance and compliance to relevant standards or to improve the QMS.

See standard operating procedure (SOP) ENV-SOP-CORQ-00015 *Document Management and Control* for more information.

1.2.2 Quality Manual

The quality manual is created from template ENV-TMP-CORQ-0007 by local quality personnel, who are also responsible for maintenance and management of the document.

- PAS locations are not permitted to alter content of the template when preparing their manual, except where specified in the template. Control of content in the manual is necessary to ensure consistency of implementation of the PAS quality program across the network.
- If additions or changes to the manual are needed to maintain regulatory compliance or conformance to relevant standards and these changes cannot be covered by addendum to the manual, the need for change must be raised to the PAS Corporate Quality Director, who will decide how to resolve the need.

The manual is approved for release by the management team listed on the Quality Manual Approval Signatory Page. The manager's electronic signature on the Title Page of the manual affirms their commitment to implement and uphold the requirements, processes, and procedures of the PAS QMS at each location for which the manual was prepared.

The manual is reviewed annually and updated with each release of a new version of the template, and as needed to update appendices and addendum. More frequent review and revision may be necessary when there are significant changes to the capabilities and resources of the laboratory during the calendar year

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 organization documents that support the QMS such as policies and standard operating procedures (SOPs).

These references may include the document's document control number (DC#) and the document title. This information is subject to change at the discretion of PAS. The manual and/or template are updated to reflect the editorial change during the manual's next scheduled review/revision cycle or the next time a version of the manual is released, whichever is sooner.

Each location maintains a current list of documents used by the location to support the QMS. This list, known as the "Master List", is readily available to personnel for their use and it provides a cross reference to the legacy document ID, where applicable. Parties external to PAS may contact the location of interest to obtain the most current version of the Master List for their use as needed.

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.
 - “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 PAS to support the QMS.

4.0 MANAGEMENT REQUIREMENTS

4.1 Organization

4.1.1 Legal Identity

Pace® Analytical Services, LLC (Pace® Analytical Services) is the responsible entity authorized by the State of Minnesota to do business as a limited liability company, under the parent company, PAS Parent, Inc.

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, PAS management is responsible to notify regulatory authorities of the change within the timeframe 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

PAS management has the responsibility and authority to establish and implement procedures and to maintain resources necessary to assure its activities are carried out in such a way to meet the federal and statutory requirements in addition to the requirements of the PAS QMS. Also See Section 1.1.

4.1.3 Scope of the Quality Management System

The QMS 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 business purpose of PAS is to deliver the highest standard of testing and scientific services in the market. We offer the most advanced solutions in the industry, backed by transparent data, a highly trained team, and the service and support that comes from over four decades of experience.

4.1.4.1 Organization Structure

Each PAS location is led by a management team referred to as local management¹. Local management is responsible for making day-to-day decisions regarding the operations of the facility and implementing, and sustaining the requirements, policies, and procedures of the PAS quality program.

The roles that make up the local management team include a Vice President of Operations (VPO), a General Manager (GM) or Director of Laboratory Operations (DLO), a Quality Program Manager (QPM), and the Quality Manager (QM).

¹ The term “local management” does not mean “on-site” management. Some of the roles that make up the local management team, work off site or from a different PAS location. Refer to the Quality Manual Approval page at the beginning of this manual for the physical address of each manager that comprises the local management team.

The local management team is supported by department specific supervisors and in some PAS locations, a site supervisor or operations manager.

Local management and supervisors are supported by personnel from functional groups that support the division, such as HR, IT, Sales & Marketing, Finance, and EHS (Environmental Health & Safety).

Technical oversight for each location is provided by local personnel with support and guidance from the PAS Chief Technical Officer (CTO), PAS corporate quality personnel, and the Pace® compliance team. Locations that hold TNI accreditation, also have personnel appointed to serve as the “acting technical manager for TNI, however named” to perform the duties and responsibilities of this designation per the TNI Standard. See Section 4.1.5.2.1 for more information on this TNI requirement.

The reporting relationships and responsibilities of quality personnel are independent of operations in order to safeguard impartiality. See Section 4.1.5.2 for more information.

Refer to the organization charts provided in Appendix D to view the organization structure, reporting relationships, and the interrelationships between positions.

4.1.5 Management Requirements

4.1.5.1 Personnel

Each PAS location is staffed with administrative and/or technical personnel who perform and verify work under the supervision of their direct line supervisor.

All personnel are expected to perform 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. PAS 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 are management positions that have the authority and responsibility to plan, direct, and control activities related to the QMS for the entire division (PAS Corporate), or for one or more PAS locations (Local).

PAS Key Personnel Positions & Deputy Assignments by Role

Job Title	Acronym	Primary / Alternate Deputy
Chief Executive Officer	CEO	President
Chief Compliance Officer	CCO	CQD
President	NA	CEO / Sr. VPO
Corporate Quality Director	CQD	CCO

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Job Title	Acronym	Primary / Alternate Deputy
Quality Program Manager	QPM	CQD / Peer QPM
Chief Technical Officer	CTO	CQD / CCO
Sr. VP of Operations	Sr. VPO	President / VPO
Vice President of Operations	VPO	Sr. VPO / Peer VPO
Director of Lab Operations ¹	DLO	VPO / Peer GM or Sr. VPO
Health and Safety Director	NA	CCO
IT Director	NA	CTO
Quality Manager	QM	Direct QPM / Peer QPM
General Manager ¹	GM	VPO / Sr. VPO or Peer GM
Operations Manager ¹	OM	GM / DL or VPO
Technical Manager ¹	TM	CTO / Peer TM
TNI Approved TM ²	TNI TM	Another Qualified Employee

¹Position is not in place at all locations.

²The TNI TM is not a PAS position. See Section 4.1.5.2.1 for more information.

Some certification and accreditation programs require notification when there is a change in key personnel. Notification requirements and timeframes 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 at Pace® are detailed in job descriptions maintained by the Pace® Human Resource personnel (HR).

The following sections provide a general overview of various management and supervisory roles and are presented in no particular order.

Chief Executive Officer (CEO): Provides leadership for overall operations; oversight of regulatory and compliance standards; development of growth strategies; and long-range capital and strategic planning for Pace®.

Chief Compliance Officer (CCO): Has overall responsibility for statutory and regulatory compliance and the environmental health and safety programs (EHS) for Pace®.

President: Provides leadership for overall operations; oversight of regulatory and compliance standards; development of growth strategies; and long-range capital and strategic planning for PAS.

Chief Technical Officer (CTO): Provides technical oversight and leadership to all PAS locations. Responsible for innovation and standardization of technical activities.

Corporate Director of Quality (CQD): Responsible for developing the PAS quality program and the policies and procedures that support the QMS. The CQD leads the quality team, establishing functions, responsibilities, duties, and organization structure for PAS.

Corporate Quality Program Manager (QPM): Responsible for helping local management implement, monitor, maintain and improve the PAS quality program for one or more locations in the network and for direct supervision of Quality Manager(s).

Director of Information Technology: Oversees and delivers the systems and processes of information technology used by PAS. These systems include Laboratory Information Management Systems (LIMS); data acquisition, reduction, and reporting software; virus-protection, communication tools, and ensuring the integrity, security of electronic data, and associated policies and procedures.

Sr. Vice President of Operations (Sr VPO): Provides leadership, direction, and insight necessary to achieve strategic initiatives. Develops and improves processes, structure, and allocation of resources for operations for all of PAS.

Vice-President of Operations (VPO): Provides leadership, guidance, and resources, including allocation of personnel, necessary to achieve the strategic goals of the organization and the PAS quality program to one or more PAS locations.

Director of Laboratory Operations (DLO): See description for General Manager.

General Manager (GM): The GM is responsible for overall administration and operation of one or more PAS locations and service centers. Although task duties associated with this responsibility may be delegated, the GM is responsible for ensuring all duties and activities of the locations they oversee comply with the PAS QMS, the PAS EHS program, and with any applicable statutory, regulatory requirements or program requirements.

Any GM of a NELAC/TNI Accredited laboratory 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 the implementation, compliance, and improvement of the QMS and communicates gaps, deviations, and opportunities for improvement to local and corporate laboratory management. The QM is independent of the operation and analytical activities for which they provide oversight and has the authority to carry out the roles and responsibilities of their position without outside influence.

The QM:

- serves as the focal point for QA/QC protocol decisions and oversees review of QC data for trend analysis;
- evaluates data objectively and performs assessments without outside influence;
- has documented training and experience in QA/QC procedures and the PAS 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;

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- 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 the training and supervision of project manager(s) and/or shipping, receiving and courier personnel. The primary responsibility of the CSM is to ensure projects are successfully managed to meet the expectations and needs of PAS customers.

Department Managers / Supervisors / Team Lead: These positions are responsible for administrative and operations management and implementation of the QMS 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 QMS; method development, validation and the establishment and implementation of SOPs to assure regulatory compliance and suitability for the 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 Approved Technical Manager (TNI Accreditation Only):

The requirements in this subsection apply to only to PAS locations that are NELAC/TNI accredited.

The TNI Standard specifies requirements for the qualification and duties of technical personnel. The TNI Standard lists these duties under the reference “technical manager(s), however named.”

At PAS, these duties closely correlate with the responsibilities and duties outlined in the PAS job descriptions for managers, supervisors, team leads, and/or scientist. However, these duties do not need to be associated with any specific job title and can be assigned to any one or more PAS employees that meets the qualifications specified in the TNI Standard.

Refer to the applicable version of the TNI Standard to view the required qualifications for each discipline.

PAS locations that are TNI accredited must designate one or more employees to perform these duties and submit these qualifications to the TNI accreditation body (AB) for approval.

Employees approved by the TNI AB, to perform these duties retain their Pace® assigned job title.

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 perform duties of the ‘technical manager, however named’ as specified in the TNI Standard and not to a PAS job title.

The duties of any approved technical manager for TNI, however named, can be completed in person or remotely. If an employee that is an approved technical manager for TNI is completely absent from work or on a leave of absence for more than 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 local QM must formally notify the TNI primary AB of the absence and the details of reassignment of duties in writing.

4.1.5.3 Conflict of Interest

A conflict of interest is a situation where a person has competing interests that may affect impartiality. It is the policy of Pace[®] to ensure business relationships, decisions and transactions do not place personal interest ahead of the organization, customers, colleagues, job responsibilities or the public we serve. Conflict of interest is avoided by making personnel aware of circumstances that conflict or appear to conflict with impartiality and/or designing process and procedures to include checks and balances to prevent conflict and ensure impartiality.

See the current version of policy COR-POL-0004 *Code of Ethics and Professional Conduct* for more information.

4.1.5.4 Confidentiality

PAS management is committed to preserving the confidentiality of Pace[®] customers and confidentiality of Pace[®] business information.

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 Pace[®] is required by law to release confidential information to another party, such as a regulatory agency or for litigation purposes. In which case, Pace[®] will notify the client of the release of information and the information provided, unless notification is prohibited by law.

When Pace[®] obtains information about the customer from a source other than the customer, Pace[®] will keep the source of the information confidential unless disclosure is agreed upon by the source.

The terms of client confidentiality are included in PAS Standard Terms and Conditions (T&C). With the acceptance of the T&C and/or the implicit contract for analytical services that occurs when the client sends samples to PAS for testing, the client authorizes Pace[®] to release confidential information when required. Other procedures used by PAS to maintain confidentiality include:

- A Code of Ethics and Professional Conduct policy that covers this topic (COR-POL-0004):
- A Confidentiality Agreement which supervisory and sales personnel and other positions 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; and
- Physical access controls and encryption of electronic data; and

See policy COR-POL-0004 *Code of Ethics and Professional Conduct* 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 people included in the communication gets the point and understands it.

4.1.5.5.1 Workplace Communication

Effective communication in the workplace is necessary to assure work is performed correctly, efficiently, and in accordance with client specifications.

Instructions for how to conduct testing and other work activities are communicated to personnel via written policies, standard operating procedures, and other work instructions.

Information about PAS performance (positive and negative) and ideas for improvement are communicated to personnel 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.

PAS management is responsible for training personnel to communicate in professional and respectful ways to build strong relationships and to avoid misunderstanding.

4.2 Quality Management System

4.2.1 Quality Management System Objectives

The objectives of the PAS QMS 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

PAS achieves and maintains impartiality by establishing an organizational structure that safeguards impartiality (see 4.1.4.1) and implementing and adhering to the policies and processes of the QMS outlined in this manual, which are based on industry accepted standards and methodologies.

PAS 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 QMS uncertain.

An opportunity is something that has potential positive consequences for the organization.

PAS personnel manage risks and opportunities on a daily basis by following policies, procedures and processes that support the QMS. Some ways in which the QMS 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 the 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 QMS.

PAS 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 PAS 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. The PAS lean program 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 PAS management to communicate the QMS to personnel.

To assure personnel understand and implement the quality program outlined in the manual:

- PAS personnel are required to sign a Read and Acknowledgement Statement to confirm the employee has:
 - 1) been informed of the manual by 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 QMS. The “how to” implementation of the QMS is provided in policy, SOPs, standard work instructions, and other instructional documents.
- This manual and supporting policies and procedures are made readily accessible to personnel in the area where the work activity is performed.

4.2.2 Quality Policy Statement

The quality policy of PAS is to provide customers with data of known and documented quality fit for their intended purpose. PAS achieves this policy by implementing the QMS 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:

- PAS will provide customers with reliable, consistent, and professional service. This is

accomplished by making sure each PAS location 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.

- PAS maintains a quality program that complies with applicable state, federal, and industry standards for analytical testing and competency.
- PAS management provides training to personnel so that all personnel are familiar with the QMS outlined in this manual and that they understand that implementation of the QMS is achieved by adherence to the Pace® and PAS policies and procedures.
- PAS management continuously evaluates and improves the effectiveness of the QMS 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

Pace® has established a comprehensive ethics and data integrity program that is communicated to all Pace® employees so 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 Pace® Ethics / Data Integrity Program include:

- Ethics Policy (COR-POL-0004);
- Ethics Officer (Chief Compliance 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 Pace® 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 PAS managers and supervisors 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.

Pace® has engaged Lighthouse Services, Inc. to provide personnel with an anonymous reporting process available to them 24 hours per day/7 days per week. The alert line may be used by any employee to report potential violations of the company’s ethics and data integrity program. Reports are forwarded to the Pace® Ethics Compliance Officer to investigate and resolve the matter. Investigations concerning data integrity are kept confidential.

See COR-POL-0001 *Compliance Alertline* for more information.

Posters and flyers with the compliance alert line information must be prominently posted in each PAS location for personnel reference.

Compliance Alert Line Information:

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 QMS is provided by the application of their signature of approval to the template and/or manual. Their signature confirms they understand their responsibility to implement the QMS 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 QMS outlined in this manual, implementing the QMS outlined in this manual, and upholding these requirements for all work activities.

4.2.5 Supporting Procedures

References to processes and procedures that support the QMS are included 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 QMS are classified into document types that identify the purpose of the document and establish how the document is managed and /or controlled.

Examples: Types of PAS Internally Created Documents

Document Type	Purpose
Quality Manual	Outlines the PAS QMS and structure and how it works for a system including policy, goals, objectives and detailed explanation of the system and

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Document Type	Purpose
	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 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. Assures that activities are performed properly in accordance with applicable requirements.
Standard Work Instruction	Provide step by step visual and/or written instruction to perform 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	Assists users in using a particular product; or a technical interpretation of a method or process by which PAS locations must abide.
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: Types of External Documents used by PAS

Certificate	Lists parameters, methods, and matrices for which the location 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 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.

These 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 agree with documents of higher rank.

PAS Document Hierarchy

Rank	Document
1	Corporate Manual
2	Corporate Policy
3	Corporate SOP
4	Corporate SWI, Templates, Guides, Forms, Guidance
5	Local Manual
6	Local SOP
7	Local SWI, Templates, Guide, Forms, Guidance

Information and requirements from project documents are not incorporated into PAS policy or SOPs in order to maintain client confidentiality. These documents are managed as external documents and any requirements for work specified is followed

when work for the project is performed. Project Documents are reviewed and maintained as part of the contract/incoming work review process (see Section 4.4). If the project document is less stringent than the PAS QMS, policies, or SOPs, and/or is less stringent than applicable federal or state requirements, PAS locations are still required to meet the minimum requirements of the PAS QMS and any applicable statutory or federal requirements in addition to the requirements specified in the project document.

Reference documents are not ranked because all PAS created documents, processes and procedures must be consistent with the applicable reference document(s) in addition to higher-ranking PAS documents.

See SOP ENV-SOP-CORQ-0015 *Document Management and Control* for more information.

4.2.6 Roles and Responsibilities

The roles and responsibilities for technical management and the quality manager are provided in section 4.1.5.2.

4.2.7 Change Management

When significant changes to the PAS QMS are planned, these changes are managed by corporate quality personnel to assure that the integrity of the QMS is maintained.

4.3 Document Control

4.3.1 General

PAS procedures for document control are provided in SOP ENV-SOP-CORQ-0015 *Document Management and Control*.

PAS locations use electronic document management software (eDMS) to perform the document control procedures of the SOP. This system provides centralized access to all documents used by PAS locations across the network. All PAS locations are required to use the eDMS system established for PAS (presently Qualtrax) unless an exemption has been granted by the PAS Corporate Quality Director.

eDMS automates the process for unique document identification, version control, approval, access, and archival and 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 each location. The master list minimally 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 QMS are reviewed by qualified personnel and approved by management prior to release for use.

Only the approved versions of documents are available to personnel for use unless use of a draft document is authorized by management.

The managers responsible for authorization of each document is situation specific.

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 documents are reviewed at least every two years to ensure the documents 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 conduct their work activities. Consequently, any concerns or problems with the document should be caught and brought to the attention of 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.

PAS 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 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

PAS management and/or client service personnel perform thorough reviews of requests and contracts for analytical services to verify the location(s) performing the work has the capability, capacity, and resources necessary to successfully meet the customer's needs. These review procedures are described in SOP ENV-SOP-MIN4-0182 *Review of Analytical Requests*.

The procedures in this SOP(s) are established to ensure that:

- The PAS locations performing the work understand 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;
- PAS locations and any external subcontractor(s) have 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;
- Any discrepancies between the PAS QMS, statutory or regulatory requirements and the client request are resolved; 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 PAS locations contracted to perform the work and any internal or external subcontractors hold required certification/accreditation for the test method, matrix, and

analyte and verifies the location 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 locations and any potential subcontractors are able to manage the sample load and deliver work production within the delivery timeframe requested.

Resource review verifies that the location 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 (Internal and External)

The terms 'subcontract' and 'subcontracting' refers to analytical work done by an organization external to Pace[®] (External Subcontracting) or by a Pace[®] location with an address different than the address listed on the cover page of the test report (Internal Subcontracting).

The PAS network offers comprehensive analytical capability and capacity to ensure Pace[®] can meet a diverse range of client needs for any type of project. If a PAS laboratory receives a request for analytical services and it cannot fulfill the project specifications, the location's client services team will collaborate with the client to place the work within the PAS network.

When it is not possible to place the work within network, the location 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 location and the client.

Whenever work is subcontracted, the PAS location responsible for management of the project verifies each of these qualifications:

- The internal or external subcontractor has the proper accreditation/certifications required for the project and these are current; and
- The use of the internal or external subcontractor is approved by the client and/or regulatory agency when such approval is required by the customer. Record of customer approval is retained in the project record.

External subcontractors selected by Pace[®] must be pre-qualified by quality personnel to verify their QMS is similar to Pace[®] and complies with all relevant Standards such, as ISO/IEC 17025 and the TNI Standard(s) and/or federal and state regulatory requirements. The list of approved subcontractors for each location is maintained by local quality personnel. Pre-qualification of a subcontractor does not eliminate the requirement for the PAS location placing work to verify the subcontractor has the certifications, capability, capacity, and resources to perform work on behalf of Pace[®] on a project-specific basis.

For all subcontracted work, the PAS location placing the work internally or externally is responsible to ensure project specifications are always communicated to and understood by the subcontractor.

4.6 Purchasing Services and Supplies

Vendors that provide services and supplies to PAS are qualified to meet the needs of Pace[®]. 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. Evidence of this qualification is the availability to purchase services and supplies from the vendor in the corporate purchasing system.

PAS locations may purchase goods and services from any supplier in the purchasing system.

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 local management who is responsible to ensure the services and supplies procured and received are fit for intended use.

4.7 Customer Service

Project details and management is managed by PAS client services personnel.

4.7.1 Commitment to Meet Customer Expectations

PAS personnel collaborate closely with our customers to ensure their needs are met and to establish their confidence in the capability of PAS to meet their needs for analytical services and expectations for service.

The project manager (PM) is the customer's primary point of contact for each analytical service request (work order). 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. Supervisors are expected to keep the PM informed of project status and any delays or key issues, so that the PM can keep the client informed.

PAS encourages customers to visit our locations to learn more about the capabilities, observe performance and to meet personnel.

PAS customers expect confidentiality. 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 *Code of Ethics and Professional Conduct* for more information on the policy for client confidentiality.

4.7.2 Customer Feedback

PAS actively seeks positive and negative feedback from customers through surveys and direct communication. Information from the client about their experience working with PAS and their satisfaction with work product is used to enhance processes and practices and to improve decision making. Customer feedback is reviewed 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 PAS and used to enhance the QMS.

4.8 Complaints

A complaint is a formal expression of dissatisfaction with the performance of a service or product originating from a party external to Pace[®]. Complaints provide opportunities to improve processes and/or build stronger working relationships with clients.

The PAS complaint resolution process depends on the situation and the nature of the complaint.

Each complaint received is reviewed to determine if it is valid. If the complaint is valid, it is either addressed immediately by the person receiving the complaint or the nature of the complaint is further reviewed and investigated prior to resolution and follow up with the customer.

Complaints (and compliments) are recorded and reviewed during Annual Management Review (see Section 4.15).

4.9 Nonconforming Work

4.9.1 Definition of Nonconforming Work

Nonconforming work is work that does not conform to customer requirements, standard specifications, 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 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 or service center manages 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 location 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 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 SOPs

Departures from an SOP may sometimes be necessary to correct for an error in an SOP or to resolve a complex problem. For example, to mitigate a complex matrix interference.

An authorized departure from a test method SOP is one that has been reviewed and approved by the department leader, however named, of the work area in which the test method is performed. The leader, when authorizing a departure from an SOP,

accepts full responsibility to ensure the departure does not conflict with Pace[®] or PAS policy or procedure, does not affect statutory, regulatory or program compliance and does not adversely affect data integrity or usability.

Departure from administrative or process-oriented SOPs must be approved by the local QM.

Documentation of the reason for the SOP departures must be retained with management approval. Approved departures from test method SOPs should be noted in the final test report to advise the data user.

See SOP ENV-SOP-CORQ-0016 *SOP for SOPs and SWI*, for more information.

4.9.1.2 Authorized Departure from Test Methods (Method Modifications)

When test results are associated to a published reference test method, the location'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, wastewater or a certification or accreditation program, such as TNI/NELAC, the SOP must comply with or include these requirements, or the resulting data and test results cannot be used for regulatory compliance purposes.

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 location may establish an SOP that is modified from these reference method or regulatory program and what is considered a modification are specified in ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification*.

Client requests to deviate from the test method are managed as client requests to depart from the test method SOP since it is the SOP that the location follows when performing work.

4.9.1.3 Stop Work Authority

Stop Work Authority provides PAS personnel with capability to stop work when there is a perceived unsafe condition or behavior that may result in an unwanted event.

All personnel have the authority to request a stop work order when necessary to preserve data integrity or safety of workers.

The need for the stop work order and resolution of the problem must be confirmed by subject matter experts and resumption of work must be approved as follows:

- For stop work orders related to environmental health and safety (EHS) and/or waste management, the decision to stop work may be made by any employee. These decisions must often be made in real-time to protect the safety of the worker. The decision to correct the problem, how, and/or to resume work after stop work has been initiated may be made by the Chief Compliance Officer or the EHS Director, or the deputies assigned to these positions.
- Any employee may recommend a stop work order for concerns related to data integrity. The need to stop work must be reviewed and affirmed by quality

personnel to confirm the concern is valid. The decision to uphold the stop work order must minimally include the local QM, the QPM, and the Corporate Quality Director. The President, the Sr. VPO, the VPO, the Chief Compliance Officer and Chief Technical Officer may also be included in the decision making and resolution process depending on the situation and/or needs for correction to ensure protocols for investigation are followed. Resumption of work after correction may be made by the Corporate Quality Director, or the Quality Program Manager assigned to the location for which the stop work order was issued or by the deputies assigned to these positions.

4.10 Continuous Improvement

The PAS QMS is designed to achieve continuous improvement through the implementation of the quality policy and objectives outlined in this manual. Information about laboratory and service center activities and performance is gained from 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 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.

PAS 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. All activities of 3P and Lean must conform with the requirements of this quality manual and supporting policies and procedures.

4.11 Corrective Action

Corrective action is a process used to eliminate the cause of a detected nonconformity. It is different from a correction. A correction is an action taken to fix an immediate problem but that does not resolve the underlying cause of why the problem occurred. The objective of corrective action 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, is one of the most effective tools used by PAS to prevent nonconforming work, identify risk and opportunity, and improve service to our customers.

PAS has two general processes for corrective action, the application of which process is used depends on the type of nonconformity:

Quality control (QC) exceptions (nonconformance) that occur during routine testing are investigated through troubleshooting and required actions for correction is specified in policies and SOPs. When 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 recurring problem. 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 7 Stage CAPA Process for PAS includes:

- 1) Identification and Containment
- 2) Evaluation
- 3) Investigation
- 4) Cause Analysis
- 5) Action Plan
- 6) Implementation
- 7) Follow Up and Effectiveness Review

PAS procedures for corrective action, are specified in corporate SOP ENV-SOP-CORQ-0018 *Procedure for Corrective and Preventive Action*. Some key concepts and activities related to the PAS corrective action process is provided in the next three subsections.

4.11.1 Cause Analysis (AKA Root Cause Analysis)

Cause analysis is the process of investigation used to identify the underlying cause(s) of the problem. After causal factors are identified, ways to mitigate the causal factors are identified and action(s) most likely to eliminate these factors are taken.

PAS uses different methods to conduct cause analysis. The most common approach is 5-Why, 4M, Fishbone Diagrams, or brainstorming may be appropriate depending on the situation. The method used is case specific and is documented in the CAPA record.

4.11.2 Effectiveness Review

Monitoring corrective actions taken for effectiveness is an essential part of the corrective action process. Effectiveness means the actions taken were appropriate and sustainable. Appropriate means the action(s) taken prevented recurrence of the problem since the time corrective action was taken and sustainable means the actions taken are still in place.

The data from CAPA records are used by PAS 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 cause analysis and investigation of a problem casts doubt on compliance with PAS policies, procedures, or to regulatory requirements; a special audit of the area of activity may be performed as part of the corrective action process. 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 grave issue or risk to the business is identified.

4.12 Preventive Action

Preventive action(s) are actions taken to eliminate the cause of a potential nonconformity before it happens.

Some examples of preventative action include, but are not limited to:

- Routine instrument maintenance (Preventative maintenance)
- Addition of Staff and Equipment

- Professional Development Activities
- Implementation of New Technology

PAS looks for opportunities for preventive action from a variety of sources including employee idea's, customer feedback, business partners input, trend analysis, business analytics, management reviews, proficiency testing results, and risk-benefit analysis.

PAS 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 PAS locations. PAS 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 another permanent form. PAS records document activities and provide evidence of conformity to the requirements established in the QMS. These records may be hardcopy or electronic on any form of media.

4.13.1 General Requirements

4.13.1.1 Procedure

PAS requirements for control of records are specified in corporate policy ENV-POL-CORQ-0013 *Record Management*.

The policy is established to assure quality and technical records are identified, retained, indexed, and filed to allow for retrieval during the entire retention timeframe. 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, 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

Record Type	Includes Records of:
	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 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 location specific SOP for more information. The laboratory's procedure for record management is specified in SOP ENV-SOP-MIN4-0184 *Data and Records Archival*.

4.13.1.3 Security

PAS locations are secure facilities and access to records is restricted to authorized personnel.

4.13.1.4 Electronic Records

The data systems used to store electronic records is backed up in accordance with SOP ENV-SOP-MIN4-0184 *Data and Records Archival*. 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 PAS locations include activities that require the application of a signature. Some 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.

The PAS policy for use and application of electronic signatures is specified in corporate policy ENV-POL-CORQ-0014 *Electronic Signature Policy*.

All employees of PAS 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 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. PAS 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. 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

Quality personnel, or their designees, perform internal systems and technical audits to assess implementation of the QMS, compliance to this manual, policy, and procedures that make up the QMS. Since the processes in this manual are based on the requirements from relevant and applicable Standards for the operation and management of laboratories when operations are assessed against the PAS QMS, compliance with regulatory program requirements and accreditation/certification program requirements are also assessed.

PAS locations are 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 local and corporate management to address deficiencies and to identify opportunities to improve customer service and quality of work, including reliability and usability of data and test results.

Deficiencies, observations, and recommendations from audits are managed by the local QM using the CAPA process. See Section 4.11 for more information.

4.14.1 Internal Audit

The PAS internal audits are conducted to ensure practice matches what we say we do and what we say we do is compliant with the PAS QMS and relevant standards and requirements.

The internal audit program is managed by the local QM who prepares an audit plan at the beginning of each calendar year. The schedule is prepared to assure that all work areas are reviewed over the course of the year and test methods are audited every two years, unless a more frequent test method audit is required by program. Conformance to the schedule is monitored on a monthly basis.

PAS management is responsible to ensure the audit schedule is maintained. PAS supervisors are expected to cooperate with the quality personnel to provide them with complete access to the work area, personnel, and records needed to conduct the audit.

Internal audits may be performed by non-quality personnel when the auditor is approved by the local QM. Non-quality personnel may not audit their own work activities unless it can be demonstrated that an effective and objective audit will be conducted. The person conducting the audit should be trained, qualified, and familiar enough with the objectives and policies of the PAS QMS and knowledgeable with process and test method SOPs related to the activities audited. The auditors should be trained in auditing practices in order to perform a thorough and effective evaluation.

Test method audits include reviews of test reports to verify the product is consistent with customer/project requirements, the work was conducted 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 as needed 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 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 the location or work area audited.

When observations and findings from any audit (internal or external) cast doubt on the validity of testing results, the location takes immediate action to investigate the problem and take corrective action (also see 4.11 and 4.16).

4.14.1.1 Corporate Compliance Audit

PAS locations may also be audited by corporate personnel at discretion. The purpose of the corporate compliance audit is to assess whether the location's practices, processes and procedures conform with the PAS QMS and to identify risk and opportunity.

4.15 Management Review

Local management conducts an annual business review of each location under their purview to assess performance and to establish goals, objectives, and action plans for the upcoming year.

The procedure used to conduct this review is specified in corporate SOP ENV-SOP-CORQ-0005 *Management Review*.

At a minimum, the following topics are reviewed and discussed during annual management review:

- Changes in internal and external issues relevant to the location;
- Fulfillment of objectives and initiatives;
- Suitability of policies and procedures, including EHS and waste management;
- Status of actions from previous performance reviews;
- 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;
- Adequacy of resources;
- Results of risk identification;

- Proficiency testing performance and other measures related to the assurance of validity of test results; other relevant factors, such as QC trends and training status.

The discussion and results of this review are documented in a report prepared by local 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 location has excelled.

4.16 Data Integrity

PAS 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. PAS locations must follow any program or project specific client notification requirements for notification, when applicable.

5.0 TECHNICAL REQUIREMENTS

5.1 General

Multiple factors contribute to the correctness and reliability of the technical work performed by PAS. These factors fall under these broad 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, PAS 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 PAS 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 location to communicate to personnel the duties, responsibilities, and authorities of their position. Qualification records may include but are not limited to diploma, transcripts, and curriculum vitae (CV).

The term “personnel” refers to individuals employed by PAS directly as full-time, part-time, or temporary, and individuals employed by PAS 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 PAS 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 location 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.

PAS 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 the required training specified in Section 5.2.2 and documentation of training is complete.

5.2.2 Training (Required)

Pace[®] training requirements are outlined in Pace[®] policies COR-POL-0023 *Mandatory Training Policy* and COR-POL-0004 *Code of Ethics and Professional Conduct*.

5.2.2.1 Required Training Requirements

The PAS training program includes these elements:

- Scheduling
- Execution
- Documentation and Tracking
- Evaluation of Effectiveness

Required training is scheduled by corporate training personnel, local quality personnel, and the employee's direct supervisor.

Training on required topics, processes and procedure 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. The supervisor and employee are responsible to make sure the employee's training status and training records for all required training is current, complete, and documentation of training is available.

Training status is tracked by the local QM, who provides the status to local management at least monthly or more frequently, as necessary, to ensure required training for personnel is complete and up to date.

The following subsections further describe the required PAS training program for new hire training and on-going training.

The laboratory's procedure for training is specified in SOP ENV-SOP-MIN4-0165 *Orientation and Training Procedures*.

5.2.2.1.1 New Hire Required 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 training on 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 prepare and 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 without direct supervision of the work activity by the supervisor or a qualified trainer.

All required training must be documented and verified complete by the local QM before the employee is authorized to work independently on client samples. Until then, the employee's direct supervisor is responsible for all work produced by the new employee under their supervision.

5.2.2.1.2 On-Going Required 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
- Changes to Policies & SOPs, relevant to their job activities.
- New Policies & SOPS, relevant to their job activities.
- Technical personnel 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).

All required training must be documented and verified complete by the local QM with training records readily accessible in accordance with the corporate policy for Record Management (ENV-POL-CORQ-0013).

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 PAS data integrity procedures will result in a detailed investigation that could lead to profound consequences including immediate termination, debarment, or civil/criminal prosecution.

Completion of data integrity training is documented using the mechanism established by Pace® 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;

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- 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 hire and/or during annual refresher training or as specified in the ethics policy. This document clearly identifies inappropriate and questionable behavior. Violations of this document result in profound consequences, including prosecution and termination, if necessary.

Also see SOP-ENV-COR-POL-0004 *Code of Ethics and Professional Conduct* for more information.

5.2.2.1.4 Management System Documents Training

The Quality Manual policies, and SOPs are the documents used by regulatory bodies and Pace[®] customers to verify 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 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 (IDOC) Requirements

An initial demonstration of capability (IDOC) must be completed and validated prior to authorization for the employee to work independently on client samples for the test method. 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.

Successful DOC is one where the DOC replicate data has been compiled, reviewed, and verified by the employee's supervisor and/or manager to be complete and to have met acceptance criteria and the DOC record has been validated by quality personnel for completeness and compliance, and placed in the employee's training file for accessibility and reference.

Demonstration of capability (DOC) procedures and requirements vary by technology.

For example, a DOC for chemistry test methods where spiking is appropriate, 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.

DOC procedures and requirements must be specified in the laboratory's test method SOP or a stand-alone SOP. Refer to these SOPs for more information.

5.2.2.1.6 Effectiveness of Training

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 QMS, 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.

5.2.2.2 Supplemental Learning

Supplemental learning objectives may be 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 the PAS 3P program and managed by the employee's direct supervisor.

Pace® also offers a wide variety of supplemental learning courses that are made available to all employees for professional development. These learning materials,

maintained by Pace[®] 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 considered part of the required PAS QMS training program.

5.2.3 Personnel Supervision

Every employee is assigned a direct supervisor, however named, who is responsible for their supervision.

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 conducted in accordance with this quality manual, policies, SOPs, and other documents that support the QMS.

5.2.4 Job Descriptions

Job Descriptions that define the required education, qualifications, experience, skills, roles and responsibilities, and reporting relationships for each Pace[®] position are established by top management and kept by corporate HR. The job descriptions apply to employees who are directly employed by Pace[®], part-time, temporary, technical, and administrative and by those that are under contract with Pace[®] 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

Technical personnel are authorized by local quality personnel to perform the technical aspects of their position after quality personnel have 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 location where the employee works.

5.3 Accommodations and Facilities

5.3.1 Facilities

PAS laboratories and service centers are designed to support the correct performance of procedures and to not adversely affect measurement integrity or safety. Access to PAS facilities is controlled by various measures, such as card access, locked doors, staffed main entry.

5.3.2 Environmental Conditions

Each location is equipped with energy sources, lighting, heating, and ventilation necessary to facilitate proper performance of calibrations and tests. The location 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. Operations are stopped if it is discovered that the environmental conditions would jeopardize the integrity of analytical results or other work product.

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 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).

Samples known or suspected to contain high concentration of analytes are separated 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 applicable SOPs.

5.3.4 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 potential safety concerns.

5.3.5 Good Housekeeping

PAS locations must maintain good housekeeping practices in work areas to maintain a standard of cleanliness necessary for analytical integrity and personnel health and safety.

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 items tested, for the intended use of the data, and to conform with applicable federal, statutory, or program requirements.

The test methods offered by PAS are industry accepted methods published by international, regional, or national standards. Each PAS 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 PAS Developed Methods

A PAS 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.

PAS 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 PAS 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 confirmation 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 explain some concepts as they are applied to chemistry. The applications of these same concepts may differ for other technologies such as microbiology, radiochemistry, whole effluent toxicity (WET), and asbestos or other

validation concepts may apply to these disciplines. Refer to the laboratory's test method SOPs for more information.

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 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.

The laboratory's procedure for LOD determination is specified in SOP ENV-SOP-MIN4-0163 *Determination of LOQ and LOQ*.

For chemistry methodology, the local SOP must comply with the current version of each of the following documents:

- EPA document EPA-821-R-16-006 Definition and Procedure for the Determination of the Method Detection Limit;
- 2016 TNI Standard V1M4; and
- TNI GUID-3-109-Rev. 0, V1M4 2016 Standard Update Guidance on Detection and Quantitation.

5.4.5.3.4 Limits of Quantitation (LOQ) and Reporting Limit (RL)

This section describes these concepts for chemistry. For non-chemistry technologies, such as microbiology, refer to laboratory SOPs.

The LOQ is the minimum level, concentration, or quantity of a target analyte that can be reported with a specified degree of confidence.

The LLOQ is the value of the lowest calibration standard included in the calibration curve. The LLOQ establishes the lower limit of quantitation; it is not the same concept as the LOQ, however, the LOQ and LLOQ may be the same value.

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 LOQ determination must be specified in the same SOP for LOD determination, (see Section 5.4.5.3.3) The LLOQ for each method must be specified in the test method SOP.

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, inorganic test methods sometimes 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.

5.4.6 Measurement Uncertainty

The location provides an estimate of uncertainty in testing measurements with analytical results on request, or when required. For example, for radiochemistry uncertainty is always reported with the test result

For chemistry methodologies, the uncertainty of the test method is reflected in the control limits used to evaluate QC performance for the test method (see 5.9.1.1.9). ISO/IEC states that 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 location 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 considered.

5.4.7 Control of Data

PAS 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 PAS 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, PAS 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 routine use. Commercial off the shelf software is considered sufficiently validated when the location 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 group that performed the validation.

The PAS process for the protection of data stored in electronic systems includes:

- Individual usernames 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
- 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.

5.5 Equipment

5.5.1 Availability of Equipment

Each PAS location 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.

When a regulation, program, or reference test method requires Class A glassware for quantitative measurements, only Class A glassware may be used. Plastic graduated cylinders, even if marketed by the vendor as comparable to Class A glassware, may not be used when Class A glassware is specified because ASTM's definition and tolerances for Class A glass cannot be applied to other materials.

5.5.2 Calibration

Equipment and instrumentation are checked prior to use to verify it performs within tolerance for its intended application.

5.5.2.1 Support Equipment

The location confirms support equipment is in proper working order, uniquely identified, and meets the specifications for use prior to placement in service. Periodic checks are performed to verify tolerance and accuracy are performed thereafter in accordance with a support equipment maintenance scheduled maintained by local quality personnel. 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 conduct and record these checks are outlined in SOP ENV-SOP-MIN4-0161 *Support Equipment*.

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 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

Each piece of equipment must be uniquely identified by serial number or any other unique ID system. The identifier is included in the equipment list maintained by the quality department and may not be reused or used interchangeably. New equipment and replacement equipment must be assigned a new unique ID.

5.5.5 Equipment Lists and Records

5.5.5.1 Equipment List

Each PAS location maintains a 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 work area where the equipment is used. 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 location 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 SOP ENV-SOP-MIN4-0091 *Preventive, Routine, and Non-Routine Maintenance* 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 PAS personnel or by outside service providers.

All maintenance activities performed by PAS 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 service must be maintained and accessible for easy retrieval. The location must provide personnel with unrestricted access to instrument maintenance logs in order to promote good instrument maintenance and recordkeeping practices.

If an instrument must be moved, the location 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 because it no longer meets tolerance specifications, the potential effect of the nonconformance may have had on previously reported analytical results should be evaluated (see Section 4.9).

5.5.7 Calibration Status

The location 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 SOP ENV-SOP-MIN4-0161 *Support Equipment*.

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 for service, the location using the equipment ensures that the function and calibration status of the equipment is checked and shown to be satisfactory before the equipment is returned to service.

5.5.9 Intermediate Equipment Checks

The location 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 SOP ENV-SOP-MIN4-0161 *Support Equipment* for more information.

5.5.10 Safeguarding Equipment Integrity

The location 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 location 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 location 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 PAS personnel will assure that results in computer software are also updated.

5.6.3 Specific Requirements

5.6.3.1 Requirements for Calibration Laboratories

The laboratory does not offer calibration services to customers; therefore, ISO/IEC and TNI requirements for calibration laboratories do not apply.

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 weights are the weight(s) used for daily balance calibration checks and the working thermometers are used for daily temperature measurements.

Working weights and thermometers must be periodically checked to verify on-going adequacy for use between calibrations performed by an external calibration laboratory using reference standards traceable to SI or a national standard and that are used solely for verification purposes.

For example:

- An acceptable reference standard to check working thermometers against include a NIST Certified Thermometer or a NIST Traceable Thermometer that is not used for any other purpose than to check the adequacy of the working thermometer.
- An acceptable reference standard for the working weights is a set of Class S weights that is not used for any other purpose than to verify the weights used daily.

The working weights must be checked against the reference standard annually and all weight sets must be recertified by an ISO accredited calibration body every 5 years. In this application, “annually” means within thirteen (13) months from the date of the last check.

Working thermometers must be checked against the reference thermometer prior to placement in service to establish a correction factor (CF)¹ and then re-checked annually (± 13 months from date of last check) or if battery operated, every three (3) months (± 100 days from date of last check).

Exceptions to the 3-month recheck for battery operated sensors are allowed when the sensor is embedded in a unit and the manufacturer/vendor has evidence to show that the accuracy of the sensor is not affected by battery life.

Liquid in Glass NIST Certified reference thermometers must be recertified by an ISO/IEC accredited calibration laboratory every 5 years. If the reference thermometer is NIST Traceable or is a digital NIST Certified thermometer, the reference thermometer must be recertified annually by an ISO/IEC 17025 accredited calibration laboratory or service provider that provides traceability to a national standard.

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 SOP ENV-SOP-MIN4-0161 *Support Equipment* and ENV-SOP-MIN4-0180 *Laboratory Supply Procedures* for more information.

5.6.4.2 Reference Materials

The location 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 SOP ENV-SOP-MIN4-0180 *Laboratory Supply Procedures*.

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 location 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 reference standards and materials must be included in test method 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 location 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 PAS.

5.7.1 Sampling Plans and SOPs

When PAS performs field collection of samples, sampling is carried out in accordance with a written sampling plan and 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 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

PAS 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 location's procedures for sample management and handling are outlined in SOP ENV-SOP-MIN4-0008 *Sample Management*, ENV-SOP-MIN4-0011 *Internal Chain of Custody*, ENV-SOP-MIN4-0095 *Regulated Soil Handling*.

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 location 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 location.

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 location. When the signature field on CoC includes company. Personnel relinquishing and/or receiving samples are expected to record this information. When the COC is transported inside the cooler, independent couriers do not sign the COC and 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 location 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 location when requested by customer or when 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 location 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 SOP ENV-SOP-MIN4-0008 *Sample Management* for more information.

5.8.2 Unique Identification

Each sample is assigned a unique identification number (Lab ID) after the sample has been checked and accepted by PAS in accordance with the PAS 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.

The lab ID is linked to the field ID (client ID) in the receipt and log-in 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 location 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: For chemical testing methods for which thermal preservation is required, temperature on receipt is typically considered acceptable if the measurement is above freezing but $<6^{\circ}\text{C}$ unless otherwise specified by federal, statutory, program or test method requirements. Refer to the location's SOP for sample receipt for specific thermal preservation requirements.

For samples that are hand-delivered to the location 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 location 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

PAS 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 location 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.

Samples that are delivered to the location 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 SOP ENV-SOP-MIN4-0008 *Sample Management*. 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 location procedures for sample storage, handling and disposal are detailed in SOPs ENV-SOP-MIN4-0008 *Sample Management*, ENV-SOP-MIN-0098 *Waste Handling*, ENV-SOP-MIN4-0095 *Regulated Soil Handling*, ENV-SOP-DUL1-0004 *Waste Handling and Management*, and ENV-SOP-VIR1-0007 *Waste Handling and Management*.

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 $\leq 6^{\circ}\text{C}$ (but not frozen) and freezer storage areas are maintained at $< -10^{\circ}\text{C}$, 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 location 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 location for sample retention and disposal are detailed in SOP ENV-SOP-MIN4-0008 *Sample Management*, ENV-SOP-MIN-0098 *Waste Handling*, ENV-SOP-MIN4-0095 *Regulated Soil Handling*, ENV-SOP-DUL1-0004 *Waste Handling and Management*, and ENV-SOP-VIR1-0007 *Waste Handling and Management*.

In general, unused sample volume and prepared samples such as extracts, digestates, distillates and leachates (samples) are retained by the location for the timeframe necessary to protect the interests of the location 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 location 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 location 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 location 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 location 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 location 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. These concepts may not apply to other technologies and disciplines such as microbiology, radiochemistry, whole effluent toxicity, and/or asbestos. For essential QC for these disciplines, refer to test method SOPs.

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 from a different lot from the same vendor, when only one vendor is available. It is a positive control used to verify the accuracy of instrument calibration relative to the purity of the standards used for calibration. This check may be referred to in published test methods and quality system standards as the initial calibration verification (ICV) or a 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. The source of the problem should be investigated and corrected prior to further use of the calibration/instrument for sample analysis.

5.9.1.1.2 Continuing Calibration Verification (CCV)

The CCV is used to determine if the analytical response has significantly changed since 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 and correction. Actions taken are technology and method specific.

5.9.1.1.3 Method Blank (MB) / Other Blanks

The MB 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.

The criteria used to assess for contamination depends on the intended use of data. In general, detections in the MB above the RL or ½ the RL

indicate contamination. 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 a 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)

The MS and MSD are replicates of a client sample that is spiked with known amount of target analyte. Matrix spikes measure the effect the sample matrix has on precision and accuracy of test results.

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, during the same sampling event. Consequently, matrix spikes should be client designated. When there is not a client-specified MS for any sample in the batch, the location 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. However, because the performance of

matrix spikes is matrix-dependent and specific to the customer whose sample was used as the MS/MSD, the results of matrix spikes are not used for quality control on the batch.

5.9.1.1.6 Sample Duplicate (SD)

A sample duplicate is a second replicate of sample that is used to measure precision.

The relative percent difference between replicates are evaluated against the established acceptance 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 location follows specific guidelines for the treatment of internal standard recoveries and further information can be found in the applicable test method 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 location develops acceptance criteria in accordance with recognized industry standards.

Some methods and programs require the location to establish control limits for LCS, MS/MSD, and surrogate evaluation using historical data.

PAS 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 SOP ENV-SOP-MIN4 *Control Chart Generation and Trend Analysis* for more information about the procedures used to establish in-house control limits.

5.9.1.2 Proficiency Testing (PT)

PAS locations participate in interlaboratory proficiency testing (PT) studies to measure performance of the test method and to identify or solve analytical problems. PT samples measure location performance through the analysis of unknown samples provided by an external source.

The frequency of PT participation is based on the certification and accreditation requirements held by the laboratory. The PT samples are obtained from accredited proficiency testing providers (PTP) and treated as field samples which means they are included in the location’s normal analytical processes and do not receive extraordinary attention due to their nature.

PAS locations do not share PT samples with other PAS locations, does not communicate with other PAS locations 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.

PT results scored unacceptable are investigated and correction action taken, when necessary.

Refer to corporate policy ENV-POL-CORQ-0002 *PT Policy* for more information.

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

PAS locations use 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 SOP ENV-SOP-MIN4-0092 *Data Review Process*. 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 PAS 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 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

Test reports may be audited by local quality personnel to verify compliance with SOPs and to check for data integrity, technical accuracy, and compliance with the PAS QMS and any applicable federal, statutory, and program requirements. The reports chosen for the data audits are selected at random and these audits are not usually done prior to issuance of the test report to the customer.

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

PAS does not perform calibration activities for its customers and calibration certificates are

not offered or issued.

5.9.5 Opinions and Interpretations

The location 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 location provide opinion or interpretation to assist them with their decisions about the data.

When opinions and interpretations are included in the test report, the location 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:

- A viewpoint on how a nonconformance impacts the quality of the data or usability of results.
- Recommendations for how the customer should use the test results and information.
- Suggestions or guidance to the customer for improvement.

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 (internal subcontracting) may be merged into a single test report so long as the test report issued clearly identifies the location and address of each network location that performed testing, and which tests each PAS location 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 PAS are designed to accommodate each type of analytical test method performed and to minimize the possibility of misunderstanding or misuse of analytical results. The format of electronic data deliverables (EDD) follows the specifications for the EDD.

5.9.9 Amendments to Test Reports

Test reports that are revised or amended by the location 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 location's data verification and review process.

The procedure for report amendments and revision are outlined in SOP ENV-SOP-MIN4-0185 *Final Report and Deliverable Contents*.

5.10 Reporting

5.10.1 General Requirements

PAS offers a wide variety of test report formats to meet project needs of Pace[®] customers and that comply with federal and state regulatory programs.

The type and level of deliverable, including the electronic data deliverable (EDD) format are established between PAS and the customer during the contracting process. 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

Regardless of deliverable or report requested, every test report issued by the location includes each of the following items:

- a) A Title
- b) The name and address of the location issuing the test report and for each location where testing was performed if different than address of the location issuing the report. When testing is done at multiple PAS locations, the report must clearly identify which PAS location performed each test method;
- c) Unique identification of the test report and on each page an identification number to link each page to the test report, and clear identification of the end of the report.
- d) The name and address of the customer
- e) Identification of test methods used
- f) Cross reference between client sample identification number (Sample ID) and the identification number for the sample (Lab ID) to provide unambiguous identification of samples.
- g) 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.
- h) Date and times of sample collection, receipt, preparation, and analysis.
- i) 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.
- j) All chains of custody (COC) including records of internal transfer between locations within PAS,
- k) Name, title, signature of the person(s) authorizing release of the test report and date of

release.

- l) A statement that the results in the test report relate only to the items tested.
- m) Statement that the test report may not be reproduced except in full without written approval from PAS.

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 lading 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 location 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.

For PAS locations accredited to ISO/IEC 17025:2017:

- Data included in the test report provided by a customer should be clearly identified. The test report should also include a statement that the test results apply only to the samples as received.

5.10.3.2 Test Reports: Sampling Information

The following items are included in the test report when samples are collected by PAS 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 (Version 2):

Section	Description of Change
Header / All	Added registered trademark after Pace as required by branding guidelines
Header	Updated the years associated with the copyright.
Signature Page	Removed Cover Page applied by MasterControl eDMS
Approval Signatory	Changed name of this page to “Management Personnel” and updated Job Titles.
All	Changed references to “laboratory” with PAS or location, where appropriate.
All	Replaced stand-alone acronym “ENV” with “PAS” except where “ENV” is embedded in document control numbers.
All	Corrected spelling, typographical, and format errors.
Various	Added language to clarify the examples in the manual are provided for chemistry, these examples may not apply in the same way to other disciplines such as radiochemistry, microbiology, asbestos, or whole effluent toxicity (WET).
1.0	Corrected Parent Company Information.
1.2	Added definitions for “location,” “laboratory” and “service center” for QMS and compliance purposes.
1.2.1	Updated job titles to match current structure.
1.2.2	Revised language for clarity.
1.2.3	Removed specificity to allow for more options
4.1.4	Updated to describe current scope of organization
4.1.4.1	Updated to describe current organization structure
4.1.5.1.1	Updated to match new organization structure and job titles
4.1.5.2	Updated to match new organization structure and job titles
4.1.5.2.1	Updated to clarify qualifications and meaning of “absent”
4.1.5.3	Updated to clarify impartiality
4.1.5.4	Reorganized section for clarity
4.2.1.1	Added statement that the organization structure is designed to safeguard impartiality
4.2.2.1	Added requirement to post compliance alertline posters in work area.
4.2.1.3	Added requirement for policies and procedures to be available in work area

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Section	Description of Change
	(previously implied but not explicitly stated)
4.2.5.1	Clarified hierarchy and application of project documents
4.5	Updated requirements for internal and external subcontracting
4.8	Updated complaint handling requirements to clarify that only valid complaints are acted on with corrective action.
4.9.1.3	Added roles responsible for authorizing return to work after stop work order.
4.11	Main and subsections updated for clarity
4.14	Main and subsections updated for clarity
5.2.2 Subsections	Content reorganized and language related to documentation of training and authorization of personnel revised to clarify expectations. Requirements of DOCs modified to clarify procedure described in manual pertains to chemistry methodology; other approaches to DOC acceptable for other disciplines such as microbiology, radiochemistry, asbestos, whole effluent toxicity.
5.4.5.3.3	Added reference documents for which the local SOP for LOD must comply with.
5.5	Added language to clarify existing requirements.
5.6.4	Clarified requirements for reference standards for working weights and thermometers and defined meaning of terms “annual” and “quarterly.” Included examples of acceptable reference standards for adequacy checks.
5.8.1	Added recommendation for Pace® personnel to add “Pace®” next to their signature on the CoC when receiving samples since the CoC form has signature/company, implying the company affiliation must be added.
5.10.3.1	Included ISO/IEC 17025:2017 to add disclaimer to test reports (applies to laboratories accredited to ISO/IEC 17025:2017 only).

This document supersedes the following documents:

Document Number	Title	Version
ENV-TMP-CORQ-0007	Quality Manual Template	01
ENV-MAN-CORQ-0001	Quality Manual	00
ENV-MAN-MIN4-0001	Quality Manual	01
ENV-MAN-NW-0001	Quality Manual	02

7.0 APPENDICES

7.1 Appendix A: Certification / Accreditation Listing

Disclaimer: The certifications / accreditation lists provided in this Appendix are those that were held by the PAS 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. This manual is not updated with each change made. Current certificates are accessible via the eDMS Portal for PAS employees. External parties should contact the location for the most current information.

7.1.1 PAS-Minneapolis MN

Authority	ID	Authority	ID
A2LA (Dept. of Defense)	2926.01	Mississippi State Dept. of Health	MN00064
A2LA (IEC/ISO 17025:2017)	2926.01	Missouri Dept. of Natural Resources	10100
A2LA (Wyoming - UST)	2926.01	Montana Dept. of Public Health and Human Services	CERT0092
Alabama Dept. of Environmental Management	40770	Nebraska Dept. of Health and Human Services	NE-OS-18-06
Alaska Dept. of Environmental Conservation (DW)	MN00064	Nevada Dept. of Conservation & Natural Resources	MN00064
Alaska Dept. of Environmental Conservation (Contaminated Sites)	17-009	New Hampshire Dept. of Environmental Services	2081
Arizona Dept. of Health Services	AZ0014	New Jersey Dept. of Environmental Protection	MN002
Arkansas Dept. of Health (DW)	MN00064	New York State Dept. of Health	11647
Arkansas Dept. of Environmental Quality (WW)	88-0680	North Carolina Dept. of Environmental Quality	530
California ELAP via State Water Resources Control Board	2929	North Carolina Dept. of Health and Human Services (DW)	27700
Colorado Dept. of Public Health & Environment	MN00064	North Dakota Dept. of Environmental Quality	R-036
Connecticut Dept. of Public Health	PH-0256	North Dakota Dept. of Environmental Quality (Waste Characterization)	R-036
Florida Dept. of Health	E87605	Ohio Environmental Protection Agency	41244
Georgia Dept. of Natural Resources	959	Ohio Environmental Protection Agency (VAP – 1700)	CL101
Hawaii Dept. of Health	MN00064	Ohio Environmental Protection Agency (VAP – 1800)	CL110
Idaho Dept. of Health and Welfare (Inorganics)	MN00064	Oklahoma Dept. of Environmental Quality	9507
Idaho Dept. of Health and Welfare (Organics)	MN00064	Oregon ELAP via Health Authority (Primary)	MN300001
Illinois Environmental Protection Agency	200011	Oregon ELAP via Health Authority (Secondary)	MN200001
Indiana State Dept. of Health	C-MN-01	Pennsylvania Dept. of Environmental Protection	68-00563

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Authority	ID	Authority	ID
Iowa Dept. of Natural Resources	368	Puerto Rico Dept. of Health	MN00064
Kansas Dept. of Health and Environmental Laboratories	E-10167	South Carolina Dept. of Health and Environmental Control	74003
Kentucky Dept. for Environmental Protection (DW)	KY90062	Tennessee Dept. of Environmental Control	TN02818
Kentucky Dept. for Environmental Protection (WW)	KY90062	Texas Commission on Environmental Quality	T104704192
Louisiana Dept. of Environmental Quality	AI-84596	Utah Dept. of Health	MN00064
Louisiana Dept. of Health (DW)	LA006	Vermont Dept. of Health	VT-027053137
Maine Dept. of Health and Human Resources	MN00064	Virginia Dept. of General Services	460163
Maryland Dept. of the Environment	322	Washington Dept. of Ecology	C486
Michigan Dept. of Environmental, Great Lakes, and Energy	9909	West Virginia Dept. of Environmental Protection	382
Minnesota Dept. of Agriculture	via "Minnesota Dept. of Health ELAP"	West Virginia Dept. of Health & Human Services (DW)	9952 C
Minnesota Dept. of Commerce (Petrofund)	1240	Wisconsin Dept. of Natural Resources	999407970
Minnesota Dept. of Health ELAP	027-053-137		

7.1.2 PAS-Duluth MN

Authority	ID	Authority	ID
Alaska Dept. of Environmental Conservation (Contaminated Sites)	21-002	Nevada Dept. of Conservation & Natural Resources	MN00037
Minnesota Dept. of Agriculture	via "Minnesota Dept. of Health ELAP"	North Dakota Dept. of Environmental Quality	R-105
Minnesota Dept. of Commerce (Petrofund)	1240	Wisconsin Dept. of Natural Resources	999446800
Minnesota Dept. of Health ELAP	027-137-152	Wisconsin Dept. of Agriculture, Trade and Consumer Protection	480341

7.1.3 PAS-Virginia MN

Authority	ID	Authority	ID
Minnesota Dept. of Commerce (Petrofund)	1240	Minnesota Dept. of Health ELAP	027-137-445

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 location for the most current information.

Table Legend:

- Air = Air

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- 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-Minneapolis MN

Parameter	Method	Matrices							
		Air	DW	NPW	SCM	Waste	Tissue	Wipes	Filters
1,2-Dibromo-3-chloropropane	EPA 8011			X					
1,2-Dibromomethane	EPA 8011			X					
Alaska Diesel Range Organics	AK102 DRO				X				
Alaska Diesel Range Organics	AK102 DRO-SV			X					
Alaska Gasoline Range Organics	AK101 GRO-MS			X	X				
Alaska Residual Range Organics	AK103 RRO			X ¹	X				
Alaska Residual Range Organics	AK103 RRO				X				
Alkalinity	SM 2320 B-2011		X	X					
Amenable Cyanide	SM 4500-CN ⁻ G-2011		X ¹	X					
Ammonia	EPA 350.1			X					
Apparent Specific Gravity	ASTM D5057-2010			X ¹	X ¹				
Chemical Oxygen Demand	EPA 410.4			X					
Chemical Oxygen Demand	SM 5220 D-2011			X					
Conductivity	EPA 120.1			X					
Conductivity	SM 2510 B-2011		X	X					
Demand (BOD, cBOD)	HACH 10360 Rev 1.2 (2011)			X					
Diesel Range Organics	EPA 8015C			X	X				
Diesel Range Organics	EPA 8015D			X	X				
Diesel Range Organics	NwTPH-Dx			X	X				
Diesel Range Organics	WI(95) DRO			X	X				
Dioxins and Furans	EPA 1613B			X	X		X	X	
Dioxins and Furans	EPA 8280B			X	X				
Dioxins and Furans	EPA 8290			X	X		X	X	
Dioxins and Furans	EPA 8290A			X	X		X	X	
Dioxins and Furans	EPA Method 23	X							

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Parameter	Method	Matrices							
		Air	DW	NPW	SCM	Waste	Tissue	Wipes	Filters
Dioxins and Furans	EPA TO-9A	X							
Dioxins and Furans (2,3,7,8-TCDD)	EPA 1613B		X						
Dissolved Oxygen	HACH 10360 Rev 1.2 (2011)			X					
Escherichia coli	SM 9223 B (Colilert®)-2004		X						
Escherichia coli	SM 9223 B-2004			X					
Fecal Coliforms	SM 9222 D (m-FC)-2006			X					
Ferrous Iron	SM 3500-Fe B-2011			X ¹					
Fixed Gases	EPA RSK-175 (GC/FID)			X					
Gasoline Range Organics	EPA 8015C			X	X				
Gasoline Range Organics	NwTPH-Gx			X	X				
Gasoline Range Organics	WI (95) GRO			X	X				
Hexavalent Chromium	SM 3500-Cr B-2011			X					
ICP Metals	EPA 200.7			X					
ICP Metals	EPA 6010C			X	X	X			
ICP Metals	EPA 6010C-SPLP				X	X			
ICP Metals	EPA 6010C-TCLP			X	X	X			
ICP Metals	EPA 6010D (Rev 2018)			X	X	X			
ICP Metals	EPA 6010D (Rev 2018)-SPLP				X	X			
ICP Metals	EPA 6010D (Rev 2018)-TCLP			X	X	X			
ICPMS Metals	EPA 200.8		X	X					
ICPMS Metals	EPA 6020A			X	X	X			
ICPMS Metals	EPA 6020A-SPLP			X	X	X			
ICPMS Metals	EPA 6020B (Rev 2014)			X	X	X			
ICPMS Metals	EPA 6020B (Rev 2014)-SPLP			X	X	X			
Inorganic Anions	EPA 300.0		X	X					
Inorganic Anions	EPA 9056A			X					
Lead in Ambient Air	EPA 6010C	X ¹							
Lead in Ambient Air	EPA 6010C	X ¹							
Mercury	EPA 245.1		X	X					
Mercury	EPA 6020A				X	X			
Mercury	EPA 6020B (Rev 2014)				X	X			

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Parameter	Method	Matrices							
		Air	DW	NPW	SCM	Waste	Tissue	Wipes	Filters
Mercury	EPA 7470A			X					
Mercury	EPA 7470A-SPLP			X					
Mercury	EPA 7470A-TCLP			X					
Mercury	EPA 7471A				X	X			
Mercury	EPA 7471B				X	X			
Moisture (Dry Weight)	ASTM D2974-07				X ¹				
Nitrate + Nitrite	EPA 300.0		X						
Nitrate + Nitrite	EPA 353.2		X ¹	X					
Oil & Grease	EPA 1664B			X					
Oil & Grease	EPA 9071B				X				
Orthophosphate	SM 4500-P G-2011			X					
Paint Filter Liquids Test	EPA 9095B			X					
PCB Congeners	EPA 1668A			X	X	X	X	X	
PCB Congeners	EPA 1668C			X	X	X	X	X	
Per- and Polyfluoroalkyl Substances (PFAS)	EPA 537.1		X						
Per- and Polyfluoroalkyl Substances (PFAS)	Isotope Dilution per DoD QSM v5.3			X	X	X	X		
Per- and Polyfluoroalkyl Substances (PFAS)	MPCA Guidance PFAS			X	X	X			
Per- and Polyfluoroalkyl Substances (PFAS)	WIDNR			X ¹	X ¹	X ¹	X ¹		
Pesticides	EPA 8081A			X	X				
Pesticides	EPA 8081B			X	X				
pH	EPA 9045D			X					
pH	SM 4500-H+ B-2011		X	X					
PM-10	EPA Quality Assurance Handbook, Volume II, Part II								X ¹
Polybrominated Diphenyl Ethers	EPA 1614						X ¹		
Polychlorinated Biphenyls	EPA 8082			X	X	X		X	
Polychlorinated Biphenyls	EPA 8082A (Rev 2007)			X	X	X	X	X	
Polychlorinated Biphenyls	EPA 8082A (Rev 2007)-SPLP				X	X			
Reformed Gases	ASTM D1946-90 (Rev 2006)	X ¹							
Sample Appearance	SM 2110-2005		X ¹	X ¹					
Semi-Volatile Organic Compounds	EPA 625.1-RV			X					

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Parameter	Method	Matrices							
		Air	DW	NPW	SCM	Waste	Tissue	Wipes	Filters
Semi-Volatile Organic Compounds	EPA 8081A-TCLP			X	X				
Semi-Volatile Organic Compounds	EPA 8081B-TCLP			X	X				
Semi-Volatile Organic Compounds	EPA 8270D (Rev 2014)			X	X				
Semi-Volatile Organic Compounds	EPA 8270D (Rev 2014) SIM			X	X				
Semi-Volatile Organic Compounds	EPA 8270D (Rev 2014) SIM-LV			X					
Semi-Volatile Organic Compounds	EPA 8270D (Rev 2014) SIM-SPLP			X	X	X			
Semi-Volatile Organic Compounds	EPA 8270D (Rev 2014)-TCLP			X					
Semi-Volatile Organic Compounds	EPA 8270D WRV			X					
Semi-Volatile Organic Compounds	EPA 8270E			X	X	X			
Semi-Volatile Organic Compounds	EPA 8270E SIM			X	X	X			
Semi-Volatile Organic Compounds	EPA 8270E SIM-LV			X					
Semi-Volatile Organic Compounds	EPA 8270E WRV			X					
Semi-Volatile Organic Compounds	EPA 8270E-TCLP			X	X				
Sodium Absorption Ratio by Calculation	USDA Handbook No. 60			X ¹					
Total Coliforms	SM 9222 B-2006			X					
Total Coliforms	SM 9223 B (Colilert®)-2004		X						
Total Cyanide	SM 4500-CN ⁻ E-2011		X	X					
Total Dissolved Solids (TDS)	SM 2540 C-1997		X						
Total Dissolved Solids (TDS)	SM 2540 C-2011			X					
Total Hardness as CaCO ₃	SM 2340 B-2011			X					
Total Petroleum Hydrocarbon (TPH)	EPA 1664B (SGT-HEM)			X					
Total Petroleum Hydrocarbon (TPH)	EPA 9071B				X				
Total Phosphorus	SM 4500-P F-2011			X					
Total Settleable Solids	SM 2540 F-2011			X					
Total Suspended Particulates (TSP)	EPA Quality Assurance Handbook, Volume II, Part II								X ¹
Total Suspended Solids (TSS)	SM 2540 D-2011			X					
Total Volatile Solids (TVS)	EPA 160.4			X					
Turbidity	EPA 180.1, Rev 2-1993		X	X					
Volatile Organic Compounds	EPA 3C	X							
Volatile Organic Compounds	EPA 624			X					

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Parameter	Method	Matrices							
		Air	DW	NPW	SCM	Waste	Tissue	Wipes	Filters
Volatile Organic Compounds	EPA 624.1			X					
Volatile Organic Compounds	EPA 8260D			X	X	X			
Volatile Organic Compounds	EPA 8260D SIM			X					
Volatile Organic Compounds	EPA 8260D-TCLP			X	X				
Volatile Organic Compounds	EPA TO-15	X							
Volatile Organic Compounds	EPA TO-15 SIM	X							
Volatile Organic Compounds	EPA TO-15 SIM Scan	X							
Volatile Organic Compounds	EPA TO-3	X							

¹ = Laboratory does not hold TNI Accreditation for this test method.

7.2.2 PAS-Duluth MN

Parameter	Method	Matrices							
		Air	DW	NPW	SCM	Waste	Tissue	Wipes	Filters
Acidity, as CaCO ₃	SM 2310 B-2011			X					
Alkalinity	SM 2320 B-2011		X	X					
Amenable Cyanide	SM 4500-CN ⁻ G-2011			X					
Ammonia	EPA 350.1 1993			X	X				
Biochemical Oxygen Demand (BOD)	Hach 10360			X					
Carbonaceous Biochemical Oxygen Demand (CBOD)	Hach 10360			X					
Chlorophyll-A	SM 10200 H			X ¹					
Chromium VI	SM 3500-Cr B-2011			X					
Color	SM 2120 B-2011			X					
Conductivity	SM 2510 B		X	X					
Dissolved Organic Carbon (DOC)	SM 5310 C-2011			X					
Dissolved Oxygen	Hach 10360			X					
Escherichia coli (E.coli)	Colisure®		X						
Escherichia coli (E.coli)	SM 9223 B (Colilert®-18)		X						
Escherichia coli (E.coli)	SM 9223 B (Colilert-18 Quanti-Tray)-2004		X	X					
Fecal coliforms	Colilert®-18 (Fecal Coliforms)			X					
Heterotrophic plate count (HPC)	SimPlate®		X						
Humidity Cell Testing	ASTM D5744-13				X				
Inorganic Anions	EPA 300.0		X	X					
Inorganic Anions	EPA 9056A				X				

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Parameter	Method	Matrices							
		Air	DW	NPW	SCM	Waste	Tissue	Wipes	Filters
Kjeldahl nitrogen – total (TKN)	EPA 351.2			X	X				
Mercury (low-level)	EPA 1631E			X	X	X			
Mercury (methyl)	EPA 1630			X ¹	X ¹	X ¹			
Nitrate as N	EPA 353.2		X						
Nitrate as N	EPA 353.2 (calc)			X					
Nitrate-Nitrite as N	EPA 353.2		X	X					
Nitrite	EPA 353.2		X	X					
Nitrogen, Amine	ASTM D2327			X ¹					
Organic Nitrogen	EPA 351.2 Minus EPA 350.1			X	X	X			
Orthophosphate	EPA 365.3			X					
Oxidation-Reduction Potential (ORP)	ASTM 1498			X ¹					
Percent Moisture	ASTM D2974				X ¹				
pH	SM 4500-H+ B 2011			X					
Pheophytin	SM 10200 H			X ¹					
Salinity	SM 2520			X ¹					
Sulfide	SM 4500-S2 ⁻ D-2011			X					
Surfactants - MBAS	SM 5540 C-2011			X					
Total coliforms	Colisure®		X						
Total coliforms	SM 9223 B (Colilert®-18)		X						
Total coliforms	SM 9223 B (Colilert-18 Quanti-Tray)-2004		X						
Total Cyanide	SM 4500-CN ⁻ E-2011			X					
Total Dissolved Solids (TDS)	SM 2540 C-2011		X	X					
Total Nitrogen	EPA 351.2 plus EPA 353.2			X	X	X			
Total Organic Carbon (TOC)	EPA 9060A			X	X				
Total Organic Carbon (TOC)	SM 5310 C-2011		X	X					
Total Phenolics	EPA 420.1			X					
Total Phosphorus	EPA 365.1			X	X	X			
Total Phosphorus	EPA 365.3			X					
Total Residual Chlorine	SM 4500-Cl G-2011		X	X					
Total Solids (TS)	SM 2540 B-2011			X	X	X			
Total Suspended Solids (TSS)	SM 2540 D -2011			X					

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Parameter	Method	Matrices							
		Air	DW	NPW	SCM	Waste	Tissue	Wipes	Filters
Total Suspended Solids (TSS)	USGS I-3765-85			X		X			
Total Volatile Solids (TVS)	EPA 160.4				X				
Total Volatile Suspended Solids (TVSS)	EPA 160.4			X ¹					
Turbidity	EPA 180.1		X	X					

¹ = Laboratory does not hold TNI Accreditation for this test method.

7.2.3 PAS-Virginia MN

Parameter	Method	Matrices							
		Air	DW	NPW	SCM	Waste	Tissue	Wipes	Filters
Escherichia coli (E.coli)	Colisure®		X						
Escherichia coli (E.coli)	SM 9223 B (Colilert® Quanti-Tray®)-97		X	X					
Escherichia coli (E.coli)	SM 9223 B (Colilert®-18)		X						
Fecal Coliforms	Colilert-18			X					
Heterotrophic plate count (HPC)	SimPlate		X						
Total Coliforms	Colisure®		X						
Total Coliforms	SM 9223 B (Colilert® Quanti-Tray®)-97		X						
Total Coliforms	SM 9223 B (Colilert®-18)		X						

¹ = Laboratory does not hold TNI Accreditation for this test method.

7.3 Appendix C: Glossary

This glossary provides common terms and definitions used by PAS. **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 PAS 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	The continuous improvement program used by PAS that focuses on Process, Productivity, and Performance.
Absence	Inability to perform assigned duties due to lack of physical presence and connectivity.
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.

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Term	Definition
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, <i>Conformity assessment: General requirements for accreditation bodies accrediting conformity assessment bodies</i> . 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.
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 \pm 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.

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Term	Definition
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 timeframe 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.
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.

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Term	Definition
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.
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.

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Term	Definition
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.
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.

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Term	Definition
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.
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: <ul style="list-style-type: none"> • 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.

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Term	Definition
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.
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.

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Term	Definition
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.
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 (C ₆ H ₁₄) 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.

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Term	Definition
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.
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).

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Term	Definition
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.
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.

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Term	Definition
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.
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.

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Term	Definition
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.

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Term	Definition
Quality System Matrix	<p>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:</p> <ul style="list-style-type: none"> • 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 sorbent 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 saltwater 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.

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Term	Definition
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.
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.

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Term	Definition
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.
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 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.

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Term	Definition
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 (INI)	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.
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.

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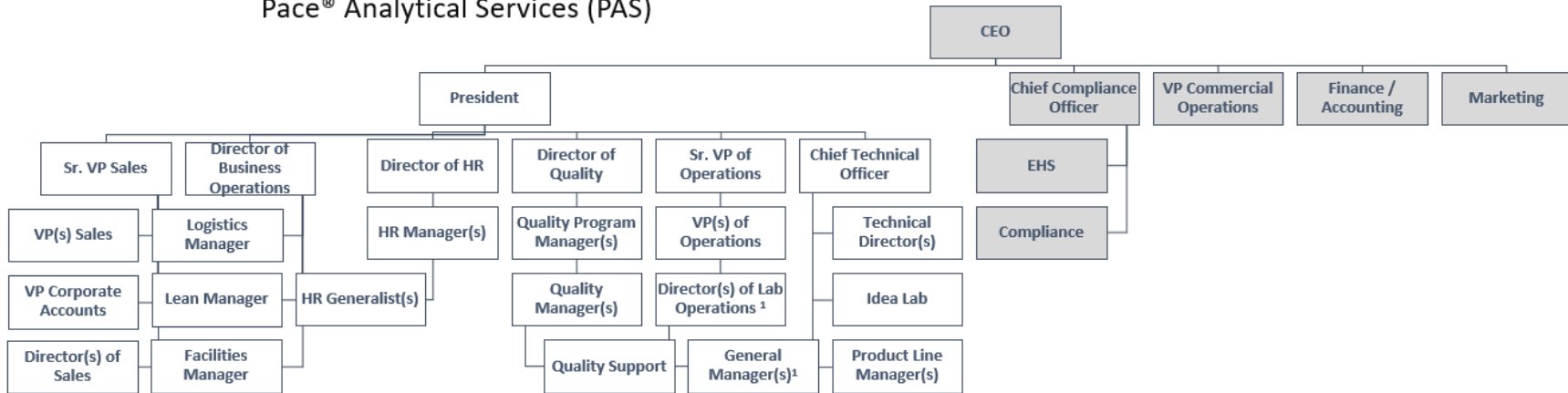
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Term	Definition
Unregulated Contaminant Monitoring Rule (UCMR)	EPA program to monitor unregulated contaminants in drinking water.
Validation	DoD- The confirmation by examination and provision of objective evidence that the particular requirements for 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 PAS Corporate Organization Chart(s)

Organization Structure: Position / Function
 Pace® Analytical Services (PAS)



White Box = PAS Positions / Functions

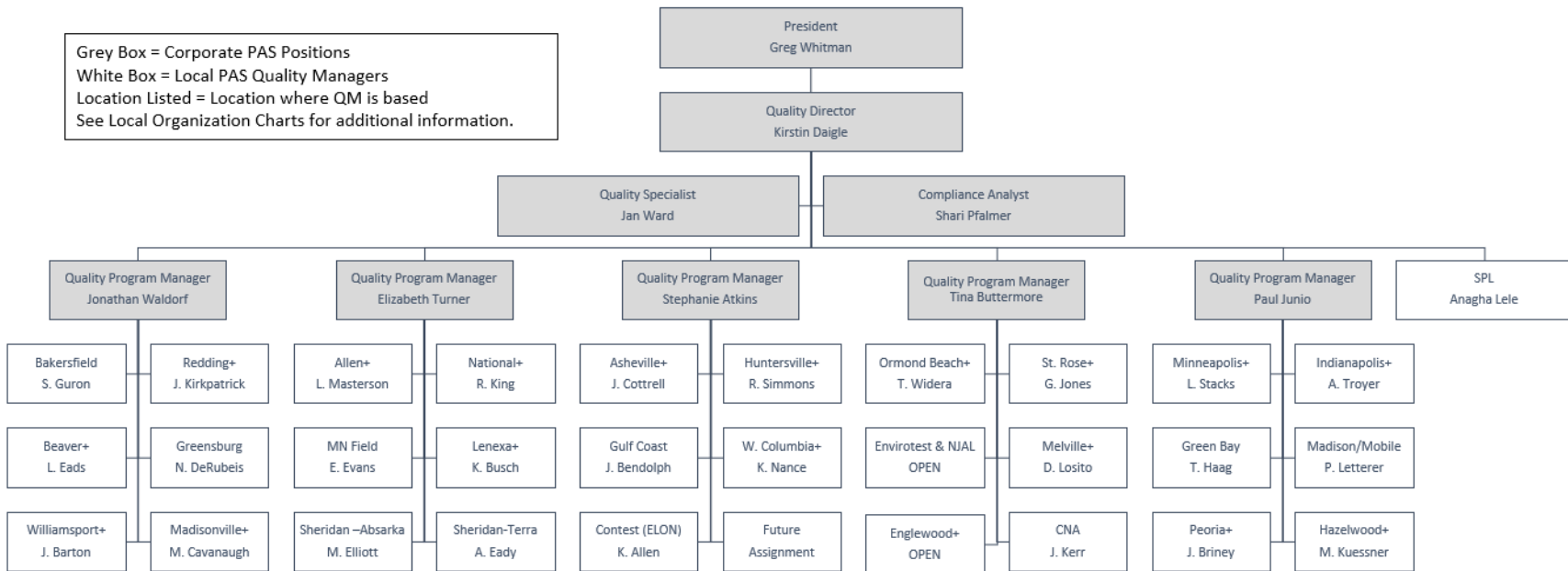
Grey Box = Pace®Corporate Positions / Functions

1= Positions not Assigned to all Locations – see location specific organization charts

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7.4.2 PAS Quality Systems Management Organization Chart

PAS Quality Management Structure



Effective 05.15.22
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7.4.3 PAS-Minneapolis MN – Organization Chart

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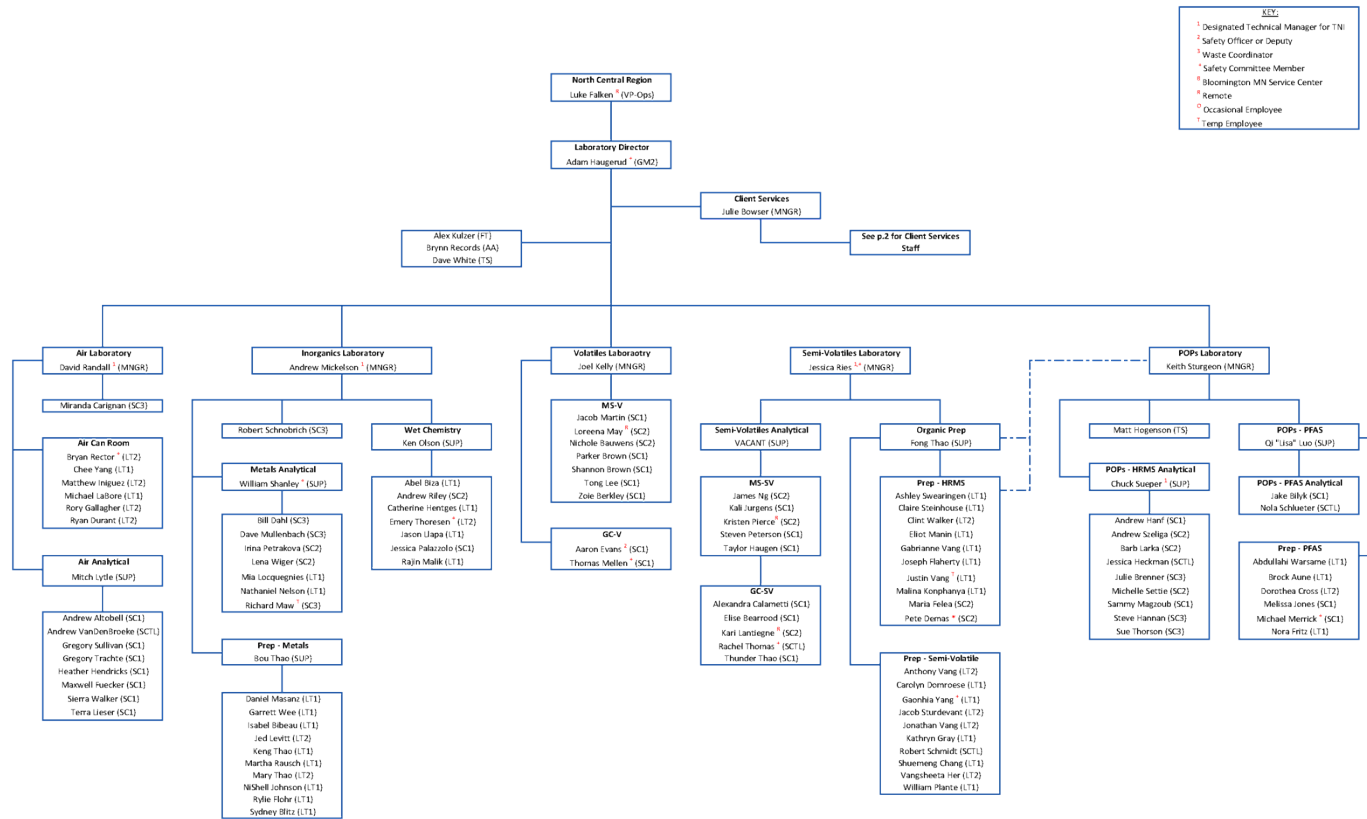
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Pace-Minneapolis MN Laboratory

Last Revised: October 26, 2022



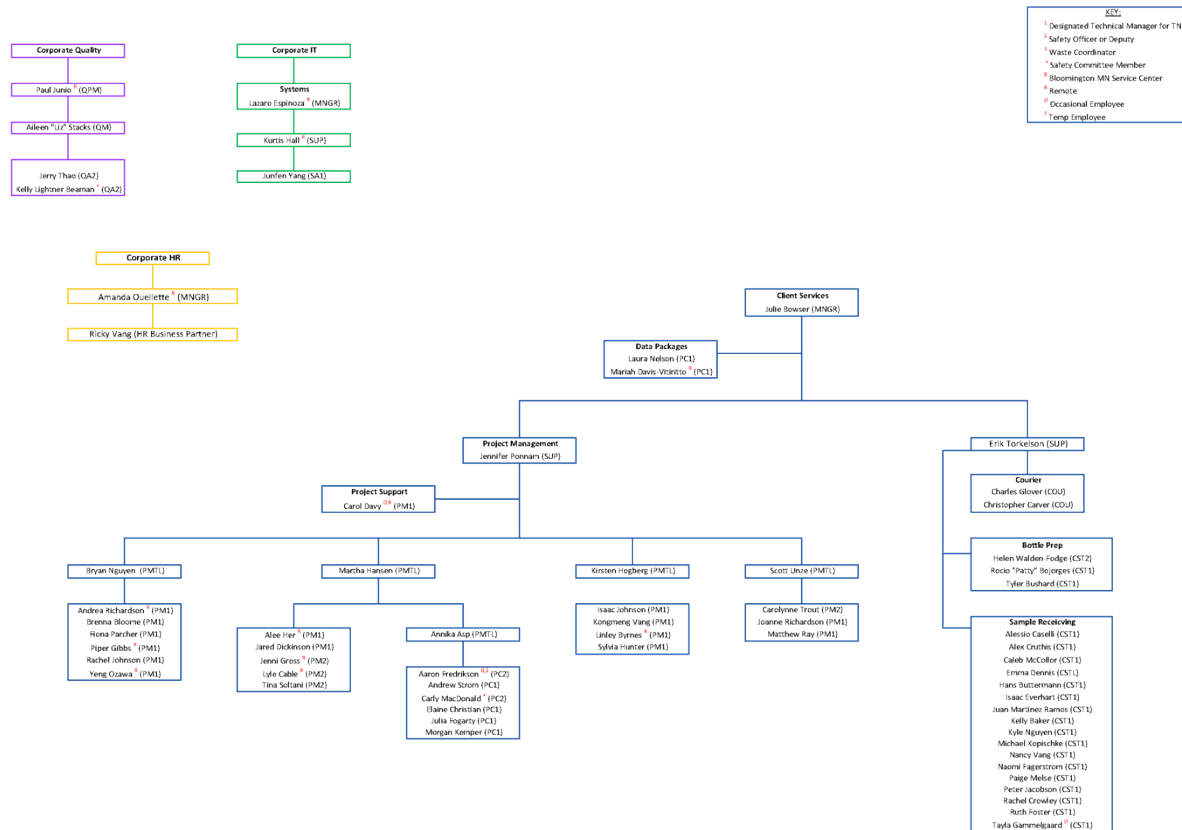
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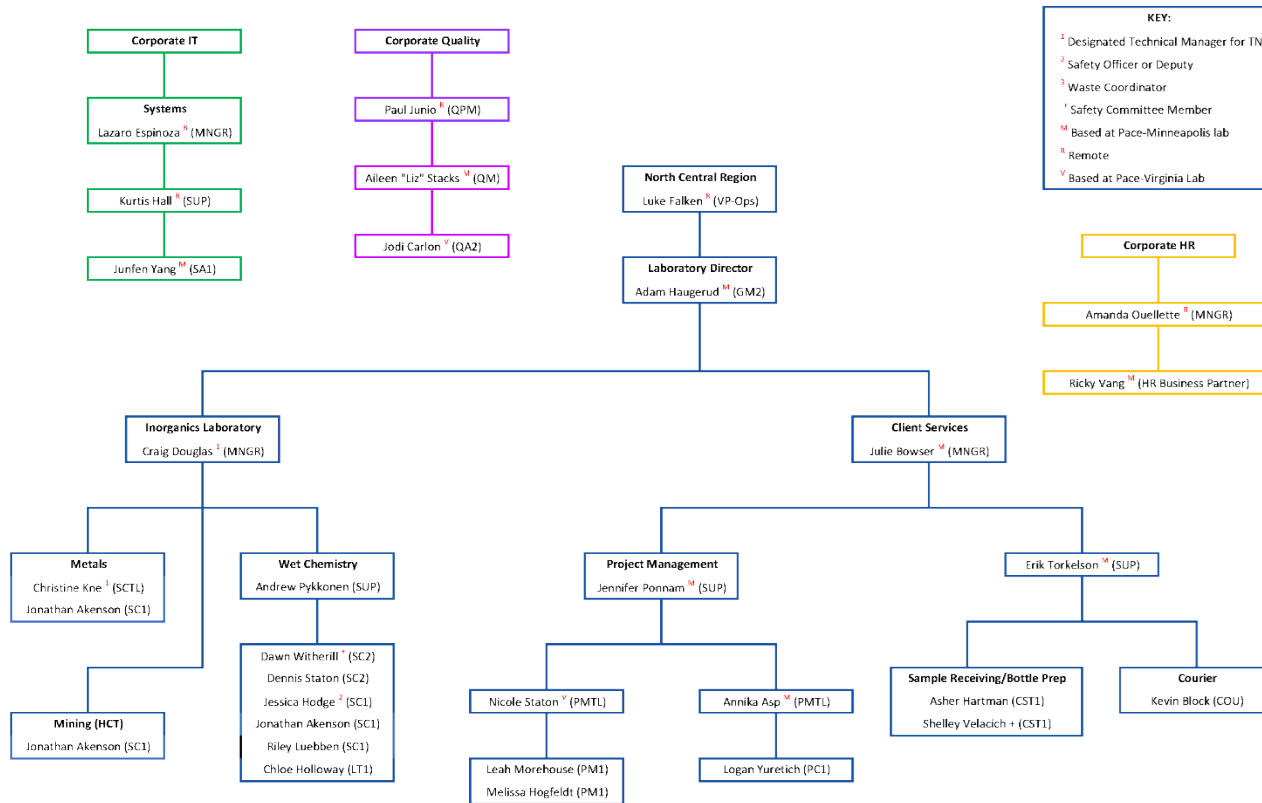
- Key:**
- 1 Designated Technical Manager for TNI
 - 2 Safety Officer or Deputy
 - 3 Waste Coordinator
 - 4 Safety Committee Member
 - 5 Bloomington MN Service Center
 - 6 Remote
 - 7 Occasional Employee
 - 8 Temp Employee

7.4.4 PAS-Duluth MN – Organization Chart



Last Revised: October 17, 2022

Pace-Duluth MN Laboratory

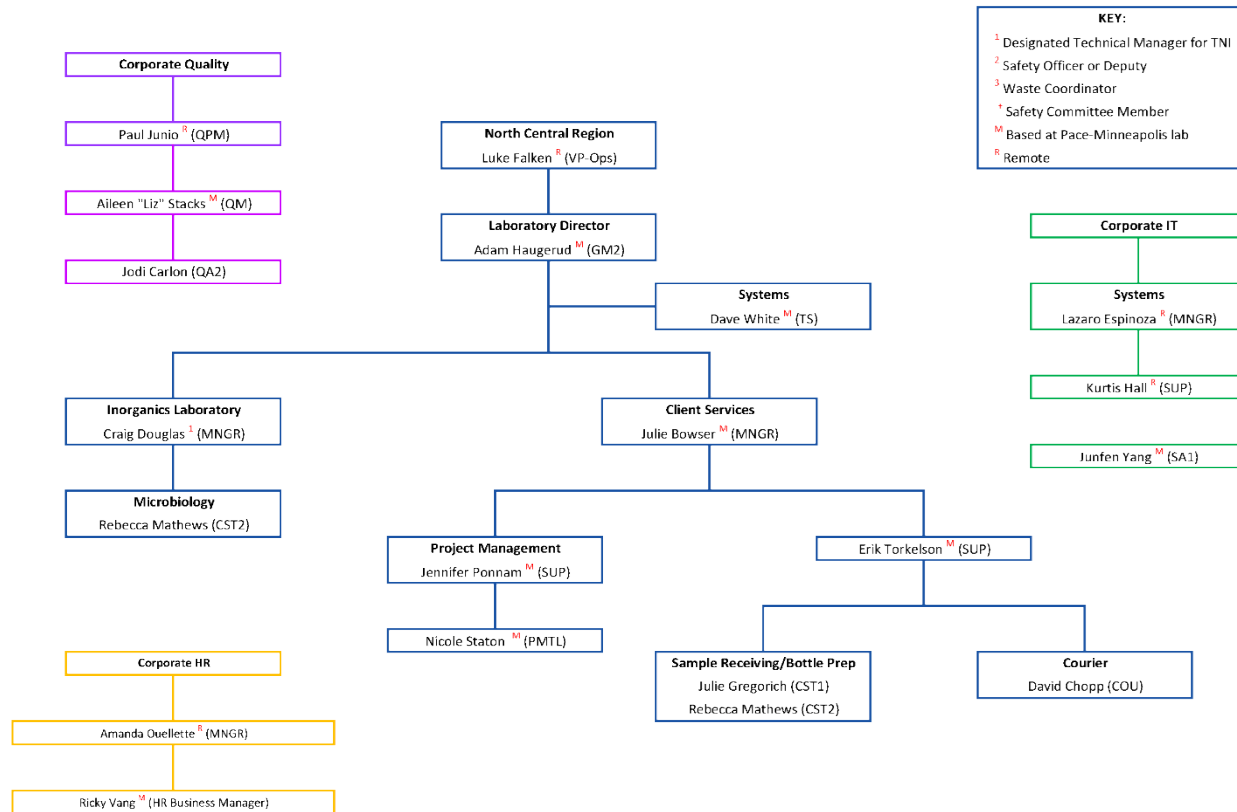


7.4.5 PAS-Virginia MN – Organization Chart



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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-Minneapolis MN

Equipment List: PAS-Minneapolis MN

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
GC	Agilent Technologies	6890N	CN10429060	2005	Unknown	Air	10AIR0	TBD
MS	Agilent Technologies	5973 Network	US43146819	2005	Unknown	Air	10AIR0	TBD
PreConcentrator	Entech Instruments, Inc.	7100A	1299	2017	Unknown	Air	10AIR0	TBD
Canister Autosampler	Entech Instruments, Inc.	7016 CA	1283	Unknown	Unknown	Air	10AIR0	TBD
Canister Autosampler	Entech Instruments, Inc.	7016D	1587	Unknown	Unknown	Air	10AIR0	TBD
GC	HP	5890	2843A20766	1985	Unknown	Air	10AIR5	TBD
GC	Agilent Technologies	6890N	CN10429056	2004	Unknown	Air	10AIR7	TBD
MS	Agilent Technologies	5973 Network	US43146821	2004	Unknown	Air	10AIR7	TBD
PreConcentrator	Entech Instruments, Inc.	7100A	1611	2018	New	Air	10AIR7	TBD
Canister Autosampler	Entech Instruments, Inc.	7016 CA	1239	Unknown	Unknown	Air	10AIR7	TBD
Canister Autosampler	Entech Instruments, Inc.	7016 CA-2	115	2019	Used	Air	10AIR7	TBD
GC	ALS Ready	6890A	US00034289	2013	Unknown	Air	10AIRA	TBD
Concentrator	Entech Instruments, Inc.	7032 AQ-L	1051	2020	Used	Air	10AIRA	TBD
MS	Agilent Technologies	5973 inert	US44621387	2009	Unknown	Air	10AIRB	TBD
GC	Agilent Technologies	6890	CN10517058	2009	Unknown	Air	10AIRB	TBD
PreConcentrator	Entech Instruments, Inc.	7200	1300	2018	New	Air	10AIRB	TBD
Canister Autosampler	Entech Instruments, Inc.	7016D	1488	Unknown	New	Air	10AIRB	TBD
Canister Autosampler	Entech Instruments, Inc.	7016D	1487	Unknown	New	Air	10AIRB	TBD
GC	Agilent Technologies	7890A	CN10742037	2010	New	Air	10AIRD	TBD
MS	Agilent Technologies	5975C	US73317788	2010	New	Air	10AIRD	TBD

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Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
PreConcentrator	Entech Instruments, Inc.	7200	1278	2017	New	Air	10AIRD	TBD
Canister Autosampler	Entech Instruments, Inc.	7016D	1497	Unknown	New	Air	10AIRD	TBD
Canister Autosampler	Entech Instruments, Inc.	7016 CA	1284	Unknown	New	Air	10AIRD	TBD
MS	Agilent Technologies	5975C	US10407503	2010	New	Air	10AIRE	TBD
GC	Agilent Technologies	7890A	CN10241030	2010	New	Air	10AIRE	TBD
Thermal Desorber	Perkin Elmer	Turbomatrix 650	TD650L1009271	2010	New	Air	10AIRE	TBD
Can Cleaning Rack	Pace	N/A	N/A	Unknown	New	Air	Rack 1	TBD
Can Cleaning Rack	Pace	N/A	N/A	Unknown	New	Air	Rack 2	TBD
Can Cleaning Rack	Pace	N/A	N/A	Unknown	New	Air	Rack 3	TBD
Refrigerator/Freezer	Keystone	KSTRC312 AW	DK25BZ	2012	New	Air	A4	TBD
Oven	Despatch	LDB Series	149432	Unknown	Unknown	Air	10AIR10	TBD
Tube Conditioner/Dry Purger	Perkin Elmer	Turbomatrix TC220	820R4051501	2015	New	Air	10AIR24	TBD
GC	Agilent Technologies	6890A	US00040933	2015	Used	Air	10AIRG	TBD
MSD	Agilent Technologies	5973	US10360131	2015	Used	Air	10AIRG	TBD
Thermal Desorber	Perkin Elmer	Turbomatrix 650	TD650L1210081	2015	New	Air	10AIRG	TBD
GC	Agilent Technologies	7890A	CN10803059	2015	New	Air	10AIRH	TBD
MS	Agilent Technologies	5975C	US80848612	2017	New	Air	10AIRH	TBD
Preconcentrator	Entech Instruments, Inc.	7200	1450	2017	New	Air	10AIRH	TBD
Canister Autosampler	Entech Instruments, Inc.	7016D	1586	2019	New	Air	10AIRH	TBD
Canister Autosampler	Entech Instruments, Inc.	7016D	1579	2017	New	Air	10AIRH	TBD
GC	Agilent	6890N	CN10514046	2017	New	Air	10AIRI	TBD
MS	Agilent	5973	US44621373	2018	New	Air	10AIRI	TBD
Preconcentrator	Entech Instruments, Inc.	7200	1623	2018	New	Air	10AIRI	TBD
Canister Autosampler	Entech Instruments, Inc.	7016D	1660	2018	New	Air	10AIRI	TBD
Canister Autosampler	Entech Instruments, Inc.	7016D	1661	2018	New	Air	10AIRI	TBD
GC	Agilent	8890	US1940A006	2019	New	Air	10AIRJ	At the instrument

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Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
MS	Agilent	5977B	US1939R003	2019	New	Air	10AIRJ	At the instrument
PreConcentrator	Entech Instruments, Inc.	7200A	00110	2019	New	Air	10AIRJ	TBD
Autosampler	Entech Instruments, Inc.	7016D	1770	2019	New	Air	10AIRJ	TBD
Autosampler	Entech Instruments, Inc.	7016D	1771	2019		Air	10AIRJ	TBD
GC	Agilent	8890	US2012A006	5/12/2020	new	Air	10AIRK	At the instrument
MS	Agilent	5977B	US2014R005	5/12/2020	new	Air	10AIRK	At the instrument
PreConcentrator	Entech Instruments, Inc.	720D	1799	5/15/2020	new	Air	10AIRK	Electronic copy on PC
Autosampler	Entech Instruments, Inc.	7016D	1818	5/15/2020	new	Air	10AIRK	Electronic copy on PC
Autosampler	Entech Instruments, Inc.	7016D	1819	5/15/2020	new	Air	10AIRK	Electronic copy on PC
GC	Agilent	8890	US2210A011	7/14/1905	new	Air	10AIRM	Electronic copy on PC
MS	Agilent	5977B	US2151R037	7/14/1905	new	Air	10AIRM	Electronic copy on PC
Preconcentrator	Entech Instruments, Inc.	7200A	00141	7/14/1905	new	Air	10AIRM	Electronic copy on PC
Canister Autosampler	Entech Instruments, Inc.	7016D	1995	7/14/1905	new	Air	10AIRM	Electronic copy on PC
Canister Autosampler	Entech Instruments, Inc.	7016D	1996	7/14/1905	new	Air	10AIRM	Electronic copy on PC
Refrigerator	Beverage Air	KR48-1AS	5227060	Braun acquisition	new	Bloomington	Q325	Electronic copy on PC
GC	Agilent	6890	CN10705008	2018	New	HRMS	10MSHR14	TBD
Autosampler-E	CTC	PAL	412290	2018	New	HRMS	10MSHR14	TBD
MS	Waters/Micro mass	Autospec	M590	2018	New	HRMS	10MSHR14	TBD
Freezer	Kenmore	564.285027	80200474	2011	Unknown	HRMS	H2	TBD
Freezer	Dynasty	E-400-C	1206544	2011	Unknown	HRMS	H1	TBD
Fridge	Premium Levella	PFR90DX	3133249	2021	New	HRMS	H3	Online
GC	Agilent	6890N	US10544001	2006	Unknown	HRMS	10MSHR09	TBD
GCMS	Waters/Micro mass	Autospec Premier	P669	2006	Unknown	HRMS	10MSHR09	TBD
Autosampler - P	CTC	PAL	161106	Unknown	Unknown	HRMS	10MSHR09	TBD
GC	Agilent	6890A	US00033386	2000	Unknown	HRMS	10MSHR06	TBD
GCMS	Waters/Micro mass	Autospec Ultima	M496	2000	Unknown	HRMS	10MSHR06	TBD

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Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Autosampler - U	CTC	PAL	1212740	Unknown	Unknown	HRMS	10MSHR06	TBD
GCMS	Waters/Micro mass	Autospec Premier	P808	2015	New	HRMS	10MSHR12	TBD
Autosampler - Y	CTC	Autospec P808	423629	Unknown	New	HRMS	10MSHR12	TBD
GC	Agilent	Autospec Premier	CN10471195	2015	New	HRMS	10MSHR12	TBD
GC	Agilent	6890A	US00036565	2000	Unknown	HRMS	10MSHR05	TBD
GCMS	Waters/Micro mass	Autospec Ultima	M488	2000	Unknown	HRMS	10MSHR05	TBD
Autosampler - F	CTC	PAL	423628	Unknown	Unknown	HRMS	10MSHR05	TBD
GC	Agilent	6890	CN10201169	2020	New	HRMS	10MSHR15	TBD
AutoSampler - L	Agilent	7683B Series Injector	CN94554262	2020	New	HRMS	10MSHR15	TBD
MS	Waters/Micro mass	Autospec Premier	P789	2020	New	HRMS	10MSHR15	TBD
LC-MS/MS	Sciex	API 4000	V23210806	2017	New	PFAS	10LCMS01	TBD
Bin pump	Agilent	G1312A	DE83103146	2018	New	PFAS	10LCMS01	TBD
LC-MS/MS	Sciex	API 4000	V1390304	2017	New	PFAS	10LCMS02	TBD
Bin pump	Agilent	G1312A	DE91604387	2018	New	PFAS	10LCMS02	TBD
Degassing Unit	SHIMADZU	DGU-20A5R	L20705569194 IX	2019	New	PFAS	10LCMS03	TBD
Liquid Chromatograph	SHIMADZU	Nexera X2 LC-30AD	L20555653493 US G	2019	New	PFAS	10LCMS03	TBD
Liquid Chromatograph	SHIMADZU	Nexera X2 LC-30AD	L20555653492 US G	2019	New	PFAS	10LCMS03	TBD
Autosampler	SHIMADZU	Nexera X2 SIL-20AC-XR	L20455250640 US G	2019	New	PFAS	10LCMS03	TBD
Reservoir Tray	SHIMADZU	Reservoir tray (Cat. No. 2258-45041-91)	L20305567270 SL	2019	New	PFAS	10LCMS03	TBD
Liquid Chromatograph	SHIMADZU	LC-20AB	L20125650517 US D	2019	New	PFAS	10LCMS03	TBD
Communications Bus Module	SHIMADZU	CBM-20A	L20235657676 US E	2019	New	PFAS	10LCMS03	TBD
Column Oven	SHIMADZU	Nexera X2 CTO-30A	L20575550727 US	2019	New	PFAS	10LCMS03	TBD
LCMS	SCIEX	QTRAP 5500	EG250621812	2019	New	PFAS	10LCMS03	TBD
LCMS	Agilent	6495 LC/TQ	SG2041D205	2021	New	PFAS	10PFA1	TBD
Column	Agilent	1290 Infinity II Multi Column Thermostat	DEBA406579	2021	New	PFAS	10PFA1	TBD
Multisampler	Agilent	1290 Infinity II Multisampler	DEBAS02792	2021	New	PFAS	10PFA1	TBD

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Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Pump Model	Agilent	MS 120 RVP LCMS High Speed Pump	IT2027N036	2021	New	PFAS	10PFA1	TBD
LCMS	Agilent	6495 LC/TQ	SG2216D203	2022	New	PFAS	10PFA2	TBD
Pump Model	Agilent	G7120A	DEBA206576	2022	New	PFAS	10PFA2	TBD
Multisampler Model	Agilent	G7167B	DEBAS03832	2022	New	PFAS	10PFA2	TBD
Column Oven	Agilent	G7116B	DEBA409759	2022	New	PFAS	10PFA2	TBD
Vortex	Fisher Scientific	cat #02215375	111220005	2018	New	Dioxin Prep	10HR21	TBD
Micro 100 Turbidimeter	Scientific Inc.	Micro 100 Turbidimeter	201309191	2005	Unknown	Dioxin Prep	10HR14	TBD
Accelerated Solvent Extractor	ACE	200	1020363	Unknown	Unknown	Dioxin Prep	10HR12	TBD
N-EVAP	Organomation	8125	57966	2012	Unknown	Dioxin Prep	DW1	TBD
N-EVAP	Organomation	8125	57529	2012	Unknown	Dioxin Prep	DW2	TBD
N-EVAP	Organomation	8125	57964	2012	Unknown	Dioxin Prep	N-EVAP 4	TBD
N-EVAP	Organomation	8125	57410	2012	Unknown	Dioxin Prep	N-EVAP 5	TBD
N-EVAP	Organomation	8125	57527	2012	Unknown	Dioxin Prep	N-EVAP 6	TBD
N-EVAP	Organomation	112	57528	Unknown	Unknown	Dioxin Prep	N-EVAP 7	TBD
Hypersep Vacuum Manifold	Thermo Scientific	60104233	1632	2017	Unknown	Dioxin Prep	10HR17	TBD
Hypersep Vacuum Manifold	Thermo Scientific	60104233	1552	2017	Unknown	PFAS	10HR16	TBD
Hypersep Vacuum Manifold	Thermo Scientific	60104233	1713-1	2017	Unknown	PFAS	10HR15	TBD
Centrifuge	IEC - International Equipment Company	HNS II	235525200	2018	New	Dioxin Prep	10HR18	TBD
Orbital Shaker	VWR	DS500	416G	2018	New	Dioxin Prep	10HR20	TBD
Oven	Lindberg Blue	GO1340A-1	O06M-568117-RM	2012	Used	Dioxin Prep	DP4	TBD
Oven	Thermo	F6018 (Med Level)	15031960120316	2012	Unknown	Dioxin Prep	DP5	TBD
Oven	Thermo	F6018 (Low Level)	15032170120319	2012	Unknown	Dioxin Prep	DP6	TBD
Oven	Carbolite	LHT/120	21-400729	2014	Unknown	Dioxin Prep	DP7	TBD
freezer	SPT	UF-214W	AS0115A228W20498	2017	New	Dioxin Prep	DP6	TBD
Kiln	SKUTT Automatic Kiln	GM-1414	000489	Unknown	Unknown	Dioxin Prep	10HR22	TBD
Vortex	Fisher Scientific	cat #02215375	111220001	Unknown	Unknown	Dioxin Prep	10HR23	TBD
Centrifuge	IEC - International Equipment Company	ICE Model CL Centrifuge	428-15985	Unknown	Unknown	Dioxin Prep	10HR24	TBD

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Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Chiller	ThermoFisher Scientific	Thermoflex 2500	0127680201150721	Unknown	Unknown	Dioxin Prep	10HR25	TBD
Chiller	ThermoFisher Scientific	Thermoflex 2500	0145040401120413	Unknown	Unknown	Dioxin Prep	10HR26	TBD
Chiller	ThermoFisher Scientific	Thermoflex 2500	0145032201120413	Unknown	Unknown	Dioxin Prep	10HR27	TBD
Vortex	Fisher Brand	G-560	2-131226	Unknown	Unknown	Dioxin Prep	10HR28	TBD
Hypersep Vacuum Manifold	Thermo Scientific	60104233	1713-2	Unknown	Unknown	Dioxin Prep	10HR29	TBD
Capping Station	CEM	574100	XC2871	Unknown	Unknown	Dioxin Prep	10HR30	TBD
1L SPE Station	CPI International	N/A	N/A	2019	New	Dioxin Prep	19562	TBD
Ultrasonic Bath	Branson	8510E-MT	EPA120597932F	Unknown	Unknown	Dioxin Prep	10HR31	TBD
PowrTrol Temperature Regulators	Glas-Col	104A PL120	11819758	Unknown	Unknown	Dioxin Prep	10HR32	TBD
PowrTrol Temperature Regulators	Glas-Col	104A PL120	11819751	Unknown	Unknown	Dioxin Prep	10HR33	TBD
PowrTrol Temperature Regulators	Glas-Col	104A PL120	11334540	Unknown	Unknown	Dioxin Prep	10HR34	TBD
PowrTrol Temperature Regulators	Glas-Col	104A PL120	11309113	Unknown	Unknown	Dioxin Prep	10HR35	TBD
PowrTrol Temperature Regulators	Glas-Col	104A PL120	11706611	Unknown	Unknown	Dioxin Prep	10HR36	TBD
PowrTrol Temperature Regulators	Glas-Col	104A PL120	11336607	Unknown	Unknown	Dioxin Prep	10HR37	TBD
Temperature Regulators	Thermolyne	CN45515	455000964338	Unknown	Unknown	Dioxin Prep	10HR38	TBD
PowrTrol Temperature Regulators	Glas-Col	104A PL120	11327046	Unknown	Unknown	Dioxin Prep	10HR39	TBD
PowrTrol Temperature Regulators	Glas-Col	104A PL120	11705717	Unknown	Unknown	Dioxin Prep	10HR40	TBD
PowrTrol Temperature Regulators	Glas-Col	104A PL120	11309114	Unknown	Unknown	Dioxin Prep	10HR41	TBD
PowrTrol Temperature Regulators	Glas-Col	104A PL120	11705712	Unknown	Unknown	Dioxin Prep	10HR42	TBD
PowrTrol Temperature Regulators	Glas-Col	104A PL120	11331266	Unknown	Unknown	Dioxin Prep	10HR43	TBD
PowrTrol Temperature Regulators	Glas-Col	104A PL120	11705714	Unknown	Unknown	Dioxin Prep	10HR44	TBD
PowrTrol Temperature Regulators	Glas-Col	104A PL120	11312697	Unknown	Unknown	Dioxin Prep	10HR45	TBD
PowrTrol Temperature Regulators	Glas-Col	104A PL120	11327705	Unknown	Unknown	Dioxin Prep	10HR46	TBD
PowrTrol Temperature	Glas-Col	104A PL120	11312700	Unknown	Unknown	Dioxin Prep	10HR47	TBD

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Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Regulators								
Chiller	ThermoFisher Scientific	ThermoFlex 900	0110204001120820	Unknown	Unknown	Dioxin Prep	10HR48	TBD
Refrigerator	Homelabs	HME030210N	HME030210N-2163	2019	New	Dioxin Prep	10HR49	TBD
Refrigerator	Homelabs	HME030210N	HME030210N-865	2019	New	Dioxin Prep	10HR50	TBD
Low speed Centrifuge	Premiere	XC-2450	C&AU070144	2019	New	Dioxin Prep	10HR51	TBD
SPE manifold	Thermo Scientific	60104233	1848	2019	New	PFAS	10HR52	TBD
SPE manifold	Thermo Scientific	60104233	1833	2019	New	PFAS	10HR53	TBD
ultrasonic bath	Fisher Scientific	FS30H	RTB011069292A	2019	Used	Dioxin Prep	10HR54	TBD
NEVAP	Organomation	112	8213	2019	Used	PFAS	NEVAP 8	TBD
Hypersep Vacuum Manifold	Thermo Scientific	60104233	1848-1	2019	New	PFAS	10HR55	TBD
Kiln	SKUTT Automatic Kiln	GM-1414	19G23-584	2019	New	Dioxin Prep	10HR56	TBD
SPE manifold	Thermo Scientific	60104233	1909	2019	New	Dioxin Prep	10HR57	TBD
SPE manifold	Thermo Scientific	60104233	1909	2019	New	PFAS	10HR58	TBD
Centrifuge	Damon/IEC Division	HN-SII	235511220	2019	New	Dioxin Prep	10HR59	TBD
Orbital Shaker	Lab-Line Instruments, Inc	3520	0185-0416	2019	New	Dioxin Prep	10HR60	TBD
Shaker	Fisherbrand	88861021	J8CF61021087	1/22/2020	New	Dioxin Prep	10HR61	PFAS area
SPE manifold	Thermo Scientific	60104233	1909	1/22/2020	New	PFAS	10HR62	PFAS area
Fridge	Avanti Fridge	ARBC17T2 PG	A58101911781862800035	3/5/2020	New	Dioxin Prep	10HR63	Dioxin
SPE manifold	Thermo Scientific	60104-233	238A638-01	3/6/2020	New	PFAS	10HR64	Dioxin
Evaporator	Zymark	Turbovap LV Evaporator	not fully visible	unknown	Used	Dioxin Prep	10HR65	TBD
Evaporator	Zymark	Turbovap LV Concentrator	TV0919N15245	unknown	Used	Dioxin Prep	10HR66	TBD
Freezer	Magic Chef	MCUF3W2	2011000840	4/13/2021	New	Dioxin Prep	DP8	Dioxin
12 position NEVAP	Organomation	NEVAP-111	0625	10/7/2021	Used	Dioxin Prep	NEVAP 9	Dioxin
SPE manifold	Thermo Scientific	60104-233	2012	4/23/2021	New	PFAS	10HR67	PFAS
SPE manifold	Thermo Scientific	60104-233	2027	4/23/2021	New	PFAS	10HR68	PFAS
SPE manifold	Thermo Scientific	60104-233	2009	8/9/2021	New	PFAS	10HR69	PFAS
SPE manifold	Thermo Scientific	60104-233	2033	8/9/2021	New	PFAS	10HR70	PFAS
Freezer	SPT	UF-214W	AS0115A228W20436	unknown	New	PFAS	10HR71	PFAS

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Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Cryomill	RETSCH	20.749.0001	1221130422I	2021	New	PFAS	10HR72	PFAS
Microwave extraction	CEM	MARS 6 230/60	MY2387	1/4/2022	New	Dioxin Prep	10HR73	Dprep
Ultrasonic Bath	Branson	CPX8800H	BGS122176990B	2/28/2022	New	PFAS	10HR74	PFAS
ICPMS	Agilent 7700	G3281A	5P13142395	6/1/2013	New	Metals	10ICM8	TBD
ICPMS - autosampler	Teledyne Cetac	ASX520	US011191A520	6/1/2013	New	Metals	10ICM8	TBD
ICPMS - chiller	Agilent	G3292-80000	2U1551028	6/1/2013	New	Metals	10ICM8	TBD
ICPMS - pump	Edwards	G31989	129449393	6/1/2013	New	Metals	10ICM8	TBD
ICPMS	Agilent 7700	G3281A	JP12412084	2014	Used	Metals	10ICM9	TBD
ICPMS - autosampler	Teledyne Cetac	ASX520	US0312120AS520	2014	Used	Metals	10ICM9	TBD
ICPMS - chiller	Agilent	6160T21QR 301	3U1621341	2014	Used	Metals	10ICM9	TBD
ICPMS - pump	Edwards	16436540	169436540	2014	Used	Metals	10ICM9	TBD
ICPMS	Agilent ICPM	7800	JP16120262	7/1/2016	New	Metals	10ICMB	TBD
ICPMS - autosampler	Agilent	G8410A	AU19156702	7/1/2016	New	Metals	10ICMB	TBD
ICP	Agilent Technologies	700 Series-ICP-OES	MY14160002	7/2/2016	New	Metals	10ICP4	TBD
ICP - autosampler	Teledyne Cetac	ASX520	12140A520	7/3/2016	New	Metals	10ICP4	TBD
ICP - chiller	Agilent	G8481-80003	1B13C1081	7/4/2016	New	Metals	10ICP4	TBD
ICP	Agilent Technologies	5100 -ICP-OES	MY15180003	2015	New	Metals	10ICP5	TBD
ICP - autosampler	Agilent	SPS4	AU15140009	2018	New	Metals	10ICP5	TBD
ICP - chiller	Agilent	G8481-80003	1A1550426	Unknown	New	Metals	10ICP5	TBD
Mercury Analyzer	Cetac	M7600	US15254007	2012	New	Metals	10HG08	TBD
Mercury Autosampler	Cetac	ASX-520	0315134A520	2010	New	Metals	10HG08	TBD
Mercury Analyzer	Teledyne Leeman Labs	M-7600	US18309003	2019	New	Metals	10HG09	TBD
Mercury Autosampler	Teledyne Cetac Technologies	ASX-560	0219146A560	2019	New	Metals	10HG09	TBD
Hot Block	Environmental Express	N/A	6083CECW2815	2006	New	Metals	10MET04	TBD
Hot Block	Environmental Express	N/A	8031CECW3358	2012	New	Metals	10MET08	TBD
Hot Block	Environmental Express	N/A	8031CECW3346	2012	New	Metals	10MET10	TBD
Hot Block	Environmental Express	SC154	8708CECW3720	2013	New	Metals	10MET23	TBD
Hot Block	Environmental Express	SC154	8793CECW3764	2014	New	Metals	10MET26	TBD
Hot Block	Environmental Express	N/A	8031CECW3342	2012	New	Metals	10MET09	TBD
Hot Plate	Cole Parmer	N/A	N/A	Unknown	New	Metals	10MP03	TBD

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TCLP agitator/tumbler	Analytical Testing Corp	DC-20	0685RKME0010	6/19/2008	New	Metals	10MET34	TBD
Hot Plate/hot block	Thermolyne	HP47135	1073970926967	1/1/2015	Unknown	Metals	10MET35	TBD
pH meter	Scientific Instruments	IQ180GLP	10240	1/1/2015	New	Metals	10MP05	TBD
pH meter	Orion Research	Expandable Ion Analyzer EA 940	1343	7/7/1905	Used	Metals	10MP06	TBD
Tumbler	Analytical Testing Corp	42R5BFC1-E3	0685SAMH002	4/1/2015	New	Metals	10MET36	TBD
Tumbler	Analytical Testing Corp	42R5BFC1-E3	0685SGMP0002	2015	New	Metals	10MET38	TBD
Tumbler	Analytical Testing Corp	42R5BFC1-E3	0685SGMQ0006	2015	New	Metals	10MET39	TBD
pH meter	Oakton	pH700	2404439	2015	New	Metals	10MP07	TBD
Temperature probe	Oakton	35613-13 (Lot code: 298)	93X052911	2015	New	Metals	10MP07	TBD
Oven/Desiccator	Fisher Isotemp	Isotemp Oven	903N0075	2017	New	Metals	10MET40	TBD
Oven	Fisher Scientific	Isotemp Oven	510N0239	2005	Unknown	Metals	10WET20	TBD
Oven	Fisher Scientific	851F	1589080190130	Unknown	Unknown	Metals	10WET49	TBD
Stir plate	Fisher Scientific	S88857200	C272000401175991	2017	New	Metals	10MET44	TBD
Oven/Desiccator	Fisher Isotemp	725F	903N0078	2017	New	Metals	10MET41	TBD
Centrifuge	ThermoScientific	Legend XT	42243876	2018	New	Metals	10MET45	TBD
Turbidity Meter	HACH	TU5200	1808718	2018	New	Metals	10WT46 (10MET46)	TBD
Oven	Quincy Labs	10GC	G1-015608	2019	New	Metals	10MET47	TBD
ICPMS	Agilent 7900 ICP-MS	G8403A	SG19304531	2019	New	Metals	10ICMC	TBD
ICPMS - chiller	Agilent	G3292-80200	1908-01399	2019	New	Metals	10ICMC	TBD
ICPMS - pump	Agilent	9599225M013	1f19325139	2019	New	Metals	10ICMC	TBD
ICPMS - autosampler	Agilent	G8410A	AU19156705	2019	New	Metals	10ICMC	TBD
Digestion Block	Environmental Express	SC154	2020CECW5296	2020	New	Metals	10MET49	Hotblock logbook drawer
Digestion Block	Environmental Express	SC154	2020CECW5341	2020	New	Metals	10MET50	Hotblock logbook drawer
Autofill station	Environmental Express	Autofill Station	ABF5000-0420-087	2020	New	Metals	10MET51	Hotblock logbook drawer
Oven	Fisher	151030521	42408312	2019	New	Metals	10MET52	Taped to the side of the oven
Hot Stir Plate	Cole Parmer	HS19 C-P	50010030	2020	New	Metals	10MET53	Hanging above instrument
Hot Block	Environmental Express	SC154	2020CECW5429	2020	New	Metals	10MET54	Hotblock logbook drawer
FIMS400	Perkin Elmer	FIMS400	401S20122101	2021	New	Metals	10HG10	Hotblock logbook drawer
Autosampler	Perkin Elmer	S25	0720032S25	2022	New	Metals	10HG10	Hotblock logbook drawer

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Digestion Block	Environmental Express	SC154	2020CECW5430	2021	New	Metals	10MET55	Hotblock logbook drawer
ICPMS Instrument	Agilent	G8403A	SG19374593	2021	New	Metals	10ICMD	Hotblock logbook drawer
Autosampler SPS4	Agilent	G8410A	AU19156705	2021	New	Metals	10ICMD	Hotblock logbook drawer
Chiller	Agilent	G3929-80200	1908-02202	2021	New	Metals	10ICMD	Hotblock logbook drawer
ICP Instrument	Agilent	5110	MY18260006	2021	New	Metals	10ICP6	Hotblock logbook drawer
Autosampler SPS4	Agilent	G8410A	AU18144765	2021	New	Metals	10ICP6	Hotblock logbook drawer
Recirculating Chiller	Agilent	G8481A	1807-00914	2021	New	Metals	10ICP6	Hotblock logbook drawer
Microwave	CEM	MARS6	MY2191	2021	New	Metals	10MP09	Hotblock logbook drawer
Stir plate	Fisher	3G11	LBKF63001	2022	New	Metals	10MET56	Hotblock logbook drawer
Stir plate	Fisher	0	C2720019041808220	2022	New	Metals	10MET57	Hotblock logbook drawer
Stir plate	Fisher	0	C1931151230188	2022	New	Metals	10MET58	Hotblock logbook drawer
Stir plate	Fisher	0	C3420010022001741	2022	New	Metals	10MET59	Hotblock logbook drawer
Hotblock	Environmental Express	SC154	2020CECW5435	2022	New	Metals	10MET60	Hotblock logbook drawer
Stir plate	N/A	N/A	HA2520222	2022	Used	Metals	10MET61	TBD
UltraSonicator	Branson	8510	RPC10096911F	unknown	New	O-Prep	10OP17	O-Prep - drawer under Muffle Furnace
Sonicator	Misonix	XL 2020	G3914	unknown	New	O-Prep	10OP01	O-Prep - drawer under Muffle Furnace
Sonicator	Misonix	XL 2015	G4180	2007	Unknown	O-Prep	10OP02	O-Prep - drawer under Muffle Furnace
Sonicator	Misonix	Sonicator 3000	R1638	2007	Unknown	O-Prep	10OP04	O-Prep - drawer under Muffle Furnace
N-EVAP	Organomation	112	8169	Unknown	Unknown	O-Prep	10OP10	O-Prep - drawer under Muffle Furnace
N-EVAP (waterbath)	Organomation	112	7537	Unknown	Unknown	O-Prep	10OP11	O-Prep - drawer under Muffle Furnace
N-EVAP (sample tray)	Organomation	112	Not readable	Unknown	Unknown	O-Prep	10OP11	O-Prep - drawer under Muffle Furnace
Refrigerator	Traulsen	G20010	T34931C10	2011	New	O-Prep	OP1	O-Prep - drawer under Muffle Furnace
Centrifuge	IEC	Centra GP8	31210390	Unknown	Unknown	O-Prep	10OP13	O-Prep - drawer under Muffle Furnace
Centrifuge	Damon/IEC Division	N/A	9304	Unknown	Unknown	O-Prep	10OP14	O-Prep - drawer under Muffle Furnace
Centrifuge	International Clinical Centrifuge	CL28899M	28899M	Unknown	Unknown	O-Prep	10OP15	O-Prep - drawer under Muffle Furnace

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Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Muffle Furnace	Lindberg/Blue M	BF51828C-1	505296	Unknown	Unknown	O-Prep	10OP16	O-Prep - drawer under Muffle Furnace
N-EVAP	Organomation	II2	4185	2014	Unknown	O-Prep	10OP18	O-Prep - drawer under Muffle Furnace
Buchi Concentrator-vacuum controller	Buchi Labortenchik Ag	V-855	10000162387	2014	Unknown	O-Prep	10OP21	O-Prep - drawer under Muffle Furnace
Buchi Concentrator-vacuum pump	Buchi Labortenchik Ag	V-700	1000166230	2014	Unknown	O-Prep	10OP21	O-Prep - drawer under Muffle Furnace
Buchi Concentrator-Recirculating Chiller	Buchi Labortenchik Ag	F-108	1019513	2014	Unknown	O-Prep	10OP21	O-Prep - drawer under Muffle Furnace
Buchi Concentrator System	Buchi Labortenchik Ag	Q101	1000167481	2014	Unknown	O-Prep	10OP21	O-Prep - drawer under Muffle Furnace
Microwave extraction	CEM	MarsXpress 230/60	MD3483	7/1/2014	Unknown	O-Prep	10OP19	O-Prep - drawer under Muffle Furnace
Sonicator	Bransonic	B8200R-3	Not readable	2014	Used	O-Prep	10OP23	O-Prep - drawer under Muffle Furnace
Buchi Concentrator-vacuum controller	Buchi Labortenchik Ag	V-855	1000171188	2014	Unknown	O-Prep	10OP24	O-Prep - drawer under Muffle Furnace
Buchi Concentrator-vacuum pump	Buchi Labortenchik Ag	V-700	1000176128	2014	Unknown	O-Prep	10OP24	O-Prep - drawer under Muffle Furnace
Buchi Concentrator-Recirculating Chiller	Buchi Labortenchik Ag	F-108	1000174259	2014	Unknown	O-Prep	10OP24	O-Prep - drawer under Muffle Furnace
Buchi Concentrator System	Buchi Labortenchik Ag	Q101	1000176659	2014	Unknown	O-Prep	10OP24	O-Prep - drawer under Muffle Furnace
Buchi Concentrator-vacuum controller	Buchi Labortenchik Ag	V-855	1000174543	2014	Unknown	O-Prep	10OP25	O-Prep - drawer under Muffle Furnace
Buchi Concentrator-vacuum pump	Buchi Labortenchik Ag	V-700	1000174270	2014	Unknown	O-Prep	10OP25	O-Prep - drawer under Muffle Furnace
Buchi Concentrator-Recirculating Chiller	Buchi Labortenchik Ag	F-108	1000172490	2014	Unknown	O-Prep	10OP25	O-Prep - drawer under Muffle Furnace
Buchi Concentrator System	Buchi Labortenchik Ag	Q101	1000176601	2014	Unknown	O-Prep	10OP25	O-Prep - drawer under Muffle Furnace
Buchi Concentrator-vacuum controller	Buchi Labortenchik Ag	V-855	1000171253	2014	Unknown	O-Prep	10OP26	O-Prep - drawer under Muffle Furnace
Buchi Concentrator-vacuum pump	Buchi Labortenchik Ag	V-700	1000176882	2014	Unknown	O-Prep	10OP26	O-Prep - drawer under Muffle Furnace
Buchi Concentrator-Recirculating Chiller	Buchi Labortenchik Ag	F-108	1000174257	2014	Unknown	O-Prep	10OP26	O-Prep - drawer under Muffle Furnace

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Buchi Concentrator System	Buchi Labortenchik Ag	Q101	1000176658	2014	Unknown	O-Prep	10OP26	O-Prep - drawer under Muffle Furnace
Microwave	Novawave	Model FA	NWX0115110105	2015	New	O-Prep	10OP27	Online
Refrigerator/freezer	Whirlpool	WH43S1E	T127161605028	2016	New	O-Prep	OP4	O-Prep - drawer under Muffle Furnace
Water Bath	NA	NA	NA	Unknown	Unknown	O-Prep	10OP28	O-Prep - drawer under Muffle Furnace
Water Bath	NA	NA	NA	Unknown	Unknown	O-Prep	10OP29	O-Prep - drawer under Muffle Furnace
NEVAP (sample tray)	Organomation	112	7955	Unknown	Unknown	O-Prep	10OP30	O-Prep - drawer under Muffle Furnace
NEVAP (water bath)	Organomation	112	10185	Unknown	Unknown	O-Prep	10OP30	O-Prep - drawer under Muffle Furnace
NEVAP (delete bath)	Organomation	112	22812	Unknown	Unknown	O-Prep	10OP31	O-Prep - drawer under Muffle Furnace
XcelVap	XcelVap	XcelVap Vaporator System	19-5680	2020	New	O-Prep	10OP32	O-Prep - drawer under Muffle Furnace
XcelVap	XcelVap	XcelVap Vaporator System	19-5681	2020	New	O-Prep	10OP33	O-Prep - drawer under Muffle Furnace
XcelVap	XcelVap	XcelVap Vaporator System	19-5684	2020	New	O-Prep	10OP34	O-Prep - drawer under Muffle Furnace
XcelVap	XcelVap	XcelVap Vaporator System	19-5683	2020	New	O-Prep	10OP35	O-Prep - drawer under Muffle Furnace
XcelVap	XcelVap	XcelVap Vaporator System	19-5682	2020	New	O-Prep	10OP36	O-Prep - drawer under Muffle Furnace
Sonicator	Fisher Scientific	XI.2020	F1250	2021	Used	O-Prep	10OP37	O-Prep - drawer under Muffle Furnace
Refrigerator	Crown	Walk-in	Unknown	Unknown	New	SR	C1	TBD
Refrigerator	Beverage Air	KR48-1AS	KR48-1AS 9029136	9/1/2011	New	SR	C17	TBD
Refrigerator	U.S. Cooler	Walk-in/FCL3476 GL1	30692	6/1/2013	New	SR	C18	TBD
Refrigerator	Carroll Coolers LLC	Walk-in	34365	9/24/2013	New	SR	C16	TBD
Refrigerator	TRUE	GDM-47-HC-LD	9199842	2017	New	SR	C22	TBD
Freezer (converted to fridge 03.25.21)	ATOSA	MBF8003	MBF8003079160617 00C40007	2017	New	SR	C23	TBD
Refrigerator	Volition	R49-S	R49S-18010046	12/28/2019	New	SR	C24	TBD
Freezer (converted to fridge)	ATOSA	MBF8003	MBF8003AUS10031 7041900C40004	2018	New	SR	C25	TBD
Refrigerator	Premium	PRF90DX	M8828208666000016 7	2019	New	SR	C27	TBD
Walk-in Freezer	NORLAKE	Walk-In	KL/DP36X78	2020	New	SR	C28	TBD

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Walk-in Cooler	Imperial Brown	Walk-In	21-IB-56205-01	2021	New	SR	C29	TBD
GC System	Agilent	7890A (G3440A)	CN10021030	2010	New	SVOA	10MSSA	TBD
Autosampler Tower	Agilent/HP	7693 Series (G4513A)	CN13460268	2010	New	SVOA	10MSSA	TBD
Autosampler Tray	Agilent/HP	7693 Series (G4513A)	CN10020004	2010	New	SVOA	10MSSA	TBD
MS Detector	Agilent/HP	5975C (G3172A)	US10030005	2010	New	SVOA	10MSSA	TBD
AutoSampler Tower	Agilent	7683B (G2913A)	CN75045773	2010	New	SVOA	10MSSB	TBD
GC/Oven	Agilent	7890A (G3440A)	CN10842006	2010	New	SVOA	10MSSB	TBD
MS Detector	Agilent	5975C (G3172A)	US73317796	2010	New	SVOA	10MSSB	TBD
AutoSampler Tray	Agilent	7683 Series (G2614A)	CN54237163	2010	New	SVOA	10MSSB	TBD
GC	Agilent	6890N (G1530N)	CN10550045	2011	Used	SVOA	10MSSD	TBD
MS	Agilent	5975 (G3172A)	US53931370	2011	Used	SVOA	10MSSD	TBD
Autosampler Tray	Agilent	7683 (G2614A)	CN54337193	2011	Used	SVOA	10MSSD	TBD
Tower 7683B	Agilent	7683B (G2913A)	CN52425737	2011	Used	SVOA	10MSSD	TBD
GC	Agilent	6890N (G1530N)	US10245155	2001	Unknown	SVOA	10MSS6	TBD
Autosampler Tower	Agilent/HP	7683 Series (G2613A)	US82901662	2001	Unknown	SVOA	10MSS6	TBD
MS	Agilent/HP	5973N (G2578A)	US21854348	2001	Unknown	SVOA	10MSS6	TBD
Autosampler Tray	Agilent/HP	7683 Series (G2614A)	US81100461	2001	Unknown	SVOA	10MSS6	TBD
GC	Agilent	6890N (G1530N)	CN10319023	2006	Unknown	SVOA	10MSS7	TBD
Tower 7683	Agilent	7683 Series (G2613A)	CN24728345	2006	Unknown	SVOA	10MSS7	TBD
Tray 7683	Hewlett Packard	7683 Series (G2614A)	US12411901	2006	Unknown	SVOA	10MSS7	TBD
Mass Spec 5973	Agilent	5973N (G2579A)	US21864477	2006	Unknown	SVOA	10MSS7	TBD
AutoSampler Tower	Agilent/HP	7683 Series (G2613A)	US10417469	2008	Unknown	SVOA	10MSS8	TBD
GC/Oven	Agilent	6890N (G1530N)	US10123035	2008	Unknown	SVOA	10MSS8	TBD
MS Detector	Agilent	5973N (G2577A)	US10440794	2008	Unknown	SVOA	10MSS8	TBD
AutoSampler Tray	Agilent/HP	7683 Series (G2614A)	CN53536362	2008	Unknown	SVOA	10MSS8	TBD
GC/Oven	Agilent	6890A (G1530A)	US00033558	1999	Unknown	SVOA	10MSS9	TBD
AutoSampler Tower	Agilent	7683 Series (G2613A)	CN15223766	1999	Unknown	SVOA	10MSS9	TBD
MS Detector	Agilent	5973N (G2577A)	US90440006	1999	Unknown	SVOA	10MSS9	TBD
AutoSampler Tray	Agilent	7683 (G2614A)	US13012239	1999	Unknown	SVOA	10MSS9	TBD
AutoSampler Tray	Agilent	6890 Series (18596M)	3643A43317	7/1/2014	Used	SVOA	10MSSE	TBD
Injector Tower	Agilent	6890 Series (18593B)	3517A42509	7/1/2014	Used	SVOA	10MSSE	TBD

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GC/Oven	Agilent	6890 Series (G1530A)	US00006288	7/1/2014	Used	SVOA	10MSSE	TBD
MS Detector	Agilent	5973 Series (G1098A)	US63810194	7/1/2014	Used	SVOA	10MSSE	TBD
Autosampler Tray	Agilent	7683 Series (G2614A)	CN91252935	7/1/2014	Used	SVOA	10MSSF	TBD
Injector Tower	Agilent	7683B Series (G2913A)	CN54128250	7/1/2014	Used	SVOA	10MSSF	TBD
MS Detector	Agilent	5975C (G3172A)	US91732455	7/1/2014	Used	SVOA	10MSSF	TBD
GC	Agilent	7890A (G3440A)	CN10920003	7/1/2014	Used	SVOA	10MSSF	TBD
GC	Agilent	6890 Series (G1530A)	US00025032	7/1/2014	Used	SVOA	10MSSG	TBD
MS	Agilent	5973 Series (G1098A)	US82311330	7/1/2014	Used	SVOA	10MSSG	TBD
Autosampler Tray	HP	7683 Series (G2614A)	US90403281	7/1/2014	Used	SVOA	10MSSG	TBD
Injector Tower	HP	7683 Series (G2613A)	US95310976	7/1/2014	Used	SVOA	10MSSG	TBD
MS	HP	5977B (G7081B)	US1703R003	2000	Unknown	SVOA	10MSSH	TBD
GC	HP	7890B (G3442B)	CN17013216	2000	Unknown	SVOA	10MSSH	TBD
Autosampler Tray	Agilent/HP	7693 Series (G4514A)	CN16480039	2000	Unknown	SVOA	10MSSH	TBD
Injector Tower	Agilent/HP	7693 Series (G4513A)	CN95203168	2000	Unknown	SVOA	10MSSH	TBD
Autosampler Tower	Agilent/HP	7693A (G4513A)	CN19390131	2019	new	SVOA	10MSSI	TBD
GC System	Agilent/HP	8890 Series (G3540A)	US1951A021	2019	new	SVOA	10MSSI	TBD
MS Detector	Agilent/HP	5977B (G7081B)	US2001R066	2019	new	SVOA	10MSSI	TBD
Autosampler Tray	Agilent/HP	7693A (G4514A)	R01950087	2019	new	SVOA	10MSSI	TBD
GC	Agilent	G3540A (8890),	US2120A017	2021	new	SVOA	10MSSJ	TBD
MS	Agilent	G7081B (5977B)	US2116R031	2021	new	SVOA	10MSSJ	TBD
Autosampler	Agilent	G4514A	R021077022	2021	new	SVOA	10MSSJ	TBD
Tower	Agilent	G4513A	R021155026	2021	new	SVOA	10MSSJ	TBD
GC	Agilent	6890N	CN10549055	2011	Unknown	SVOA	10GCSE	TBD
Autosampler Tray	Agilent	G2614A	US14213141	2011	Unknown	SVOA	10GCSE	TBD
Tower	Agilent	G2613A	CN54929639	2011	Unknown	SVOA	10GCSE	TBD
ECD 1	Agilent	G2397A	U8977	2011	Unknown	SVOA	10GCSE	TBD
ECD 2	Agilent	G2397A	U8978	2011	Unknown	SVOA	10GCSE	TBD
GC	Agilent	7890A	CN11201069	2011	Unknown	SVOA	10GCSM	TBD
Autosampler Tray	Agilent	64514A	CN11130097	2011	Unknown	SVOA	10GCSM	TBD
Tower	Agilent	64513A	CN91200383	2011	Unknown	SVOA	10GCSM	TBD

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Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
ECD 1	Agilent	G2397A	U19081	Unknown	Unknown	SVOA	10GCSM	TBD
ECD 2	Agilent	G2397A	U19082	Unknown	Unknown	SVOA	10GCSM	TBD
GC	Agilent	6890 N	US10126008	2004	Unknown	SVOA	10GCSD	TBD
AutoSampler Tray	Agilent/HP	G2614A	US13612659	2004	Unknown	SVOA	10GCSD	TBD
Tower	Agilent/HP	G2613A	US11818906	2004	Unknown	SVOA	10GCSD	TBD
ECD 1	Agilent	G2397A	U33680	Unknown	Unknown	SVOA	10GCSD	TBD
ECD 2	Agilent	G2397A	U33677	Unknown	Unknown	SVOA	10GCSD	TBD
Tower	Agilent	64513A	CN91200383	2009	Unknown	SVOA	10GCS9	TBD
GC	Agilent	7890A	CN10915106	2009	Unknown	SVOA	10GCS9	TBD
Autosampler Tray	Agilent	64514A	CN91100084	2009	Unknown	SVOA	10GCS9	TBD
GC	Agilent	7890B	CN18203068	2018	Unknown	SVOA	10GCSL	TBD
Autosampler - Front	Agilent	G4514A	CN18140044	2018	Unknown	SVOA	10GCSL	TBD
AutoInjector - Front	Agilent	G4513A	CN18160194	2018	Unknown	SVOA	10GCSL	TBD
Autosampler - Rear	Agilent	G4514A	CN18140044	2018	Unknown	SVOA	10GCSL	TBD
AutoInjector - Rear	Agilent	G4513A	CN18160191	2018	Unknown	SVOA	10GCSL	TBD
Freezer	Haier	HUM013E A	BB01H1E0100BHD7 S0358	2019	Unknown	SVOA	SV4	TBD
Refrigerator	Frigidaire	FFTR1814T WG	BA04428653	2020	NEW	SVOA	SV5	Above the unit
Freezer	Frigidaire	FFTR1814T WG	BA04428653	2020	NEW	SVOA	SV5	Above the unit
Autoinjector - Front	Agilent	G2913	CN44659505	7/1/2014	Unknown	SVOA	10GCSF	TBD
Autosampler - Front	Agilent	G2614A	CN00654640	7/1/2014	Unknown	SVOA	10GCSF	TBD
GC	Agilent	7890A	CN10848062	7/1/2014	Unknown	SVOA	10GCSF	TBD
Autoinjector - Rear	Agilent	G2913A	CN91756454	7/1/2014	Unknown	SVOA	10GCSF	TBD
Autosampler - Rear	Agilent	G2614A	CN00654640	7/1/2014	Unknown	SVOA	10GCSF	TBD
GC	Agilent	6890A	US00035764	7/1/2014	Unknown	SVOA	10GCSG	TBD
Autosampler Tray	Agilent	G2614A	CN43530410	Unknown	Unknown	SVOA	10GCSG	TBD
Tower	Agilent	G2613A	US00411307	Unknown	Unknown	SVOA	10GCSG	TBD
ECD 1	Agilent	G2397A	U26804	Unknown	Unknown	SVOA	10GCSG	TBD
ECD 2	Agilent	G2397A	U26805	Unknown	Unknown	SVOA	10GCSG	TBD
GC	Agilent	7890A	CN11141025	6/1/2017	Used	SVOA	10GCSI	TBD
Autosampler Tray	Agilent	G2614A	CN84951713	6/1/2017	Used	SVOA	10GCSI	TBD
Tower	Agilent	G2613A	CN85154856	6/1/2017	Used	SVOA	10GCSI	TBD

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ECD 1	Agilent	G2397A	U28247	6/1/2017	Used	SVOA	10GCSI	TBD
ECD 2	Agilent	G2397A	U30564	6/1/2017	Used	SVOA	10GCSI	TBD
GC	Agilent	7890A	CN10906059	6/1/2017	Used	SVOA	10GCSJ	TBD
Autosampler	Agilent	G2614A	CN85252214	6/1/2017	Used	SVOA	10GCSJ	TBD
Tower	Agilent	G2313A	CN85154864	6/1/2017	Used	SVOA	10GCSJ	TBD
ECD 1	Agilent	G2397A	U27008	6/1/2017	Used	SVOA	10GCSJ	TBD
ECD 2	Agilent	G2397A	U30558	6/1/2017	Used	SVOA	10GCSJ	TBD
GC	Agilent	7890A	CN10906049	6/1/2017	Used	SVOA	10GCSK	TBD
Autosampler Tray	Agilent	G4514A	CN11080020	6/1/2017	Used	SVOA	10GCSK	TBD
Tower	Agilent	G4513A	CN1170127	6/1/2017	Used	SVOA	10GCSK	TBD
ECD 1	Agilent	G2397A	U27007	6/1/2017	Used	SVOA	10GCSK	TBD
ECD 2	Agilent	G2397A	U16942	6/1/2017	Used	SVOA	10GCSK	TBD
Refrigerator/Freezer	Frigidaire	FFTR1814TW	BA14129815	11/10/2021	New	SVOA	SV6	Above the unit
Refrigerator/Freezer	Frigidaire	FFTR1814TW	BA14126235	11/10/2021	New	SVOA	SV7	Above the unit
GC	Agilent	G1530A	US00021845	1/5/2022	Used	SVOA	10GCSN	Above the unit
Tower	Agilent	G2613A	US00411307	1/5/2022	Used	SVOA	10GCSN	Above the unit
Tray	Agilent	G2614A	CN21920654	1/5/2022	Used	SVOA	10GCSN	Above the unit
GC	Agilent	G1530A	US00020689	9/19/2022	Used	SVOA	10GCSO	Above the unit
Tower	Agilent	G2613A	CN14222693	9/19/2022	Used	SVOA	10GCSO	Above the unit
Tower	Agilent	G2613A	CN34433512	9/19/2022	Used	SVOA	10GCSO	Above the unit
Tray	Agilent	C2614A	CN81648202	9/19/2022	Used	SVOA	10GCSO	Above the unit
AutoSampler	EST Analytical	Centurion	cents211121510	2000	Unknown	VOA	10MSV5	TBD
Concentrator	Encon Evolution	N/A	EV331120210	2000	Unknown	VOA	10MSV5	TBD
GC	HP	6890	DE00020316	2000	Unknown	VOA	10MSV5	TBD
MS	HP MS	5973	US81221500	2000	Unknown	VOA	10MSV5	TBD
Concentrator	Tekmar	3000	173001	2006	Unknown	VOA	10MSV6	TBD
AutoSampler	Varian Archon	N/A	13352	2006	Unknown	VOA	10MSV6/ 10MSV9	TBD
GC	Agilent	6890A	US00036184	2006	Unknown	VOA	10MSV6/ 10MSV9	TBD
MS	Agilent	5973	US01140180	2006	Unknown	VOA	10MSV6/ 10MSV9	TBD
AutoSampler	Environmental Sample Tech, Inc.	N/A	cents207121110	2008	Unknown	VOA	10MSV7	TBD

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GC	Agilent Technologies	6850	CN107520005	2008	Unknown	VOA	10MSV7	TBD
Concentrator	Tekmar	3000	(94251012) US02060004	2008	Unknown	VOA	10MSV7	TBD
MS	Agilent Technologies	5975C	US74818132	2008	Unknown	VOA	10MSV7	TBD
GC	5975C	5975C	(CN10742012) US73337433	2011	Unknown	VOA	10MSV8	TBD
AutoSampler	EST Analytical	Centurion	cents205112310	2011	Unknown	VOA	10MSV8	TBD
Concentrator	Encon Evolution	N/A	EV333120210	2011	Unknown	VOA	10MSV8	TBD
MS	Agilent	5975C	US73337433	2011	Unknown	VOA	10MSV8	TBD
Concentrator	Tekmar	14-3100-OEL	1064004	2012	Unknown	VOA	10MSV9	TBD
GC	Agilent	6890	US10215113	2013	Unknown	VOA	10MSVA	TBD
MS	Agilent	5973	US10442746	2013	Unknown	VOA	10MSVA	TBD
autosampler/concentrator	Tekmar	Atomx 15-0000-100	US11203002	2013	Unknown	VOA	10MSVA	TBD
GC	HP	6890	US40620426	Unknown	Unknown	VOA	10MSVE	TBD
Concentrator	Teledyne Tekmar	14-9800-100	CN10427049	Unknown	Unknown	VOA	10MSVE	TBD
AutoSampler	Teledyne Tekmar	15-0500-000	US12058001	Unknown	Unknown	VOA	10MSVE	TBD
MS	HP	5973	US40620426	Unknown	Unknown	VOA	10MSVE	TBD
GC	Agilent	7890B	CN16433144	2017	New	VOA	10MSVF	TBD
AutoSampler	EST Analytical	Centurion	CENTS205112310	2000	New	VOA	10MSVF	TBD
Concentrator	EST Analytical	Encon Evolution	EV332120210	2000	New	VOA	10MSVF	TBD
MS	Agilent	5977B	US1701R009	2017	New	VOA	10MSVF	TBD
GC	Agilent	7890B	CN18043128	2018	New	VOA	10MSVG	TBD
MS	Agilent	5977B	US1816R028	2018	New	VOA	10MSVG	TBD
Auto-sampler	EST	CenturionW	CENTW646061218	2018	New	VOA	10MSVG	TBD
Concentrator	EST	Encon EV	EV974061218	2018	New	VOA	10MSVG	TBD
AutoSampler	Environmental Sample Tech, Inc.	N/A	13713	1990	Unknown	VOA	10GCV5	TBD
Concentrator	Tekmar	3100	99343009	1990	Unknown	VOA	10GCV5	TBD
GC	HP	G1530A	US00020223	2012	Unknown	VOA	10GCV5	TBD
AutoSampler	EST Analytical	Archon 8100	13719	2012	Unknown	VOA	10GCV6	TBD
Concentrator	Tekmar	14-3100-EOL	US020600004	2012	Unknown	VOA	10GCV6	TBD
GC	Agilent/HP	HP 6890	US00042909	6/1/2013	Unknown	VOA	10GCV6	TBD
AutoSampler	EST Analytical	Centurion	CENT244112907	7/1/2014	Unknown	VOA	10GCV9	TBD

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Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Concentrator	EST Analytical	Encon	580013108P	7/1/2014	Unknown	VOA	10GCV9	TBD
GC	Agilent Technologies	7890A	CN12071022	Unknown	Unknown	VOA	10GCV9	TBD
GC	Agilent Technologies	G1530A	US00002531	2005	Used	VOA	10GCVA	TBD
Headspace Sampler	Agilent Technologies	G1888	IT00507022	2005	Used	VOA	10GCVA	TBD
GC	Agilent	8890	US1951A018	2006	Used	VOA	10GCVB	TBD
Autosampler	EST Analytical	Centurion	W695050819	Unknown	Used	VOA	10GCVB	TBD
Concentrator	Tekmar - Dohrmann	3100 Sample Conc.	1064004	Unknown	Used	VOA	10GCVB	TBD
Oven	Thermo Scientific	N/A	6520-6528	Unknown	Unknown	VOA	10VOA03	TBD
Refrigerator	Crown	Walk-In	Unknown	Unknown	Unknown	VOA	C2	TBD
Refrigerator	Beverage Air	KR74-1AS	6331221	Unknown	Unknown	VOA	C7	TBD
Sonicator	Fisher Scientific	FS220	RWA040963796A	Unknown	Unknown	VOA	10VOA04	TBD
Freezer	Frigidaire	LFFH21F7 HWG	WB94954367	2013	Unknown	VOA	V5	TBD
Refrigerator	Norlake Scientific	NSLF482W AW/1	96020404	Unknown	Unknown	VOA	V6	TBD
Oven	Lindberg/Blue M	MO1450PS A-1	U19R-507936-UR	Unknown	Unknown	VOA	10WT56	TBD
Refrigerator/Freezer	Frigidaire	BA72845548	FRT8G7HW0	Unknown	Unknown	VOA	V8	TBD
Refrigerator	Amana	ABB2221W EB1	K13809596	7/1/2014	Used	VOA	V7	TBD
Fridge	Danby Designer	DBC120BLS	4316063619504	2016	New	Wet Chem	MP1	TBD
Incubator	Fisher Scientific	Isotemp Incubator	115770704-57744	2006	Unknown	Wet Chem	10WET16	TBD
Incubator	Fisher Scientific	307C	2018090423462	2009	Unknown	Wet Chem	10WET35	TBD
Incubator	Thermo Forma	3940	300789-1711	2012	Unknown	Wet Chem	10WET60	TBD
Autotitrator	Metrohm	888 Titrando Titrator	1888001004148	2010	Unknown	Wet Chem	10WET6	TBD
Autosampler	Metrohm	778 Sample Processor	1778001003123	2010	Unknown	Wet Chem	10WET6	TBD
probe	Metrohm	778 Sample Processor	263664	2010	Unknown	Wet Chem	10WET6	TBD
AutoClave	Harvey	N/A	12770804/02244	2009	Unknown	Wet Chem	10WET29	TBD
Colony Counter	Gallenkamp	CNW-325-030Y	4A 4294	2004	Unknown	Wet Chem	10WET30	TBD
Colony Counter	Darkfield Quebec	Colony Counter	N/A	Unknown	Unknown	Wet Chem	10WET38	TBD
Water Bath	Fisher Scientific	Isotemp 210	1605680347017	Unknown	Unknown	Wet Chem	10WET27	TBD
Refrigerator	Carroll Coolers LLC	Walk-in	6584	Unknown	Unknown	Wet Chem	C11	TBD
Spectrometer	Hach	DR 3900	1811411	2018	New	Wet Chem	10WETF	TBD
Hot Plate	Presto	Tilt'n Drain Big Griddle	2608US	2009	New	Wet Chem	10WET34	TBD

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Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Hot Plate	Corning	N/A	440895	Unknown	Unknown	Wet Chem	10WET40	TBD
Stir Plate	Fisher Scientific	N/A	1889080719259	Unknown	Unknown	Wet Chem	10WET41	TBD
Stir Plate	Barnstead/Thermolyne	S46725/Cimarec 2	776940355770	Unknown	Unknown	Wet Chem	10WET42	TBD
Refrigerator	Summit Commercial	SCR485L	A091200156	2010	New	Wet Chem	WC2	TBD
BOD meter	Hach	HQ40d	80900024869	2011	New	Wet Chem	10WT54	TBD
BOD/pH probe	Hach	LBOD10101	123213032021	2011	New	Wet Chem	10WT54	TBD
pH probe	HACH	PHC108	191282867899	2011	New	Wet Chem	10WT54	TBD
pH/BOD meter/Fluoride	Hach	HQ40d	110300052350	2011	New	Wet Chem	10WT53	TBD
pH/BOD meter/Fluoride - probe	Hach	HQ40d	152392938004	2011	New	Wet Chem	10WT53	TBD
Hot Block	Environmental Express	N/A	N/A	2009	Used	Wet Chem	10WET55	TBD
Oven	Fisher Scientific	13-247-650G(6905)	611729-434	2012	New	Wet Chem	10WET65	TBD
pH Probe	Hach	PHC301	11662571034	2011	New	Wet Chem	11662571034	TBD
pH Probe	Hach	PHC301	121952571033	2012	New	Wet Chem	121952571033	TBD
pH Probe	Hach	LBOD101	122143032067	2012	New	Wet Chem	122143032067	TBD
pH Probe	Switchcraft	PHW77-SS	712202002	2012	New	Wet Chem	712202002	TBD
Turbidity Meter	Hach	2100Q	11050C0092997	2011	New	Wet Chem	10WT59	TBD
Handheld Brix Refractometer	Fisher	N/A	Fisher catalog # 13-946-21	2011	New	Wet Chem	10WT60	TBD
Quanti-Tray Sealer Model 2x	Quanti-Tray	89-10894-02	4836	2012	New	Wet Chem	10WET56	TBD
IC	Metrohm	881 Compact IC	1881000121132	2012	New	Wet Chem	10WT61	TBD
Lachat	Quick Chem	8500	120400001409	5/7/2012	New	Wet Chem	10WT62	TBD
Autotitrator	Metrohm	905 USB Sample Processor	1814001009181	5/7/2012	New	Wet Chem	10WT63	TBD
Probe	Metrohm	905 USB Sample Processor	1281705	5/7/2012	New	Wet Chem	10WT63	TBD
JT Backer Speedisk Expanded Extraction Station	J.T. Baker	Speedisk Expanded Extraction Station	L02N23	2012	New	Wet Chem	10WET66	TBD
Desiccator	Sanplatec Corp	DryKeeper	N/A	2013	Unknown	Wet Chem	10WET68	TBD
Desiccator	Boekel	N/A	N/A	2013	Unknown	Wet Chem	10WET69	TBD
Desiccator	Boekel	N/A	N/A	2013	Unknown	Wet Chem	10WET70	TBD
Desiccator	Boekel	N/A	N/A	2013	Unknown	Wet Chem	10WET71	TBD
Desiccator	Boekel	N/A	N/A	2013	Unknown	Wet Chem	10WET72	TBD

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Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Desiccator	Boekel	N/A	N/A	2013	Unknown	Wet Chem	10WET73	TBD
Desiccator	Boekel	N/A	N/A	2013	Unknown	Wet Chem	10WET74	TBD
Desiccator	Boekel	N/A	N/A	2013	Unknown	Wet Chem	10WET75	TBD
Meter	Hach	HQ440d	120400069964	7/1/2013	New	Wet Chem	10WETE	TBD
Meter - probe	Hach	PHC20101	172612618021	7/1/2013	New	Wet Chem	10WETE	TBD
Meter - probe	Hach	PHC28101	200923042945	5/15/2020	New	Wet Chem	10WETE	TBD
Oven	Fisher Isotemp Oven	6905	614389-852	2014	New	Wet Chem	10WT77	TBD
Oven	Fisher Isotemp Oven	6905	614389-853	2014	New	Wet Chem	10WET78	TBD
Hot Plate	Presto	Tilt'n Drain Big Griddle	21-697	2014	New	Wet Chem	10WT81	TBD
Water Bath	Precision Scientific Water Bath	Coliform Incubator Bath	601061689	2015	Used	Wet Chem	10WT86	TBD
Oven	Fisher Scientific	151030521	41762572	2015	New	Wet Chem	10WT88	TBD
Fridge	Danby Designer	DBC120BLS	4315123638037	2016	New	Wet Chem	WC4	TBD
Hot Block	Environmental Express	N/A	4952CEC2361	2006	Used	Wet Chem	10MET03	TBD
COD Reactor	Environmental Express	B3000	2019CODW130	2019	New	Wet Chem	10WT91	TBD
Instrument	Lachat	QuickChem QC8500 Series 2	191200002263	2020	New	Wet Chem	10WT92	TBD
Autosampler	Lachat	Autosampler ASX560	101951A560	2020	New	Wet Chem	10WT92	TBD
Sep Funnel Tumbler	Analytical Testing Corp	DC-20	5046VMBP0026	2020	New	Wet Chem	10WT93	TBD
Sonicator	Fisher	FB11203	100417056	2020	New	Wet Chem	10WT94	TBD
Instrument	Metrohm	940 Professional IC Vario	1940000031101	2020	New	Wet Chem	10WT95	TBD
UV Lamp	UVP	UVL-56	not visible	unknown	Used	Wet Chem	10WT96	TBD
Water Bath	ThermoScientific	SC150	1121345701190806	2019	Used	Wet Chem	10WT97	TBD
Microscope	Fisher	3000014	20200104649	2021	New	Wet Chem	10WT99	At the instrument
Freezer	Elecwish	US-KC1008BK	UWC200271-2021/01-1042	2021	New	Wet Chem	WC6	At the instrument
Hotblock	CAI	SmartBlock 125i	129002	2022	Used	Wet Chem	10WT100	At the instrument
Conductivity meter	Fisher Scientific	06-662-6111704226	221128443	2022	New	Wet Chem	10WETH	At the instrument
Oven	Themofisher	Heratherm OMH 180	42947789	2022	New	Wet Chem	10WET79	At the instrument
Instrument	SEAL Analytical	AQ400	41004	2022	New	Wet Chem	10WTA5	At the instrument
Autotitrator	Metrohm	OMNIS	7613337331605	2022	New	Wet Chem	10WTA6	At the instrument

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7.5.2 PAS-Duluth MN

Equipment List: PAS-Duluth MN

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Water Filtration/DIW System (main)	Culligan	N/A	N/A	Unknown	Unknown	Glassware Cleaning	13DI1	Online
Fridge	Turbo Air	MSR23NM	MR23NM	Unknown	Unknown	Wet Chem 2	13DUL9	Online
Incubator	Labline CO2	3010	12	Unknown	Unknown	Wet Chem 2	13INC4	Online
pH Meter	Thermo Orion	Star Series	B07284	9/1/2015	Unknown	HCT	DUWT17	HCT Desk Drawer
pH /Conductivity Meter	Thermo	Star A215 Benchtop	X45992	9/24/2018	New	HCT	DUWT19	Online
Mercury Analyzer	Brooks Rand	Model III CVAFS	1103401	10/19/2017	Unknown	LL Hg	DUHG02	LL Hg Desk Drawer
Autosampler	Brooks Rand	Brooks Rand 17420	4936A14632	5/1/2018	Unknown	LL Hg	DUHG02	LL Hg Desk Drawer
Total Hg Purge and Trap	Brooks Rand	N/A	11078001	Unknown	Unknown	LL Hg	DUHG03	LL Hg Desk Drawer
Hg Speciation Purge and Trap	Brooks Rand	N/A	41107301	Unknown	Unknown	LL Hg	DUHG02	LL Hg Desk Drawer
Software	Hg guru	Version 4.1	N/A	Unknown	Unknown	LL Hg	DUHG03	Online
Distillation Block	Brooks Rand	Distillation Block AB	1021401	Unknown	Unknown	LL Hg	DUHG02	LL Hg Desk Drawer
Distillation Block	Brooks Rand	Distillation Block CD	1034401	Unknown	Unknown	LL Hg	13DT01	LL Hg Desk Drawer
Fridge	Magic Chef	MCBR445 W1	N/A	Unknown	Unknown	LL Hg	13DT02	Online
Fridge	Absocold	AR101MW1 3R	951005923	Unknown	Unknown	LL Hg	13DUL15	Online
Hood	N/A	N/A	N/A	6/23/2017	Unknown	LL Hg	13HOOD 5	Online
Hood	Hamilton	SAFEAIRE II	DLLKA-PTD	Unknown	Unknown	LL Hg	13HOOD 6	Online
Hood	ESCO	STL/04-EVC	2001-2764	Unknown	Unknown	LL Hg	DB-1	Online
Oven	Blue-M	MO1440A-1	S175-517150-SS	Unknown	Unknown	LL Hg	13OVN4	Online
Water Filtration/DIW System	Barnstead	D4641	1090090938202	Unknown	Unknown	LL Hg	13DI1-A	Online
Walk in Cooler	Carroll Coolers	N016898	CL-251150	3/7/2019	New	Sample Receiving	13DUL13	Online
Freezer	Arctic King	WHS-185C1WS	D80-28459101-17105-130313	10/26/2017	New	Storage Room	13FRZ2	Online
Autoclave	Market Forge	Sterilmatic STM-E	37827	1/1/2018	Unknown	Wet Chem	13CLV1	Online
Autoclave Pressure Gauge	Market Forge	BUILT-IN	37828	1/1/2018	Unknown	Wet Chem	13CLV1P	Online
Autoclave Temperature Gauge	Market Forge	BUILT-IN	37829	1/1/2018	Unknown	Wet Chem	13CLV1T	Online
Autodispenser	North Central Labs	DO-250	N/A	Unknown	Unknown	Wet Chem	13DSP1	Online
Autodispenser	SCIOLOGEX	DispenseMate Plus	JY16291	Unknown	Unknown	Wet Chem	13DSP2	Online
Distillation Unit Microblock	Environmental Express	EMD1920-106	2109	Unknown	Unknown	Wet Chem	13WETE	Online

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Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Distillation Unit Microblock	Lachat	1700-000	2000-209	Unknown	Unknown	Wet Chem	13WETF	Online
Freezer	Wood's	CO5BBA	01778768HJ	Unknown	Unknown	Wet Chem	13FRZ1	Online
Fridge	TurboAir	M3R47-2	M3R4L43095	Unknown	Unknown	Wet Chem	13DUL5	Online
Fridge	Gibson	RM18F5W X	NA	Unknown	Unknown	Wet Chem	13DUL6	Online
Fridge	TurboAir	TSR49	01749500MR	Unknown	Unknown	Wet Chem	13DUL7	Online
Hood	LABCONCO	031214227 H	728040010814	Unknown	Unknown	Wet Chem	13HOOD 1	Online
Hood	NA	NA	NA	Unknown	Unknown	Wet Chem	13HOOD 2	Online
Hot Plate	Thermolyne	Ciramec 3 HP 47135	61920359996	Unknown	Unknown	Wet Chem	13HTP1	Online
Hot Plate	Thermolyne	Ciramec 3 HP 47135-60	1073030511305	Unknown	Unknown	Wet Chem	13HTP2	Online
Hotblock (TKN)	Lachat	BD 40	TSLA1013511403	Unknown	Unknown	Wet Chem	13HB03	Online
Hotblock (TKN)	Seal Analytical	BD 50 Block	STU6U00860	Unknown	Unknown	Wet Chem	13HB04	In rack by 13WET7
Hotblock (TKN)	Lachat	BD 40 Block	HTLC1015510459	Unknown	Unknown	Wet Chem	13HB05	Online
Incubator	LabLine	460NS	0469	Unknown	Unknown	Wet Chem	13INC3	Online
BOD Incubator	Thermo	Isotemp	300168083	10/19/2017	New	Wet Chem	13IB12	In drawer under 13BOD1
BOD Incubator	Thermo	Precision	601111536	Unknown	Unknown	Wet Chem	13IB13	TBD
Incubator	Precision Scientific	66551	9209-113	5/1/2018	Used	Wet Chem	13INC7	Online
Lachat	Hach	8500	5010000097	Unknown	Unknown	Wet Chem	DUWT05	Online
Lachat Autosampler	Hach	ASX 520	010591A520	Unknown	Unknown	Wet Chem	DUWT06	Online
LDO Meter/Probe	Hach	HQ30d Flexi	121000079722	Unknown	Unknown	Wet Chem	DUWT13	Online
Microscope	American Optical Corp	Forty	814602	Unknown	Unknown	Wet Chem	13MC01	Online
Muffle Furnace	Lindberg	51442	899152	Unknown	Unknown	Wet Chem	10MF1	Online
Oven	VWR	1370G	1200600	Unknown	Unknown	Wet Chem	13OVN1	Online
Oven	Precision Scientific	Thelco #28	N/A	Unknown	Unknown	Wet Chem	13OVN2	Online
Oven	ThermoFisher	Cat #151030508	42094122	6/23/2017	Unknown	Wet Chem	13OVN5	Online
Oven	Shel Lab	SM05	4052114	VM to DUL 5/21/21	Good	Wet Chem	13OV06	TBD
Oven	Fisher	100 L	42130594	VM to DUL 2/25/21	Good	Wet Chem	13OV07	TBD
pH Meter	Orion	720A	13043	Unknown	Unknown	Wet Chem	DUWT18	In Drawer by pH supplies
pH pen	Sper Scientific	850051	143496	9/1/2016	Unknown	Wet Chem	13WET11	In Drawer under 13WET5
pH pen	Eutech Instruments	pHTestr 30	68X546501	3/7/2019	Unknown	Wet Chem	13WET13	In Drawer under 13WET6

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Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
pH/Conductivity Meter	Orion	A215	X37428	8/1/2017	New	Wet Chem	DUWT20	In Drawer by pH supplies
QuantiTray Sealer	IDEXX	2x/89-10894-00	01174	Unknown	Unknown	Wet Chem	13QT1	Online
Shaker	Labline Instruments	1345	10021791	Unknown	Unknown	Wet Chem	13SH1	Online
Sonicator	VWR	Auqasonic 50-T	N/A	Unknown	Unknown	Wet Chem	13SON1	Online
Spectrophotometer UV VIS	Thermo	9423AQ210 0E	HEDN238001	Unknown	Unknown	Wet Chem	DUWT08	Online
Spectrophotometer UV VIS	Hach	DR 3900	1648363	Unknown	Unknown	Wet Chem	DUWT09	Online
Sterilizer	EZE	N/A	N/A	Unknown	Unknown	Wet Chem	13STL1	Online
UV Lamp	UVP, Inc.	UVGL-25	691	Unknown	Unknown	Wet Chem	13UVL1	Online
UV Lamp	UVL	UVGL-58	OCT-2011	Unknown	Unknown	Wet Chem	13UVL2	Online
Water Filtration/DIW System	Barnstead	Nanopure II	N/A	Unknown	Unknown	Wet Chem	13DI1-B	Online
Color Test Kit	Hach	co-1	LOT#A8068	Unknown	Unknown	Wet Chem 2	DUWT24	Online
Evaporator for SPE System	Horizon Technology	Speed Vap III	08-0701	9/1/2015	Unknown	Wet Chem 2	13VAP01	Online
Hood	Kewaunee	N/A	N/A	9/1/2015	Unknown	Wet Chem 2	13HOOD 3	Online
Hood	Kewaunee	N/A	N/A	9/1/2015	Unknown	Wet Chem 2	13HOOD 4	Online
pH Meter	Orion	301	43996	Unknown	Unknown	Wet Chem 2	DUWT23	Online
SPE StepSaver 7-station Funnel	Environmental Express	Cat#G1106	N/A	6/14/2016	Unknown	Wet Chem 2	13SPE1	Online
SPE StepSaver 7-station Funnel	Environmental Express	Cat#G1106	N/A	6/14/2016	Unknown	Wet Chem 2	13SPE2	Online
Dessicator	Labconco	55300	171400	Unknown	Unknown	Wet Chem	12Des1	TBD
Dessicator	Labconco	55300	232878	Unknown	Unknown	Wet Chem	12Des2	TBD
Dessicator	Glass	N/A	N/A	Unknown	Unknown	Wet Chem	12Des3	TBD
Dessicator	Fisher	Metal	N/A	Unknown	Unknown	Wet Chem	12Des4	TBD
Dessicator	Boekel	Metal	N/A	Unknown	Unknown	Wet Chem	12Des5	TBD
Dessicator	Plas Labs	Plexi glass	N/A	Unknown	Unknown	Wet Chem	12Des6	TBD
Dessicator	Plas Labs	Plexi glass	N/A	Unknown	Unknown	Wet Chem	12Des7	TBD
Dessicator	Plas Labs	Plexi glass	N/A	Unknown	Unknown	Wet Chem	12Des8	TBD
Dessicator	SanPlatec	Dry Keeper	N/A	Unknown	Unknown	Wet Chem	12Des9	TBD
Rotator	Labline	1345	1002-1791	Unknown	Unknown	Wet Chem	12RTR1	TBD
Centrifuge	Sorvall	RT6000B	N/A	Unknown	Unknown	Wet Chem	12CFG2	TBD
Lachat	Lachat	QC 8500 Series 2	181200002196	1/7/2019	New	Wet Chem	DUWT06	TBD
Lachat reagent pump	Lachat	RP-150 Series	L18002784	Unknown	Unknown	Wet Chem	DUWT06	TBD

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Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Autodiluter	Lachat	PDS 200 Precision Diluter	181200000877	Unknown	Unknown	Wet Chem	DUWT06	TBD
Autosampler	Lachat	ASX-580 XYZ	111839A560	Unknown	Unknown	Wet Chem	DUWT06	TBD
Hardware	Dell	3035	N/A	Unknown	Unknown	Wet Chem	DUWT06	TBD
Software	Omnion	FIA Data System	N/A	Unknown	Unknown	Wet Chem	DUWT06	TBD
Lachat	Lachat	Lachat Quikchem 8500 Series 2	10070000129	Unknown	New	Wet Chem	DUWT07	TBD
Lachat Reagent Pump	Lachat	RP 150 Series	A82000-1961	Unknown	Unknown	Wet Chem	DUWT07	TBD
Autosampler	Cetac	ASX-500 Model No 510	010025ASX	Unknown	Unknown	Wet Chem	DUWT07	TBD
Hardware	Hewlett Packard	Compaq	N/A	Unknown	Unknown	Wet Chem	DUWT07	TBD
Software	Omnion	FIA Data System	N/A	Unknown	Unknown	Wet Chem	DUWT07	TBD
Ion Chromatograph	Metrohm	930 Flex IC	1930100006132	Unknown	New	Wet Chem	DUWT04	TBD
Regenerant Dispenser	Metrohm	IC-05	N/A	Unknown	Unknown	Wet Chem	DUWT04	TBD
Autosampler	Metrohm	850 Sample Processor	1858002005627	Unknown	Unknown	Wet Chem	DUWT04	TBD
Hardware	Dell	N/A	CBDUC284-70821-553-OGIP	Unknown	Unknown	Wet Chem	DUWT04	TBD
Software	Metrohm	IC Net 2.3	N/A	Unknown	Unknown	Wet Chem	DUWT04	TBD
Ion Chromatograph	Metrohm	881 Advanced Compact IC	1881000122119	Unknown	New	Wet Chem	DUWT03	TBD
Regenerant Dispenser	Metrohm	800 Dosino	N/A	Unknown	Unknown	Wet Chem	DUWT03	TBD
Autosampler	Metrohm	Model 858 Advanced Sample Processor	N/A	Unknown	Unknown	Wet Chem	DUWT03	TBD
Hardware	Dell	N/A	N/A	Unknown	Unknown	Wet Chem	DUWT03	TBD
Software	Metrohm	IC Net 2.3	N/A	Unknown	Unknown	Wet Chem	DUWT03	TBD
Ammonia Micro Distillation Equipment	Lachat	Lachat MicroDist	081200001033	5/1/2009	New	Wet Chem	12DST1	TBD
TKN Block Digester	Lachat	BD-40	TSLA1013511403	8/4/2017	New	Wet Chem	12TKN2	TBD
Autotitrator	ManTech	TitraSip Sample Module	PC 1300 475	Unknown	New	Wet Chem	DUWT01	TBD
Autosampler	ManTech	AutoMax 73 Sampler	PC 1000-681	Unknown	Unknown	Wet Chem	DUWT01	TBD
Buret Module	ManTech	Burivar 1/2 Buret Module	PC 1104-00	Unknown	Unknown	Wet Chem	DUWT01	TBD
Hardware	Hewlett Packard	Prodesk	N/A	Unknown	Unknown	Wet Chem	DUWT01	TBD
Software		PC Titrate for	N/A	Unknown	Unknown	Wet Chem	DUWT01	TBD

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Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
		Windows v.3						
pH meter	Orionstar	A215	X27234	Unknown	Unknown	Wet Chem	12WETG	at instrument
Turbidimeter	Orion	AQ3010	3494427	10/14/2017	Unknown	Wet Chem	12WETF	at instrument
Autoclave	Tuttnaur/Brinkman	3545 EP	2105018	Unknown	Unknown	Wet Chem	12CLV2	TBD
BOD incubator	Fisher	Model 3720	300007704	Unknown	New	Wet Chem	13IB10	TBD
BOD incubator	Fisher	Model 3720A	300064399	2/26/2016	New	Wet Chem	13IB11	TBD
Drying Oven	Shel Lab	Model SM05	0405Z114	Unknown	Unknown	Wet Chem	13OV06	TBD
Drying Oven	Fisher	Model 100L	42130594	Unknown	Unknown	Wet Chem	12OV06	TBD
TOC Analyzer	OI Corporation	1030	M129732449E	Unknown	Unknown	Wet Chem	DUWT10	at instrument
TOC Autosampler	OI Corporation	1088 AS	E129788451	Unknown	Unknown	Wet Chem	DUWT10	at instrument
Autosampling Module	OI Corporation	N/A	621290637-92120	Unknown	Unknown	Wet Chem	DUWT10	at instrument
TOC Analyzer	OI Corporation	N/A	A1129733824	Unknown	Unknown	Wet Chem	DUWT12	at instrument
IR Detector	OI Corporation	1030	2A0002T	Unknown	Unknown	Wet Chem	DUWT12	at instrument
TOC Analyzer	OI Corporation	1030	P407730312P	Unknown	Unknown	Wet Chem	DUWT11	at instrument
TOC Autosampler	OI Corporation	1088 AS	N/A	Unknown	Unknown	Wet Chem	DUWT11	at instrument
IR Detector	OI Corporation	1030	B622737366	Unknown	Unknown	Wet Chem	DUWT11	at instrument
Pass through fridge	Continental Refrigerator	3RE-PT	16149751	Unknown	New	Sample Receiving	13DUL19	TBD
Pass through fridge	Continental Refrigerator	3RE-PT	16149752	Unknown	New	Sample Receiving	13DUL20	TBD
Fridge	SubZero	249R	234547	Unknown	Unknown	Wet Chem	13DUL21	TBD
Fridge	Hotpoint	HPS15BTH MLWW	FM726711	1/25/2022	Used	Wet Chem	13DUL22	TBD
Micro Incubator	Fisher	3720A	300088990	Unknown	New	Microbiology	13INC8	TBD
Mercury Analyzer	Brooks Rand	Model III CVAFS	12032101	2022	New	LL Hg	DUHG03	at instrument
Mercury Autosampler	Brooks Rand	MERX	6046A62073	2022	New	LL Hg	DUHG03	at instrument
Fecal Water Bath	Thermo Fisher Scientific	TSCOL35	300534649	2022	New	Wet Chem	13INC10	TBD
Freezer	Insignia	NS-CZ70WH0	22B24A01197	2022	New	LL Hg	13FRZ3	TBD
Fridge/Freezer	Insignia	NS-UZ14SS0	22E27C01172	2022	New	LL Hg	13DUL23	TBD

7.5.3 PAS-Virginia MN

Equipment List: PAS-Virginia MN

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Bacteria Incubator	Shel Lab	1545	11052906	2007	Unknown	VM micro	12IB01	TBD

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Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Coliform Incubator Bath	Thermofisher	253	202682-185	2007	New	VM micro	12WB02	TBD
Bacteria Incubator	Shel Lab	1520	N/A	1996	New	VM micro	12IB02	TBD
Coliform Incubator Bath	Precision Scientific	183	9205-008	Unknown	Unknown	VM micro	12IB05	TBD
Microscope	National Optical	N/A	446TBL-10	2007	New	VM micro	12MC01	TBD
UV Lamp	Spectroline	EA160	1594366	12/19/2019	Unknown	VM micro	12UV02	TBD
Refrigerator 2R	Sanyo	SR-362OK	051105496	Unknown	Unknown	VM	N/A	TBD
Refrigerator 3	True Mfg. Co	T-49	1-2953805	Unknown	Unknown	VM	N/A	TBD
Refrigerator 5	True Mfg. Co	T-49	1-3060851	Unknown	Unknown	VM	N/A	TBD
Refrigerator 8	True Mfg. Co	T-35	1-3016399	Unknown	Unknown	VM	Refrigerator 8	TBD
Refrigerator 10	Gibson	T-35	BA31823513	Unknown	Unknown	VM	VM SR freezer	TBD
Refrigerator 12	Beverage - Air	9029136	KR481AS	Unknown	Unknown	VM	Refrigerator 12	TBD
Refrigerator 13 (Walk-in)	US Cooler	N/A	29716	Unknown	Unknown	VM	N/A	TBD
QuantiTray Sealer Plus	IDEXX	QuantiTray Sealer Plus	QTP13222602145	2022	New	VM micro	12QTS2	TBD

8.0 ADDENDUM: PROGRAM REQUIREMENTS

Section 8.0 provides additional requirements the locations covered by this manual are required to follow when performing work under the program. Only requirements that are not covered by the main body of the manual are listed in addendum.

8.1 DoD/DOE

PAS-Minneapolis MN 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

In addition to the requirements specified in Section 4.2.5, 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 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., $2X DL \leq LOD \text{ Spike} \leq 4X DL$). 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 SOP ENV-SOP-MIN4-0163 *Determination of LOD and LOQ*.

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 ENV-SOP-MIN4-0163 *Determination of LOD and LOQ*.

Section 5.4.7: Control of Data

The laboratory will ensure 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 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 SOP ENV-SOP-MIN4-0090 *Reagent Water Quality* or applicable test method SOP.

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 consult 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 QMS.

8.2 Ohio VAP

PAS–Minneapolis maintains accreditation for Ohio's Voluntary Action Program (VAP).

This addendum outlines additional policies and processes established by the laboratory to maintain compliance with Ohio's Voluntary Action Program (VAP). Specific requirements outlined in Ohio Administrative Code (OAC) 3745-300-04 include additional program-specific requirements for laboratories that perform analytical testing services for Ohio VAP and which must be followed for Ohio VAP projects.

This addendum is used in conjunction with the main body of the quality manual and with standard operating procedures (SOPs) and other quality management documents used to carry out activities. Only program requirements for the quality management system that are more stringent than the content of the main body of the manual are listed in this addendum.

In addition to the requirements outlined in the main body of the quality manual; the laboratory's procedures for implementation will also include the following:

Section 5.4.5.3.3 Limit of Detection (LOD)

A valid MDL must be in place prior to sample analysis. MDLs must be spiked at or below the reporting limit and will not be accepted if it was spiked higher than the reporting limit.

Section 5.5.2.2 Analytical Instrument Calibration

Samples must be reanalyzed to obtain results within the linear range unless there is insufficient sample volume for reanalysis.

Section 5.6.4.2 Reference Materials

The use of expired standards is prohibited even if they can be verified, with the exception of air standards that are revalidated against unexpired reference material or recertified by the vendor (documentation is required to be kept on file).

Section 5.8.3.2 Sample Acceptance Policy

- a. The narrative for any report that includes qualified data must also include a discussion of any bias in the results when requirements outlined in the SOP cannot be performed, for example: insufficient volume for re-extraction/re-analysis, holding time exceedances, and incorrect preservative.
- b. The case narrative must also include, at a minimum, discussion of any issues that impact the quality of the data with sample receipt, sample processes, or sample analyses.

Section 5.9.1: Quality Control

- a. For Ohio VAP projects, the laboratory must minimize the use of qualified data. The laboratory must make every effort to take the appropriate corrective actions and resolve any anomalies prior to reporting. When requirements outlined in the SOP cannot be performed, the narrative for any report that includes qualified data must also include a discussion of any bias in the results.
- b. In the event of a method blank having any reportable contamination, the laboratory is required to reanalyze the associated samples and the method blank if there is sufficient sample remaining. Acceptable method blanks are those that are free of contamination below the reporting limit. If the method blank fails, appropriate corrective actions may include flagging, elevating reporting limits, or re-preparation of the entire batch, including re-digestion, re-distillation, or re-extraction, as appropriate.
- c. In the event of LCS failures, the laboratory is required to reanalyze the associated samples and the LCS for all target compounds if there is sufficient sample remaining. The laboratory must make every effort to take the appropriate corrective actions and resolve any anomalies regarding LCS's and the MS may not be used in place of passing LCS. If the LCS fails, appropriate corrective actions may include re-preparation of the entire batch, including re-digestion, re-distillation, or re-extraction, as appropriate.
- d. MS/MSDs are optional and will be directed by the Certified Professional. In the case of MS/MSD failures, the laboratory is required to reanalyze the associated samples only when the associated LCS also fails acceptance criteria and if there is sufficient sample remaining. When an LCS is acceptable and the MS results are outside of criteria, and no system anomaly is detected, the samples will be reported with appropriate data qualifiers indicating matrix interference.

- e. Sample duplicates are optional and will be directed by the Certified Professional. In the case of duplicate samples exceeding the RPD criteria found in applicable analytical SOPs, the laboratory is required to reanalyze the associated sample and duplicate as long as no sampling error was detected if there is sufficient sample remaining. If the sample and duplicate still do not agree, a comment would be made stating there may be sample non-homogeneity.
- f. Surrogates are not evaluated for Ohio VAP samples analyzed via EPA Method TO-15.
- g. Samples with internal standard that are outside of method criteria must be reanalyzed to confirm sample matrix effect.

Section 5.8.5: Sample Disposal

All documents and data prepared or acquired in connection to VAP work must be retained for a period of 10 years after the data of reporting. After 10 years, if the laboratory wishes to dispose of the records, the laboratory must notify the VAP agency by certified mail of such intent and provide the agency an opportunity to request the materials from Pace. The documents must not be disposed of until notification has been received in response to the Pace request for disposal.

Section 5.10.3 Test Reports: Supplemental Items

- a. Affidavits that summarize any exceptions to what has been reported, including but not limited to, itemizing any analytes or methods that the laboratory is not approved for under the VAP program must be prepared by project, notarized, and submitted with each final report. Any analytes reported that are not part of a scope of accreditation or approval program must be clearly identified as such on the final report.
- b. The report must be accompanied by a copy of a sample receipt form that records, at a minimum, the following information:
 - i. Temperature of samples when received by the laboratory if the method requires monitoring.
 - ii. Date and time samples were received by the laboratory.
 - iii. Notation of whether holding times specified in standard operating procedures for sample preparation and analysis were exceeded.
 - iv. Any exceptions or special instructions for sample handling, analysis, or reporting.
 - v. Notation of whether samples have appropriate labeling, such as the date and time of sample collection and a sample identification notation.
 - vi. Notation of whether sample containers contain appropriate sample preservatives, if applicable.
 - vii. Description of the general condition of sample containers, including whether any containers are damaged or improperly filled.



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ENV-SOP-MIN4-0008 - Sample Management

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Janielle Ward (007319)	Manager - Quality	23 Nov 2021, 06:10:23 PM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Julie Bowser (007380)	Manager	23 Nov 2021, 04:49:13 PM	Approved
Adam Haugerud (005828)	General Manager 2	23 Nov 2021, 05:38:11 PM	Approved



TEST METHOD STANDARD OPERATING PROCEDURE

TITLE: Sample Management

ISSUER: Pace ENV – Minneapolis – MIN4

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1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes the laboratory procedure for the receipt, login, storage, and disposal of samples received by Pace Analytical Services.

Personnel: The policies and procedures contained in this SOP apply to all personnel involved in the receipt, login, storage, and disposal of samples.

The Pace ENV policy F-All-C-006 *Sample Acceptance Policy* (current version or equivalent replacement) contains the guidelines for acceptable sample conditions. Any deviation from these guidelines requires detailed documentation within the report, usually as a footnote, or on the chain-of-custody (COC), or Sample Condition Upon Receipt (SCUR) form and may require client contact.

2.0 SUMMARY OF METHOD

Samples are delivered to the laboratory via several delivery mechanisms. Samples received are checked for adherence to Pace ENV policy F-All-C-006 *Sample Acceptance Policy* (current version or equivalent replacement) with any discrepancies noted. Discrepancies are communicated to the client if necessary for their acknowledgement and decision making.

The Laboratory Information Management System (LIMS) assigns all samples with a unique sample number and manages the analyses assigned to each sample.

Samples are labeled with the appropriate information and staged in refrigerated sample storage coolers if temperature preservation is required or possibly stored on open shelves for samples not requiring sub-ambient temperature preservation. Samples will remain under these conditions until prepared and/or analyzed. Samples received under United States Department of Agriculture (USDA) protocols need to be stored separately (please refer to the lab's Regulated Soils SOP, if applicable).

Samples and associated sub-samples (digestates, extracts, etc.), with the exception of Air cans, are retained for 21 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

3.0 INTERFERENCES

Samples may be prone to cross contamination from others within the same delivery group or from other client projects. The sample receiving personnel must make every effort to minimize cross-contamination.

Preservation checks are one of the most likely situations where cross-contamination may occur. Materials used in the process must be specific to each sample and may not be used for multiple samples or multiple containers of the same sample.

Samples are stored under specific conditions and in specific locations, typically per the requirements of the analytical method. However, consideration must be given to samples that are uniquely different from others. Samples that are anticipated to be severely contaminated must be segregated from others in anticipation that the high levels of contaminants may cross-contaminate others in close proximity. USDA samples must also be distinctly segregated for storage.

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4.0 DEFINITIONS

Refer to the Laboratory Quality Manual for a glossary of common laboratory terms and definitions.

Chain-of-Custody (COC) – Form used to record the field identification of samples collected, analyses requested, date and time of collection, sample preservation used, and traceability of samples from time of collection until delivery to the laboratory. This is a legal document.

Laboratory Information Management System (LIMS) – Computer system used to manage the flow and traceability of environmental samples and associated data within the laboratory.

Matrix – The bulk characteristics of a sample. See table below.

NELAC/TNI defined matrix	Corresponding EPIC Pro matrices
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.	Air (AR)
Aqueous: any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, ground water effluents, and TCLP or other extracts.	Water (WT)
Biological tissue: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.	Tissue (TS) or Tissue Dry (TD)
Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined.	Oil (OL) or Other (OT)
Drinking Water: any aqueous sample that has been designated a potable or potentially potable water source.	Drinking Water (DW)
Non-aqueous liquid: any organic liquid with < 15% settleable solids.	Other (OT)
Saline/Estuarine: any aqueous sample from an ocean or estuary, or other saltwater source such as the Great Salt Lake.	Water (WT)- not assigned as a separate matrix.
Solids: includes soils, sediments, sludges, and other matrices with > 15% settleable solids.	Solid (SL)
(No corresponding matrix to wipes; wipes would be included in with solids)	Wipe (WP) or Swab (SW)

Safety Data Sheet (SDS) – Contains information on chemicals used in the laboratory.

Sample Custody – A sample is considered to be in someone's custody if:

- It is in one's physical possession;
- It is in someone's view, after being in someone's physical possession;
- It is kept in a secured area, restricted to authorized personnel only.

Sample Condition Upon Receipt (SCUR) – Form used to record the condition of samples received in the laboratory.

Sample Receipt Form (SRF) – Form generated by LIMS system after a project is logged in. Contains sample and project information.

UN Number - Identification numbers preceded by the letters UN are associated with proper shipping names considered appropriate for international and domestic transportation. These shipping names along with the identification numbers can be found the Federal Register, 49 CFR 172.101.

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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 handling 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.

All personnel involved in sample management are responsible for complying with OSHA and DOT regulations. These regulations pertain to the safe handling and/or shipping of the chemicals specified in this procedure. Refer to the Sample Control Supervisor for any questions or concerns related to the safe handling and shipment of hazardous materials.

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 will provide containers for the collection of samples upon client request for analytical services. Bottle kits are prepared in accordance with laboratory SOP ENV-SOP-MIN4-0009 *Bottle Preparation* (current version or equivalent replacement).

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.

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with this SOP. Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are stored at the appropriate temperature, per the method specific SOP.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 21 days from date of final report and then disposed of in accordance with Federal, State, and Local

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regulations.

7.0 EQUIPMENT AND SUPPLIES

7.1 Supplies

Equipment/Supplies	Description	Vendor/Item #
Sample Labels	Thermal transfer 3x1 rolls	Thermal Transfer 1700
Thermometers	Infrared, digital, NIST traceable	Grainger or equivalent
Sample storage cooling units	Capable of holding required storage temperatures	Walk-in: Carroll Coolers, Imperial Brown, Norlake, or equivalent. Refrigerator/freezer: Frigidaire, Keystone, Whirlpool, or equivalent.
COC forms	Corp. supplied chain of custody	N/A
SCUR forms	Sample condition upon receipt form	Pace forms ENV-FRM 0113, 0142, 0149, 0150, 0151, 0152 (current version or equivalent replacement)
pH paper	Hydriion	Narrow Range
Label Printer	Zebra GK420t	CDW or equivalent
LIMS computer system	EPIC Pro	Epic Pro
Disposable pipettes	Inert, single use pipette	Fisher Scientific or equivalent
Sample containers	Single use, pre-preserved per method	C&G Containers or equivalent
Residual chlorine strips	For UCMR, capable of measuring 0.1mg/L of chlorine	Hach or equivalent
Narrow range pH paper	For UCMR, capable of distinguishing pH of 7.9 from 8	Whatman or equivalent
Narrow range pH paper	For hexavalent chromium, capable of distinguishing pH between 9.3-9.7	Whatman or equivalent
Lead acetate paper	H ₂ S detection paper	Fisher Scientific or equivalent
Temperature blank	60mL plastic	C&G Containers or equivalent

8.0 REAGENTS AND STANDARDS

8.1 Reagents

8.1.1 All reagents used in this procedure must be labeled with:

- 8.1.1.1** Laboratory reagent identification number;
- 8.1.1.2** Unless otherwise noted, the name and concentration of the reagent;
- 8.1.1.3** Date the reagent was received, opened, and as needed, prepared;
- 8.1.1.4** Person preparing reagent; and
- 8.1.1.5** Expiration date.

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8.1.2 Types of Reagents

Reagent	Chemical Formula	Concentration
Glacial Acetic Acid (PFAS-NPW)	C ₂ H ₃ CO ₂ H	HPLC grade
Hydrochloric Acid (O&G)	HCl	1:1
Hydrogen Peroxide Solution (Cyanide)	H ₂ O ₂	3%
Nitric Acid (Metals)	HNO ₃	Concentrated
Sodium Hydroxide (Cyanide, pH, Pesticides-base, PFAS-NPW)	NaOH	50% or Pellets; 10 mL for PFAS NPW.
Sodium Sulfite (BOD, cBOD)	Na ₂ SO ₃	0.25 N to 1 L
Sodium Thiosulfate Solution (Cyanide, Nitrate, Dioxin-DW, SVOCs)	Na ₂ S ₂ O ₃ ·5H ₂ O	0.02 N; 80 mg for Dioxin-DW; 80 mg per 1 L for SVOCs
Sulfuric Acid (O&G, PCBs-acids/neutral, Pesticides-acids/neutral, TOC)	H ₂ SO ₄	Concentrated
Trizma (PFAS DW)	NH ₂ C(CH ₂ OH) ₃	1.25 g every 250 mL
Zinc Acetate Solution (Sulfide)	Zn(CH ₃ CO ₂) ₂	Lab prepared: 2N, in preserved bottle. Dissolve 110 g (CH ₃ COO) ₂ Zn·2H ₂ O in 500 mL DIW to make 2N ZnOAc for preservation of samples.

8.1.2.1 For acids, bases, and other reagents obtained from other laboratory Departments, the information is recorded in that respective Department's reagent preparation log.

8.1.2.1.1 If these reagents are managed within the sample management, the Department must maintain its own reagent preparation log.

8.1.2.2 Some Pace laboratories use pre-preserved sample containers. In this case, documentation from the vendor must be maintained for bottle-ware and preservation traceability.

9.0 PROCEDURE

9.1 Equipment Preparation

9.1.1 Support Equipment

9.1.1.1 Thermometers, infrared thermometer (IR) guns, and other equipment used for measuring temperatures must be calibrated according per laboratory SOP ENV-SOP-MIN4-0161 *Support Equipment* (current version or equivalent replacement).

9.2 Sample Receipt

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The laboratory receives client samples via three major methods: mail/commercial delivery service, Pace Analytical courier/field services, and hand delivery.

For air sample receipt, see section 9.3.

9.2.1 COC Procedures

9.2.1.1 Courier – Pace laboratories use courier services that pick-up client samples on either a regular schedule or on an as-needed basis as communicated by Project Managers (PMs) or by the client.

9.2.1.1.1 When the client is present during courier pick-up, the client signs the COC relinquishing custody to the courier. The courier signs the COC as accepting the samples and provides the client with a copy of the COC. When the courier returns to the laboratory with the client samples, the courier signs the COC as relinquishing the samples to the lab.

9.2.1.1.2 If the client is not present during courier pick-up, the courier signs the COC as accepting the samples and leaves a copy of the COC (if it has carbon copies) for the client. If a client also has a sample log in use, the courier must sign and date the log when the samples are picked up. When the courier returns to the laboratory with the client samples, the courier signs the COC as relinquishing the samples to the lab. The date/time of delivery to the laboratory by the courier is the official date/time received by the laboratory (analogous to the official date/time of receipt by an outside commercial carrier or courier).

9.2.1.1.3 To ensure the sample security, the Pace courier vehicle is locked at each client pick-up location. If a courier will be shipping samples, they must ensure there is enough ice in the cooler and that contents are packaged properly before they ship samples to the appropriate lab.

9.2.1.2 Laboratory – The COC (see Attachment I for example) is signed immediately upon receipt of the samples from the client. If the client drops off the samples, a copy of the signed COC is given to the client at that time. If samples are received via commercial carrier or mail delivery, the COC should be signed immediately when the cooler or package is opened and ultimately placed in the project file. The delivery date and time is considered the date/time received.

9.2.1.3 Samples Dropped Off – Sample receiving personnel must review the COC for any evidence of rush turnaround requests, analyses with short hold times, or samples with very little hold time remaining. Projects that fall under these conditions must be given immediate attention. Once the samples are received and logged into the LIMS, the sample technician and PM will coordinate the notification and delivery of samples to the laboratory. The PM is notified of the rush or short via the subject line of the scanned paperwork (email) after sample log-in.

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9.2.1.4 Internal Chain-of-Custody – If the laboratory uses an internal chain-of-custody (ICOC) procedure, the PM must determine, prior to log-in, which projects require ICOC processing.

9.2.2 Sample Acceptance Policy

Copies of the Pace ENV policy F-All-C-006 *Sample Acceptance Policy* (current version or equivalent replacement) must be provided, in the form of a letter, fax, or e-mail to each client or sampler, as necessary. Samples are considered acceptable if they meet the criteria listed in the Policy. This form is accessible from the “Resources” section in PacePort.

9.2.2.1 Some laboratories may have agreements with clients, regarding exceptions to the client contact requirements for deviations from the Policy. If a laboratory has such agreements, two conditions must be met:

9.2.2.1.1 The agreement must be a formal document showing client approval; and

9.2.2.1.2 The laboratory must qualify the final report as appropriate to their applicable regulatory bodies.

9.2.2.2 Wisconsin Samples: Samples that do not meet the criteria in the Policy will be rejected by sample management personnel. The sample management personnel will notify the PM, and the client will be notified before proceeding with login. If the client wishes to proceed with analysis, the PM will retain documentation of the request to proceed.

9.2.3 Measuring and Recording Temperature

9.2.3.1 Temperature Blank Present

9.2.3.1.1 Open the cooler and verify the temperature of the samples by taking the temperature of the temperature blank.

9.2.3.1.2 When using an IR gun, the temperature must be taken from an opaque surface such as the bottle label, approximately six inches away from the container. Measurements taken through a transparent surface (clear or amber glass) may not be reliable and must incorporate a specific temperature correction factor for that surface reading.

9.2.3.2 NO Temperature Blank Present

9.2.3.2.1 If there is no temperature blank in the cooler, measure the temperature of representative sample bottles.

9.2.3.2.1.1 If the contents of the coolers allow, four different temperatures should be taken, recorded on the SCUR exceptions form ENV-FRM-MIN4-0142 *Sample Condition Upon Receipt-SCUR Exception Form* (current revision or equivalent replacement) and averaged. The average temperature is then

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documented on the COC and SCUR.

9.2.3.2.1.2 The representative samples will reflect an “average” condition of the samples in the cooler and, depending on the manner in which they are packed, may not necessarily be in direct contact with the cooling material.

9.2.3.3 West Virginia Samples: The temperature must be taken of all samples in the cooler even if a temperature blank is present. If all samples are within 0-6° C it can be recorded as “all below 6° C”. If any samples are out of temperature, they must be noted on the SCUR.

9.2.3.4 Record the uncorrected and corrected cooler temperatures on the COC and/or laboratory form ENV-FRM-MIN4-0150 *Sample Condition Upon Receipt - MN SCUR* (current version or equivalent replacement). In addition, record the type of “ice” used for packing the cooler (e.g., wet ice, “blue ice”, gel packs, etc.).

9.2.3.5 If samples within a project are spread over multiple coolers and one or more of the coolers are outside of the temperature criteria, then the contents of the cooler must be itemized, and the samples and sample containers affected by the out-of-control temperature must be listed on the SCUR exceptions form ENV-FRM-MIN4-0142 *Sample Condition Upon Receipt-SCUR Exception Form* (current version or equivalent replacement) for qualification in the final report. This itemization must be retained in the project file for future reference. The out of temp containers will also be marked to indicate it was out of temp.

9.2.3.6 Unpack the cooler and COC. Organize the samples, grouped by client sample ID, according to the order on the COC. Review COC against samples to make sure the bottles received match the analysis requested. All anomalies must be recorded on the SCUR.

9.2.3.6.1 Care must be taken to minimize samples warming over the thermal preservation requirements for the samples received. Care should be taken with small containers such as 40 mL VOA vials as these samples will warm quicker than 1 L samples. Triage and log in should be performed in 20 minutes or less as a guideline, samples should be placed in the storage units or back in the cooler on ice if extend time is required to review the received materials for a project.

9.2.3.6.2 Discard any ice or water that remains in the cooler and the packing material used to secure the samples. Water or ice should be discarded down a drain that connects to the local sewer. Packing materials should be placed in the garbage. If a sample container was broken, the contents remaining in the cooler **MUST** be discarded in a manner consistent with the hazardous waste handling standard operating procedure.

9.2.4 USDA Regulated Samples

The cooler and all contents must be decontaminated. See laboratory SOP ENV-SOP-

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MIN4-0095 *Procedure for Handling of USDA Regulated Soils* (current version or equivalent replacement) for detailed information.

9.2.5 pH Verification

The pH of the sample must be verified on all preserved sample bottles requiring pH preservation (see section 9.2.5.2).

Duluth/Virginia – All pH verification check will be completed at the bench. See section 9.2.5.4 for additional information.

Minneapolis – All pH verification check will be completed upon receipt by sample management personnel, with the exception of some analyses. See section 9.2.5.3 for detailed information.

9.2.5.1 Samples should not be preserved without first contacting the PM.

9.2.5.1.1 PMs should verify client specific technical requirements to determine if the client needs to be notified before the addition of acid to samples. Some technical requirements also require the addition of acid to equipment blanks and field blanks received with the project.

9.2.5.2 General pH Preservation Requirements by Preservative

Sample Preservatives	Sample pH Requirement
Hydrochloric Acid (HCl)	must be < 2
Nitric Acid (HNO ₃)	must be < 2
Sulfuric Acid (H ₂ SO ₄)	must be < 2
Sodium Hydroxide (NaOH)	must be > 12
Zinc Acetate and Sodium Hydroxide (NaOH)	must be > 9

9.2.5.2.1 If the pH for a sample container that is supposed to be preserved is not within the required range, indicate the anomaly on the SCUR.

9.2.5.2.2 If a sample does not require preservation, check N/A in the applicable section of the SCUR.

9.2.5.3 pH Preservation Adjustments – Minneapolis

If a sample container does not meet the pH preservation required, the pH of the sample must be recorded on the SCUR exceptions form ENV-FRM-MIN4-0142 *Sample Condition Upon Receipt-SCUR Exception* (current revision or equivalent replacement). Additional preservative is added so that the preservative content is < 1% of the sample container volume. For example:

- For a 100 mL container – a maximum of 1 mL of preservative may be added;
- For a 250 mL container – a maximum of 2.5 mL of preservative may be added;
- For a 500 mL container – a maximum of 5 mL of preservative may be added;

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added;

- For a 1 L container – a maximum of 10 mL of preservative may be added.

EXCEPTIONS: The following analyses will have pH checked at the bench by the respective Department prior to analysis—sample containers **must not be opened** by sample management personnel:

- Coliform;
- Volatiles;
- Total Organic Carbon (TOC);
- Wisconsin Diesel Range Organics (WI-DRO);
- Oil and grease;
- Hexane extractable materials (HEM); or
- PFAS.

9.2.5.3.1 Open each preserved bottle. Use a new disposable pipette, a stirring rod, or another inert utensil to withdraw a small portion of the sample. Dispense the aliquot on a sample specific pH strip and check the pH.

9.2.5.3.2 The appropriate preservative is added to the sample container, the sample is mixed, and the pH is taken again. The new pH reading is recorded on the SCUR exceptions along with the initials of the person who takes the pH of the samples, the date/time it was added, the amount, type, lot number of the preservative added and if it was in or out of compliance after the addition of the preservative.

9.2.5.3.2.1 In addition, the sample container is marked via a label the preservative added, volume added, date, time, and initials of the technician.

9.2.5.3.2.2 For Metals analyses specifically, the laboratory must wait 24 hours after pH adjustment to pH < 2 before sample preparation can begin.

9.2.5.4 pH Preservation Adjustments – Duluth/Virginia

All pH verification will be completed at the bench by the respective Department

9.2.5.4.1 Upon receipt, sample management personnel will check the pH.

9.2.5.4.1.1 If a sample container does not meet the pH preservation required, the sample management personnel will add a red greater than sign (“>”) on the cap, denoting that the sample will need to be adjusted. Once the sample is at the bench, the analyst will adjust accordingly and record the adjustment.

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9.2.5.4.1.2 If a sample container meets the pH preservation required, no additional action is required by the sample management personnel.

9.2.6 Total Residual Chlorine Verification

Total residual chlorine must be verified at the time of receipt or at the bench, as required by the method or individual state regulatory agency for certain analyses:

Analyses Requiring Residual Chlorine Verification
Ammonia (NH ₃) by EPA 350.1
Biochemical Oxygen Demand (BOD) by HACH 10360
carbonaceous BOD (cBOD) by HACH 10360
Fecal Coliform by Colilert-18, MF
Cyanide by SM 4500-CN
Dioxin DW by1613B
HPC by Pour Plate, Simplate
Nitrate (NO ₃) by EPA 353.2
PBDE by EPA 1614
PCBs by EPA 1668
Organochlorine Pesticides and PCBs by EPA 8081, 8082
SVOCs by EPA 625, 625.1, 8270
Total Coliforms/E. coli bacteria by MF, Colilert, Quanti tray
PFAS DW (<0.1 mg/L)

9.2.6.1 Minneapolis: Only cyanide samples will be checked for residual chlorine verification upon receipt by sample receiving personnel.

9.2.6.1.1 Open the appropriate sample container. Utilizing a new disposable pipette, stirring bar or other inert utensil; withdraw a small portion of the sample. Dispense the aliquot on a sample specific residual chlorine test strip and tape the strip to the side of the container before verifying if it's present or not.

9.2.6.1.2 If any chlorine is detected, regardless of amount, note the information on the laboratory form ENV-FRM-MIN4-0150 *Sample Condition Upon receipt – MN SCUR* (current version or equivalent replacement).

9.2.6.2 Duluth/Virginia: All analyses will be checked at the bench.

9.2.7 Sample Discrepancies

Note any discrepancies pertaining to samples as defined per Pace ENV policy F-All-C-006 *Sample Acceptance Policy* (current version or equivalent replacement) on the COC or SCUR. Any discrepancies involving temperature, preservation, hold time, collection dates and times, sample volume, sample containers, and unclear analysis, must be reported to the PM as soon as possible.

9.2.8 Short Hold Times

Upon receipt of short hold samples, sample management will notify the respective

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Department. The sample is then accordingly per section 9.5.

Analyses with Hold Times Less Than 72 Hours		
Short Hold Time	Analyses	Details
15 minutes	Field Parameters	pH, Dissolved Oxygen, Residual Chlorine, Ferrous Iron
8 Hours	Fecal coliform	Up to 24 hours, per regulatory and/or method compliance.
8 Hours	Total Coliform (MPN, MF), Enterococci, Fecal Streptococci MPN, E. coli	Non-potable water only
8 Hours	Heterotrophic Plate Count (HPC)	
24 Hours	Hexavalent Chromium	
24 Hours	Conductivity EPA 120.1	Filtered within 24 hours
30 Hours	Total Coliform (Presence / Absence), E. coli	Drinking water
48 Hours	BOD, cBOD	
48 Hours	LL Mercury (lab filtered)	
48 Hours	Nitrate (unpreserved)	If preserved, reported as NO ₃ +NO ₂
48 Hours	Nitrite (unpreserved)	If preserved, reported as NO ₃ +NO ₂
48 Hours	Orthophosphate	
48 Hours	Settable Solids	
48 Hours	Turbidity	
48 Hours	VOCs – 624.1 (unpreserved)	
48 Hours	VOCs - Soils by Unpreserved EPA5035	Jars, Encores, Sleeves
48 Hours	Chlorophyll A	48 hours to filtration
48 Hours	Volatiles – Air TO-15, TO-15SIM	Tedlar bag or equivalent

9.3 Sample Receipt – Air

Air samples are received at the Air laboratory. When an air canister or Tedlar bag arrives to the facility, there are several important steps that are necessary to follow when logging in each sample.

9.3.1 Remove canister(s) and/or Tedlar bag(s) from box.

9.3.2 Record the condition and quantity of items received with the sample, including flow controllers, gauges, puff cartridges, filter paper, and individually certified containers on the laboratory form ENV-FRM-MIN4-0113 *Sample Condition Upon receipt (SCUR) – Air* (current version or equivalent replacement).

9.3.2.1 Also note the initial pressure upon receipt and the final pressure after adding nitrogen, if applicable.

9.3.3 Record the bottle type (i.e., Tedlar bag, canister, Air Filter, Thermal Distillation Tube, or puff cartridge) in the “bottle type” field.

9.3.4 TO-15 Analysis

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Upon receipt, the summa canister pressure of each sample is measured and recorded on the canister sample tag.

9.3.4.1 If the canister pressure is less than 5 psig, the canister pressure must be increased before analysis can occur.

9.3.4.1.1 Add clean nitrogen or helium gas to the sample canister. For a six-liter canister, 5 psig is the desired final pressure. A one-liter canister requires a final pressure of 10 psig for adequate sample volume for analysis.

9.3.4.1.2 Record the final canister pressure on the canister sample tag noting which gas was added. Also, note the information in the final analytical results report.

9.3.4.2 If a sample is collected into a Tedlar bag, the client has two days to get the bag to the facility for analytical testing.

9.3.4.2.1 Pace Analytical recognizes a two-day holding time for samples collected in Tedlar bags for TO15, and 14 days for fixed gases and TO3M, unless an alternative timeframe is specified per client specific QAPPs.

9.3.4.2.2 Upon receipt at the laboratory, the sample in the Tedlar bag is transferred into a batch certified, evacuated one liter SUMMA canister for analysis.

9.3.4.2.3 The sample is subsequently analyzed by the appropriate method within 30 days of transfer.

9.4 Sample Login

All samples received by the laboratory must be logged into the LIMS. Rush projects and/or projects with short holds should be prioritized. After these projects have been addressed, projects should be addressed on a first in, first out basis unless communication from the PM has been received instructing special handling of an incoming cooler.

9.4.1 Samples must be logged into the LIMS so the samples can be uniquely identified (laboratory sample identification numbers). These laboratory sample ID numbers are used to track the prep and analysis activities of the samples, as well as identify the sub-samples, digestates, extracts, and other sample byproducts. This laboratory code maintains an unequivocal link with the unique client field sample ID code assigned to each sample.

Reference laboratory guide ENV-GUI-MIN4-0005 *EPIC Pro Module 03: Sample Login* (current version or equivalent replacement) for additional information on how to login samples in LIMS.

9.4.1.1 Air Samples – Samples are logged into LIMS by the Air laboratory.

9.4.1.1.1 For summa canister and flow controllers, the associated identification number is entered into the “AUX” field, during the sample login on the “Container Login” screen in LIMS, per sample.

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9.4.2 Generate Sample Labels and Sample Receipt Form (SRF).

Once the samples are logged in the SRF is printed from LIMS to the e-reports folder. No hard copy is printed.

9.4.2.1 The COC is scanned to the PM.

9.4.2.2 Samples are labeled after they are logged in LIMS.

9.4.2.2.1 Attach the sample labels to the appropriate sample bottles.

9.4.2.2.2 Place labels so that the client sample ID and tared weights (if applicable) is still visible. If Client Sample ID does not match COC, any inconsistencies must be listed on the SCUR and an additional label should be placed on each sample container indicating the sample ID does not match the COC.

9.4.3 If any samples require analyses performed outside of the laboratory, prepare the samples for subcontracting according to the procedures detailed in laboratory SOP ENV-SOP-MIN4-0010 *Subcontracting Samples* (current version or equivalent replacement).

9.4.4 Review COC to SRF
9.4.4.1 The PM, Project Coordinator (PC), or designated Client Services personnel must review and verify the following information by comparing the COC to SRF. Some of this information may not be provided by the client and those fields should be left blank:

- Report Recipient;
- Invoice Recipient;
- Additional Report Recipient;
- PO #;
- Project Name;
- Project Number;
- Requested Due Date;
- Sample ID;
- Matrix;
- Collection Date & Time;
- Received Date & Time;
- Analysis: Double check compound lists;
- Certification / regulatory requirements
- Report format requirements
- Price;
- Region Codes;
- Work Region % Split (for Pace internal subcontracted work).

9.4.4.2 Refer to ENV-GUI-MIN4-0030 *Work Order Releasing* (current version equivalent replacement) for additional items to review at releasing.

9.5 Sample Storage
9.5.1 Once unpacked, samples will be logged into the LIMS in a timely manner and returned

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to appropriate storage conditions as soon as possible. Laboratories must make every effort to keep samples under the required thermal conditions during the login process. For the exceptional case where samples are not logged in the day they were received, they must be stored under appropriate temperature-controlled conditions until login takes place. In all cases, the sample temperatures must be taken as soon after receipt as possible (before samples are placed into storage) and the samples stored to maintain the required storage conditions while awaiting log-in.

NOTE: For client specific projects, samples must be kept in the cooler while being processed. If not kept in the cooler, the temperature must be checked and documented every 20 minutes during processing.

9.5.2 Once logged into the LIMS and labeled, samples are placed in the appropriate storage areas. Specific temperature requirements are outlined in the analytical methods, but general guidelines are outlined below:

9.5.2.1 Short Hold – Placed in the short hold storage area or delivered directly to the laboratory.

9.5.2.2 Biological Tissue – Staged by receiving date or project number on shelves in a freezer for all types of analyses.

9.5.2.3 Summa Canisters and Tedlar Bags – Stored on designated shelving at ambient temperature.

9.5.2.4 Volatiles (Aqueous) – Stored by receiving date or by project number in a segregated volatiles cooler. Associated trip blanks are stored with the samples.

9.5.2.5 Volatiles (Soil and Other Solid)

9.5.2.5.1 Samples received preserved in methanol are stored by receiving date or by project number in a segregated volatile cooler. Associated trip blanks are stored with the samples.

9.5.2.5.2 Samples received preserved with a stir bar, or deionized water and a stir bar, are stored by receiving date or by project number in a segregated volatiles freezer. The vials should be placed at an angle to avoid the vial breaking once it freezes and expands. Associated trip blanks are stored with samples. The date/time placed in the freezer should be recorded.

9.5.2.5.3 Samples received in 4 oz containers or similar bottle-ware must be brought down to the laboratory so they can preserve within 48 hours. In order to preserve these samples, it is necessary to collect a 5g aliquot of the sample and transfer it to a 40mL vial. One of the following preservation options must be utilized:

9.5.2.5.3.1 The 5 g aliquot is preserved with a stir bar, 5mL of deionized water and a stir bar, or 5mL of sodium bisulfate and a stir bar and stored in a freezer until analysis; or

9.5.2.5.3.2 Within 48 hours of collection in the field, the 5 g

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aliquot must be immediately extracted with 5mL of methanol and stored in a segregated volatiles cooler until analysis; or

9.5.2.5.3.3 Within 48 hours of collection in the field, the 5 g aliquot can be preserved with 10mL of deionized water and a stir bar, stored in a segregated volatile cooler and analyzed within 48 hours of collection.

9.5.2.5.4 Samples received in Encore samplers must be managed within 48 hours of collection by freezing the Encore or extruding it.

NOTE: If samples are not received within 48 hours of collection or are not received with enough time to process the samples correctly within 48 hours of collection, this must be noted in a way that will be visible on the final report (e.g., footnote in LIMS).

9.5.2.5.4.1 If extruding the sample into a 40mL vial containing a stir bar or a stir bar and 10mL of deionized water, then the sample is stored in the segregated volatile freezer until analysis.

9.5.2.5.4.2 If extruding the sample into methanol, then the sample is extracted within 48 hours of collection and the sample is stored in a segregated volatile cooler until analysis.

9.5.2.6 General Chemistry/Semi-Volatiles

9.5.2.6.1 Waters and other liquid samples are staged by receiving date or by project number on the shelves in the appropriate sample storage cooler.

9.5.2.6.2 Soils and other solid samples are staged by receiving date or by project number on the shelves in the appropriate sample storage cooler.

9.5.2.7 Metals Solids and Liquids – These samples are staged by receiving date or by project number on designated shelving in the laboratory or appropriate designated area. These samples may be stored at ambient temperature unless mercury in soils or hexavalent chromium analysis is needed.

9.5.2.7.1 If mercury or hexavalent chromium analysis will be performed, the samples are staged by receiving date or by project number in the appropriate sample storage cooler.

9.6 Internal Chain-of-Custody (ICOC)

9.6.1 When an ICOC is needed it must be generated out of LIMS. When an analyst removes samples from a storage unit, the ICOC form must be completed. The following items must be documented: laboratory sample ID, analyst initials, date and time samples are removed, and sample container type. Project number is optional and only necessary

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when it is needed to uniquely identify a specific sample container. Once the analyst is finished with the sample, the sample must be returned to the applicable storage unit. The analyst must again document the necessary information on the ICOC (date and time samples are returned to the storage unit and the analyst's initials). If the sample was entirely consumed, then document with the appropriate comment code.

- 9.6.2** Similar steps must be taken for sample by-products such as extracts, digestates, and leachates. Once a sample is prepared for analysis, sample custody of the sample by-product must be transferred to the appropriate analytical group sample storage unit. Analytical staff must document in the ICOC when removing and returning the sample by-products from and to the analytical sample storage location. If the sample by-product is entirely consumed during analysis, then document with the appropriate comment code.

9.7 Sample Retention and Disposal

- 9.7.1** If samples must be returned to customers, the laboratory must take special care to ensure that the samples are not damaged during any handling, testing, storing, or transporting processes. Before samples are returned, they must first be scanned out of LIMS and sent back under a chain-of-custody.
- 9.7.2** Samples may need to be retained longer than the normal sample retention time (21 days from final report). Reasons for this extended sample retention include: customer, program, or contract requirements so that samples can be retained in a secure location for the customers that is designated as a long-term storage area.
- 9.7.3** For disposal of unconsumed samples refer to laboratory SOPs ENV-SOP-MIN4-0098 *Waste Management and Handling* (current version or equivalent replacement) and ENV-SOP-MIN4-0095 *Regulated Soil Handling* (current version or equivalent replacement).

10.0 QUALITY CONTROL AND METHOD PERFORMANCE

10.1 Quality Control

- 10.1.1** For any sample received at the laboratory that does not meet the sample acceptance, hold time or preservation criteria, the client must be contacted by the PM and advised of the situation.
- 10.1.1.1** If the client instructs the laboratory to proceed with the analysis, all appropriate personnel/departments must be informed, and the client approval must be documented. This can be included on the SCUR, but the documentation should also be saved in the report folder. Data will be appropriately qualified.
- 10.1.1.2** The client may also instruct the laboratory to preserve the samples at the laboratory prior to proceeding with analysis. This must be documented on the SCUR, so it is noted in the final laboratory report. Save documentation in the report folder as well.
- 10.1.2** All supporting documentation related to sample custody must be retained by the laboratory. This includes: memorandums, fax transmissions, the original COC, all paperwork received with the COC, the completed SCUR and copies of email transmissions. Reference laboratory SOP ENV-SOP-MIN4-0184 *Data and Records Archival* (current version or equivalent replacement) for additional information on data

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retention.

10.1.3 Documenting Discrepancies During Receipt of Samples

10.1.3.1 The following are examples of client discrepancies that need to be documented on the appropriate paperwork (e.g., SCUR):

- Lost or Missing samples;
- Insufficient sample volume;
- Broken or missing bottles;
- Missing COC;
- Mislabeled bottles;
- Preservation error;
- Missing sample related details (date, time, sample type);
- Missed holding times;
- Coolers/samples received out of temperature compliance

10.1.3.2 Pace sample management discrepancies will be documented on the SCUR. Discrepancies attributable to errors and omissions on the part of the laboratory will be addressed and resolved through the formal corrective action process.

10.1.3.3 MS/MSD Container Volume – It is the PM's responsibility to add Matrix Spike/Matrix Spike Duplicate (MS/MSD) volume to bottle orders to ensure volume is available for quality control samples. Additional containers for MS/MSD will be added at a rate of two additional containers/10 samples/method for each project bottle kit requiring additional volume for MS/MSD. Examples:

10.1.3.3.1 20 samples add two additional containers per analytical method.

10.1.3.3.2 40 samples add four additional containers per analytical method.

10.1.3.3.3 60 samples add six additional containers per analytical method.

10.1.3.3.4 Etcetera.

10.1.3.4 Reference the "Method_Policy Master List" located in the "Minneapolis Lab QA Library" for a list of method(s) that require additional volume for MS/MSD analysis, if applicable.

10.2 Qualifications and Training

10.2.1 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, client service staff that process samples and projects must successfully demonstrate proficiency in executing the required task. Successful means the employee can perform the full extent of steps necessary to complete the process they are working on without error. Additional details on the procedures, policies, documentation, and tracking used for training activities can be found in laboratory SOP ENV-SOP-MIN4-0165 *Orientation and Training Procedures* SOP (current version or equivalent replacement).

10.3 Corrective Action

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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.

11.0 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.

12.0 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.

13.0 ATTACHMENTS

Attachment I – Example Chain-of-Custody

14.0 REFERENCES

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Pace Quality Assurance Manual- most current version.

TNI Standard, *Management and Technical Requirements for Laboratories Performing Environmental Analyses*, EL-V1-2009.

TNI Standard, *Management and Technical Requirements for Laboratories Performing Environmental Analyses*, EL-VI-2016-Rev.2.1.

Department of Defense (DoD) Quality Systems Manual- most current version.

SW-846, Test Methods for Evaluating Solid Waste Physical/Chemical Methods, USEPA, current revision.

American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 1995, Standard Methods for the Examination of Water and Wastewater, A.E. Greenberg, L.W. Clesceri, A.D. Eaton and M.A.H. Franson, eds., 19th ed., American Public Health Association, Washington D.C.

U.S. Environmental Protection Agency, 1983, Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

U.S. Environmental Protection Agency, 1988, Methods for Determination of Organic Compounds in Drinking Water, EPA/600/4-88/039, Environmental Monitoring Systems Laboratory, Cincinnati, Ohio.

Code of Federal Regulations- most recent version.

Pace Analytical Services, LLC – Master Method List, most current version.

15.0 REVISION HISTORY

This Version:

Section	Description of Change
ALL	Converted to new template and Consolidated to include Pace-Duluth and Pace-Virginia.
1.0	Deleted reference to Attachment I; and Added applicable doc number and name for sample acceptance policy.
2.0	Deleted reference to Attachment I; Added applicable doc number and name for sample acceptance policy; Replaced "maintained for a minimum...regulatory agency" with "retained for 21 days...regulations; and Deleted "Samples are disposed...requirements" for repetitiveness.
6.0	Updated sample retention from 30 days to 21.
7.1	Updated supplies table with missing info.
8.1.2	Updated the concentration info from 1:1 to concentrated for reagents, Nitric Acid and Sulfuric Acid; Added missing info for reagents; Added reference of applicable parameters for the reagents; Deleted non-applicable reagents that not used by the laboratory for pH and/or residual chlorine checks.
8.1.2.1	Replaced "laboratory departments, this information is located in the department" with "Departments, the information is recorded in that respective Department's".
8.1.2.1.1	Replaced "In the event" with "If";

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	Replaced "Sample Receiving group" with "sample management".
9.1.1.1	Added "infrared thermometer".
9.2.1.2	Replaced reference from Attachment II to Attachment I.
9.2.2	Deleted reference to Attachment I; Updated reference for sample acceptance policy; and Added info for where to access the sample acceptance policy.
9.2.3.2.1	Added document number and name reference for SCUR.
9.2.5	Replaced "see exceptions below" with "see section 9.2.5.2"; and Added info for Duluth/Virginia and Minneapolis.
9.2.5.3.1	Deleted "(except as noted below)".
9.2.5.3	Updated verbiage for exceptions.
9.2.5.4	New section added for Duluth/Virginia specific process.
9.2.6	Moved "Sample receiving...cyanide samples" to section 9.2.6.1; Deleted "Do not check...section 9.2.5.3"; Added Fecal Coliform to the table; Added HPC to the table; Added Total Coliforms/E. coli to the table; Added additional verbiage to clarify what the applicable analysis/parameter was for each method; and Deleted non-applicable analyses performed by the laboratory.
9.2.6.1.2	Added SCUR info for where chlorine detection should be documented.
9.2.6.2	New section added for Duluth/Virginia specific process.
9.2.7	Updated reference for sample acceptance policy.
9.2.8	Updated verbiage for clarity; Replaced "Sample Storage section of this SOP" with "section 9.5"; Updated the short hold time from 6 hours to 8 for analysis, Fecal coliform; Added info to details section for analysis, Fecal coliforms; and Added LL Mercury info.
9.3	Added "Air samples are received at the Air laboratory".
9.3.2	Added air SCUR document number and name reference.
9.4.1	Added "Reference laboratory guide...samples in LIMS".
9.4.1.1	New section added.
9.4.1.1.1	Updated AUX data verbiage for clarification.
9.4.3	Replaced "listed in the SOP...analytical services" with "detailed in laboratory SOP".
9.4.4.1	Added "certification/regulatory requirements"; and Added "report format requirements".
9.4.4.2	New section added.
9.7.1	Added "and sent back under a chain-of-custody".
9.7.2.	Replaced "45 days from sample receipt" with "21 days from final report".
9.7.3	Deleted "regarding waste handling and disposal"; and Updated verbiage for clarity.
10.1.1.1	Added "This can be included".
10.1.2	Replaced "Please contact...time frames required" with "Reference laboratory SOP...on data retention".
10.1.3.1.1	Added "Missing".
10.1.3.1.8	New section added.
10.1.3.1.9	New section added.
10.1.3.4	Updated reference for the method master list; and Added reference to where the list is maintained.
10.2.1	Updated verbiage and info to be consistent with training applicable for this SOP.
13.0	Deleted Sample Acceptance Policy; Deleted Example SCUR;

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	Deleted Procedure for Tedlar Bags; and Deleted SCUR Exceptions form.
14.0	Replaced "From F-MN-C-306" with "Master Method List".

This document supersedes the following document(s):

Document Number	Title	Version
ENV-SOP-MIN4-0008	Sample Management	02
ENV-SOP-DUL1-0001	Sample Management	01

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Attachment I – Example Chain-of-Custody Form

Section A
 www.pacelabs.com
 Submitting a sample via this chain of custody constitutes acknowledgment and acceptance of the Pace Terms and Conditions found at <https://info.pacelabs.com/hubfs/pas-standards-terms.pdf>

Section B
 Invoice Information:
 Company Name
 Address
 City
 State
 Zip
 Project Name
 Project #

Section C
 Required Project Information:
 Report To
 Copy To
 Purchase Order #
 Project Name
 Project #

Section D
 Regulatory Agency
 State / Location
 MIN

CHAIN-OF-CUSTODY / Analytical Request Document
 The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

ITEM #	MATRIX	CODE	SAMPLE TYPE (C=CRAB, C-COMP)	COLLECTED		SAMPLE TEMP AT COLLECTION	# OF CONTAINERS	Preservatives	Analyses Test	Requested Analysis Filtered (Y/N)	Received on	SAMPLE CONDITIONS				
				START DATE	END TIME							TEMP in C	Sealed	Custody	Intact	
1	Water	DW						HCl								
2	Water	WT						HCl								
3	Water	WV						HCl								
4	Water	WS						HCl								
5	Water	WSL						HCl								
6	Water	WSV						HCl								
7	Water	WV						HCl								
8	Water	WS						HCl								
9	Water	WSL						HCl								
10	Water	WSV						HCl								
11	Water	WV						HCl								
12	Water	WT						HCl								

ADDITIONAL COMMENTS


RELINQUISHED BY / AFFILIATION **DATE** **TIME**

ACCEPTED BY / AFFILIATION **DATE** **TIME**

SAMPLER NAME AND SIGNATURE
 PRINT Name of SAMPLER:
 SIGNATURE of SAMPLER: **DATE Signed:**

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	ENV-SOP-MIN4-0184 v02_Data and Records Archival
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Management Approval:

Adam Haugerud Approved on 12/29/2022 4:01:01 PM

Aileen Stacks Approved on 1/3/2023 9:04:21 AM

1.0 PURPOSE

This standard operating procedure (SOP) describes the procedure used for processes involved in the archiving of laboratory data and records.

2.0 SCOPE AND APPLICATION

The policies and procedures contained in this SOP are applicable to all personnel involved in the archiving of laboratory data and records (both paper and electronic).

In conjunction with the electronic media storage, all paper documentation is catalogued and stored for a period of time relating to the federal program guidelines the samples were analyzed under.

3.0 SUMMARY

The laboratory must have a system for the identification, collection, indexing, access, filing, storage, maintenance and disposal of all quality, technical records and all laboratory data.

Archives for orderly storage and expedient retrieval of all raw data, documentation, and records generated in the design and operation of the automated data collection system will be maintained by the laboratory. Conditions of storage shall minimize potential deterioration of documents or magnetic media in accordance with the requirements for the retention period and the nature of the documents or magnetic media. Only authorized personnel with documentation are permitted access to the archives.

4.0 DEFINITIONS

Refer to the Laboratory Quality Manual for a glossary of common laboratory terms and definitions.

5.0 HEALTH AND SAFETY

There are no specific health and safety considerations for the execution of the procedures in this SOP.


6.0 PROCEDURE

6.1 Equipment and Supplies

- Archive box (dimensions 15"L x 12"W x 10"H), Bertelsons Bankers Box, Iron Mountain 2000 or 2000A, or equivalent.
- Microsoft Excel.
- Iron Mountain RFID Labels, Iron Mountain blue RFID T-LAB label for boxes, or equivalent.
- Laboratory box ID labels, Brightly colored (1" x 2.625"), Avery 5972, or equivalent.

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- Master archive box ID spreadsheet, maintained by Quality Department (QA).
- Hardware and/or software capable of storing and retrieving data, provided by Pace IT.

6.2 Types of Archiving

6.2.1 General Records

All quality and technical records must include the identity of the personnel responsible for the sampling, performance of each test, and checking of results, as applicable. All records must be maintained by the laboratory per the retention time requirements listed in section 6.5. Records shall include, but are not limited to, the following:

- Reports from internal audits;
- Reports from management reviews;
- Records of corrective and preventive actions;
- Calibration records (support equipment, etc.);
- Personnel training records;
- Copies of each test report and data package issued;
- Current and past SOPs, forms, worksheets, and workbooks;
- Control graphs and control limits;
- Logbooks (maintenance, sequence, standard prep, etc.);
- Contracts;
- Invoices, Chains-of-custody, phone logs, emails, etc.


6.2.2 General Data

The laboratory must retain all original observations and derived data. All information necessary for the historical reconstruction of laboratory data must be maintained by the laboratory per the retention time requirements listed in section 6.5. This material includes, but is not limited to, the following:

- All LIMS data;
- All raw data, whether hardcopy or electronic, for calibrations, samples and quality control samples, including analyst's worksheets and data output records (chromatograms, strip charts, and other instrument response readout records);
- A written description or reference to the specific method used, which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;
- Laboratory sample ID;

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- Date of analysis;
- Time of analysis is required if the holding time is 72 hours or less, or when time critical steps are included in the analysis (e.g., extractions and incubations);
- Instrumentation identification and instrument operating conditions/parameters (or reference to such data);
- All manual calculations;
- Analyst's or operator's initials/signature or electronic identification;
- Sample preparation, including cleanup, separation protocols, incubation periods or subculture, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;
- Test results;
- Standard and reagent origin, receipt, preparation, and use;
- Calibration criteria, frequency, and acceptance criteria;
- Data and statistical calculations, review, confirmation, interpretation, assessment, and reporting conventions;
- Quality control protocols and assessment;
- Electronic security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries;
- Method performance criteria including expected quality control requirements;
- Proficiency test results;
- Records of demonstration of capability for each analyst;
- A record of names, initials, and signatures for all individuals who are responsible for signing or initialing any laboratory records.

6.3 Paper Hardcopy Data and Record Archival

6.3.1 Filing/Storage

All records (including paper) must be held secure and in confidence (NELAC/TNI requirement).


NOTE: The laboratory must archive paper records and data in a secure facility, which is easily accessible and climate-controlled.

6.3.2 Access

Access to archived information must be documented with an access log (NELAC/TNI requirement). This is to prevent unauthorized access to these paper records.

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Records must be available to the laboratory's accreditation body (NELAC/TNI requirement) or any other applicable auditing body.

6.3.3 Disposal

The laboratory or the outside company in charge of data storage can dispose of paper archives after the appropriate retention time has expired. Paper records should be shredded and the laboratory must retain certificates of disposal from any applicable outside data storage/disposal vendors.

6.3.4 Data Archival

Each Department collects hardcopy data for archiving in a standard sized Archive Box within the department. Examples of retained hardcopy files include raw data, logbooks, standard and reagent traceability information, reports, etc.

- 1) Each archive box filled must have an inventory form ENV-FRM-MIN4-0170 *Document Recall Spreadsheet* (current version or equivalent replacement), completed. The form is accessible via Qualtrax.
- 2) Boxes are retained on-site in the secured QA storage room until the data or records are at least 6-months old. Once the data or records are older than 6-months, the archive box will be shipped off-site for long-term storage at Iron Mountain.

NOTE: Some data or records may need to remain on-site longer than 6-months before it is shipped off-site. These include laboratory waste inspection reports and certifications. At minimum, the laboratory should maintain the three most recent years of records on-site. Anything older can be shipped off-site to Iron Mountain for long-term storage.

- Pace-Duluth and Virginia MN laboratories will maintain archived data on-site until ready to be shipped off-site for long-term storage, if applicable. Transfer of archive boxes will be coordinated with Pace-Minneapolis QA personnel.
- 3) QA will schedule a pick-up with Iron Mountain at least once every 6-months in order to move archive boxes off-site for long-term storage.


NOTE: When applicable, QA will notify Department supervisors/managers at least 2-weeks prior to the scheduled pick-up in case there are archive boxes being stored by the Departments that are ready to be shipped off-site.

- 4) Data will be stored at Iron Mountain as follows:
 - Minimum of 5-years for administrative records;
 - Minimum of 10-years for technical and quality records; and/or
 - Longer, depending on federal program or contractual guidelines. See section 6.6 for additional requirements.

See ENV-SWI-MIN4-0074 *Data Archiving and Recall SWI – Lab* (current version or equivalent replacement) for detailed information on the data archival process.

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6.3.5 Data Retrieval

To retrieve data from an archived box, laboratory form ENV-FRM-MIN4-0171 *Request for Archived Data* (current version or equivalent replacement), must be completed and submitted to QA.

See ENV-SWI-MIN4-0074 *Data Archiving and Recall SWI – Lab* (current version or equivalent replacement) for detailed information on the data retrieval process.

6.4 Electronic Data and Record Archival

6.4.1 Filing and Storage

All records (including electronic) must be held secure and in confidence (NELAC/TNI requirement).

The laboratory must archive electronic records and data in a secure facility and/or secure cabinet, which is easily accessible and climate-controlled.

For ESI laboratories:

- Laboratories are required to store electronic data in a limited-access area with redundant copies stored in fireproof vaults and stored off-site from the laboratory.
- Laboratories must exercise “best practices” in terms of frequent, redundant electronic backup procedures on proper long-term storage media to ensure that all electronic data representing the client’s sample analyses will be maintained for the designated storage period.
- See section 6.5 for additional archiving requirements.

6.4.2 Access

Access to archived information (including electronic) must be documented with an access log (TNI requirement). This is to prevent unauthorized access to or amendment of these electronic records.

Records (including electronic) must be available to the laboratory’s accreditation body (NELAC/TNI requirement) or any other applicable auditing body.

Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval (NELAC/TNI requirement).

6.4.3 Disposal and/or Deletion of Data


The laboratory or the outside company in charge of data storage can dispose of or delete electronic archives after the appropriate retention time has expired. The laboratory must retain certificates of disposal from any applicable outside data storage/disposal vendors.

6.4.3.1 Electronic System Backup – ChemStation/Target

Data is acquired throughout the laboratory on various ChemStation based instruments. This data is then uploaded to the Target server for processing and

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reporting. Processing the ChemStation data files results in the generation of Target data files which must then be backed up as part of the laboratory process and archived for up to 10 or more years, with the ability to quickly retrieve that data when required. The Target server itself runs as a VMWare virtual machine, running on an ESXi host.

Backup and archiving occurs at three levels to satisfy different requirements, from short term protection from hardware failure to long term offsite archiving for regulatory reasons. The main levels are described here:


- **Protection from Hardware Failure of the Main Server** – After acquisition on the instruments, the data is uploaded to the main Target server from where it is accessed by the Target application for processing and reporting. Storage on the target server consists of mirrored hard drives in the RAID-1 storage format. This provides protection against one of the disks failing. In addition, the data is backed up nightly to a disk backup system in our Corporate IT data center, which provides protection against a catastrophic server failure.
- **Live Backups (allows rapid retrieval of data for re-processing)** – During processing and reporting, Target generates multiple additional files which are stored along with the data files. A typical one-day analysis batch can easily result in almost two thousand files being generated on the server. With multiple instruments, this easily expands into multiple millions of files over a period of months. This amount of data causes performance issues on the server, both from a file management and a user point of view. For this reason, only a few months of data are stored on the live Target server, with the remaining being stored on a local Network Attached Server (NAS) backup server. Currently, three to four months of live data are stored on the Target server. On a twice daily basis, an automated process is run which backs up any changed files from the Target server to the local NAS backup server. After backing these up, the data older than three months old is purged from the live Target server. Data is never purged from the live backup server, so all Target data will always be easily retrievable.
- **Offsite Redundant Storage and Archival** – Electronic Data must also be stored offsite, to protect against catastrophic damage to the lab. In case of damage to the server room, or a total loss of the lab, a copy of the data is stored in an offsite location, remote from the primary lab. As data must be retained for a period of 10 or more years to satisfy regulatory requirements, these offsite archives also serve to satisfy that requirement.

6.4.3.2 Electronic System Backup – Avalon (LIMS)

The Persistent Organic Pollutants (POPs) department analytical data is acquired on various Waters and Sciex instruments. This data is then uploaded to the Avalon server for processing and reporting. The processed results of this data are stored in a relational database, also housed on the Avalon server. Several PDF files are also associated with the results, such as scanned chain of custody documents, and final reports. In addition, if a client requests EDDs with their results, these are

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also generated and stored on the Avalon server. All of this data must be backed up and archived for up to ten or more years and be easily retrievable. The Avalon server itself runs as a dedicated VMWare virtual machine, running on an ESXi host.

Backup and archiving occurs at three levels to satisfy different requirements, from short term protection from hardware failure to long term offsite archiving for regulatory reasons. The main levels are described here:

- **Protection From Disk Failure of the Main Server** – After acquisition on the instruments, the data is uploaded to the main Avalon server from where it is accessed by the Avalon application for processing and reporting. Storage on the Avalon server consists of mirrored hard drives in the RAID-6 storage format. This provides protection against two of the disks failing. Data is never purged from this server, resulting in instant live access to data from any time frame.
- **Protection From Hardware Failure of the Main Server** – This server virtual machine is backed up twice each day to a local NAS which provides protection against a catastrophic server failure. These backups are also never purged.
- **Offsite Redundant Storage and Archival** – Electronic Data must also be stored offsite, to protect against catastrophic damage to the lab. In case of damage to the server room, or a total loss of the lab, a copy of the data is stored in an offsite location, remote from the primary lab. As data must be retained for a period of 10 or more years to satisfy regulatory requirements, these offsite archives also serve to satisfy that requirement. As the other labs in our region also have local NAS systems for their local backup, we are able to back up the data from one lab to another in a different city, giving us offsite backup.

6.4.3.3 Processes

6.4.3.3.1 Hardware Failure Backup

The Target server is backed up nightly to a disk-based system in our Corporate IT Data Center which is offsite from the lab. This process is integral to corporate IT's backup process and is described in their backup documentation.


6.4.3.3.2 NAS Backup of Live Target Server

The Target server is backed up daily to an external NAS backup system using the Forever-incremental (also known as progressive incremental) scheme. The forever-incremental scheme is similar to the regular incremental one, but in this case a full backup is made only once, rather than periodically. After the initial full backup, the software copies only increments:

- **INITIAL** – The first backup is a full backup of the virtual machine

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containing the Target server. Under normal conditions, this backup is taken when the server is initially set up and is the only full backup taken.

- **INCREMENTAL** – These are the DAILY backups, run mid-day, and again in the evenings, seven days a week. Included in this backup are all files on the Target server, that have changed since the last backup process was run. These are added to the other files in the backup creating an auditable record of every file change. Due to the powerful data deduplication employed by the backup system, storage space is minimized, and backups may be run for many years.
- **FULL** – This is the complete backup of all data contained on the Target server. The full backups are automatically generated by the backup system by combining data from the incremental backups. Due to the way the forever incremental system works, it is possible to restore the Target server, or its data, to any point in time with the granularity of the backup process (i.e. twice per day).

This backup process is completed through the use of the Synology Active Backup for Business application on a Synology rack mounted NAS.

- Server Name: s10LabNAS01
- Login: administrator
- Password: *****


6.4.3.3.3 NAS Backup of Live Avalon Server

The Avalon server is backed up daily to an external NAS backup system using the Forever-incremental (also known as progressive incremental) scheme. The forever-incremental scheme is similar to the regular incremental one, but in this case a full backup is made only once, rather than periodically. After the initial full backup, the software copies only increments:

- **INITIAL** – The first backup is a full backup of the virtual machine containing the Avalon server. Under normal conditions, this backup is taken when the server is initially set up and is the only full backup taken.
- **INCREMENTAL** – These are the DAILY backups, run mid-day, and again in the evenings, seven days a week. Included in this backup are all files on the Avalon server, that have changed since the last backup process was run. These are added to the other files in the backup creating an auditable record of every file change. Due to the powerful data deduplication employed by the backup system,

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storage space is minimized, and backups may be run for many years.

- **FULL** – This is the complete backup of all data contained on the Target server. The full backups are automatically generated by the backup system by combining data from the initial backup and the incremental backups. Due to the way the forever incremental system works, it is possible to restore the Target server, or its data, to any point in time with the granularity of the backup process (i.e., twice per day). Data may be restored either at the individual file level, or as a complete server image. This has the advantage that if the server were to fail catastrophically, the whole system could be restored in a matter of minutes.

This backup process is completed through the use of the Synology Active Backup for Business application on a Synology rack mounted NAS.

- Server Name: s10LabNAS01
- Login: administrator
- Password: *****

6.4.3.3.4 Archival Data and Offsite Redundant Storage

Data is stored offsite at the Pace-Virginia MN laboratory using an automated script running in Pace-Minneapolis. On a weekly basis, starting on Saturdays, all Target batches that have been modified since the last archival run (should be one week earlier, unless there was an outage), are compressed into a 7Zip file, one per batch, and copied to a NAS in the Pace-Virginia laboratory. This results in an additional backup of the data, and an offsite copy in another city.

6.5 Data and Records Retention Time

Pace's retention time policy is to follow the most conservative of the retention time requirements listed below.

6.5.1 NELAC/TNI Retention Time Requirements

The laboratory must retain all records for a minimum of 5 years from generation of the last entry in the records.


6.5.2 State and/or Program Retention Time requirements

The laboratory must be aware of and comply with all state/program retention times that exceed the NELAC/TNI requirement of 5 years.

- **Manual for the Certification of Laboratories Analyzing Drinking Water (fifth edition, Chapter IV Chemistry, section 8.2)** – “Public Water Systems are required to maintain records of chemical analyses of compliance samples for 10 years (40 CFR

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§141.33) and lead and copper for 12 years (40 CFR §141.91). The laboratory should maintain easily accessible records for 5 years or until the next certification data audit is complete, whichever is longer. Changes in ownership, mergers, or closures of laboratories do not eliminate these requirements. The client water system should be notified before disposing of records so they may request copies if needed. This includes all raw data, calculations, and quality control data. These data files may be either hard copy, microfiche or electronic. Electronic data should always be backed up by protected tape or disk or hard copy. If the laboratory changes its computer hardware or software, it should make provisions for transferring old data to the new system so that it remains retrievable within the time frames specified above. Data which is expected to become part of a legal action may need to be maintained for a longer period of time. Check with your legal counsel”.

- **EPA Manual for the Certification of Laboratories Analyzing Drinking Water (fifth edition, Chapter V Microbiology, section 8.2)** – “Public Water Systems are required to maintain records of microbiological analyses of compliance samples for 5 years (40 CFR 141.33). The laboratory should maintain easily accessible records for 5 years or until the next certification data audit is complete, whichever is longer. A change in ownership, merger, or closure of a laboratory does not cancel this requirement. The client water system should be notified before disposing of records so they may request copies if needed. This includes all raw data, calculations, and quality control data. These data files may be either hard copy, microfiche or electronic. Electronic data should always be backed up by protected tape or disk or hard copy. If the laboratory changes its computer hardware or software, it should make provisions for transferring old data to the new system so that it remains retrievable within the time frames specified above. Data which is expected to become part of a legal action may need to be maintained for a longer period of time. Check with your legal counsel”.
- **EPA Manual for the Certification of Laboratories Analyzing Drinking Water (fifth edition, Chapter VI Radiochemistry, section 8.2)** – “Public Water Systems are required to maintain records of radionuclide analyses of compliance samples for 10 years (40 CFR 141.33). The laboratory should maintain easily accessible records for 10 years. The client water system should be notified before disposing of records so they may request copies if needed. This includes all raw data, calculations, and quality control data. These data files may be either hard copy, microfiche or electronic. Electronic data should always be backed up by protected tape or disk or hard copy. If the laboratory changes its computer hardware or software, it should make provisions for transferring old data to the new system so that it remains retrievable within the time frames specified above. Data which is expected to become part of a legal action may need to be maintained for a longer period of time”.
- **State of Florida Chapter 62-160 Quality Assurance (section 62-160.340.(1).c)** – “Records shall be retained for a minimum of 5 years after the date of generation or completion of the records unless otherwise specified in a Department contract, order, permit or Title 62 rules”.
- **State of Massachusetts DEP WSC-CAM document (dated 7/1/2010) states in section 1.5.2.e** – “In order to achieve “Presumptive Certainty” for analytical data,

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parties must comply with all the reporting requirements specified in WSC-CAM-VII A, including retention of reported and unreported analytical data and information for a period of 10 years”. In Appendix IV A-2, it states: “If requested by MassDEP, submission of the information listed below may be required to perform a data audit to verify compliance with the analytical methods and to evaluate accuracy and reliability of the reported results. These deliverables represent a “full data package” including all sample documentation from receipt through preparation, analysis, and data reporting. The laboratory must ensure that these deliverables are available, in the event a data audit is performed. The laboratory is required to retain these deliverables for a period of 10 years from the date generated”.

- **State of New York DOH Environmental Laboratory Approval Program Certification Manual (Item No. 176)** – “The following record retention time frames shall be implemented by laboratories. The records retained shall identify precisely the samples collected, accepted and examined; procedures used, and personnel involved; and document test conditions, observations and results of analyses. Training records are also to be retained according to the time frames listed below:
 - **Potable Water Chemistry (samples collected from public water supply systems):** 10 years per NYS Part 55-2.4(a)(3).
 - Records associated with lead and copper analyses shall be retained for no fewer 12 years by any system subject to Subpart I of the Code of Federal Regulations (Title 40, Part 141, Subpart I, §141.91 Recordkeeping requirements).
 - **Potable Water Chemistry (samples collected from private drinking water wells):** 5 years per NYS Part 55-2.4(a)(3).
 - **Potable Water and Non-Potable Water Microbiology:** 5 years per NYS Part 55-2.4(a)(3).
 - **Non-Potable Water Chemistry:** 5 years per NYS Part 55-2.4(a)(3).
 - **Solid and Hazardous Waste Chemistry and Asbestos:** 5 years per NYS Part 55-2.4(a)(3).
 - **Air and Emissions Chemistry and Asbestos:** 5 years per NYS Part 55-2.4(a)(3).
 - **Critical Agents:** 5 years per NYS Part 55-2.4(a)(3) and 55-2.13(d)(3) and (7).
 - Access records, chain of custody records, and records of analyses of confirmed positive samples are maintained for 10 years.
 - The training records of laboratory staff engaged in collecting and/or transporting critical agent samples shall be maintained a minimum of 3 years”.
 - **Medical Marijuana:** 5 years per NYS Part 55-2.4(a)(3).
- **State of Pennsylvania DEP Laboratory Accreditation manual [section 252.706 (d)]** – “Records required under this chapter shall be maintained for a minimum of 5

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years unless otherwise specified”.

- **State of Wisconsin DNR Laboratory Certification and Registration NR149 section 149.39.c** – “The laboratory shall retain all records and documents that are part of its quality system and that are required to demonstrate compliance with this chapter for a minimum of 3 years after the generation of the last entry in a record or document. The laboratory shall retain records and documents for a longer minimum period, if they are necessary to reconstruct analytical results generated during a 3-year period”.


6.5.3 Client Retention Time Requirements

The laboratory must be aware of and comply with all client or agency retention times that exceed the NELAC/TNI requirement of 5 years.

- **Amtrak Appendix I Statement of Work** – “Electronic and hardcopy data reports shall be kept at the Subcontractor facility for 5years after termination or expiration of this subcontract. Storage areas shall be secured. Documents shall be protected from damage due to fire, moisture and electromagnetic fields”.
- **Anadarko Services and Technical Specifications Manual, section 3.7 (most current version)** – “All data, instrument output (inclusive of electronic media), logbooks, reports, hardcopy and electronic copy of all data packages delivered, and applicable peripheral documentation, including but not limited to, financial documents and invoices generated by each laboratory must be stored in an organized, categorized, inventoried fashion for 7 years after completion of the Anadarko contract; at Anadarko’s request, any and all data must be submitted to Anadarko or its designated consultant/authorized representative at any point prior to the completion or at completion of the contract. Overwriting or disposal of any electronic media prior to this expiration period is strictly prohibited. Information sufficient to generate a Level 4 data package (if not initially requested) must be maintained...After the 7 year storage period, the laboratory must contact Anadarko to determine if data should be properly disposed of, maintained for an extended period, or shipped to Anadarko for storage. No data can be disposed of without contacting Anadarko for approval”.
- **Beazer Minimum Standards document, section 3.10 (most current revision)** – “All data, instrument output (inclusive of electronic media), logbooks, reports, hardcopy and electronic copy of all data packages delivered, and applicable peripheral documentation, including but not limited to, financial documents and invoices generated by each laboratory must be stored in an organized, categorized, inventoried fashion for 10 years after completion of a project. At Beazer/TRMI’s request, any and all data must be submitted to Beazer/TRMI and/or its designated consultant at any point prior to the completion or at completion of the contract. Overwriting or disposal of any electronic media prior to this expiration period is strictly prohibited. Information sufficient to generate a Level 4 data package (if not initially requested) must be maintained...After the 10 year storage period, the laboratory must contact Beazer/TRMI to determine if data can be properly disposed of, kept for an extended period, or shipped to Beazer/TRMI for storage. No data can be disposed of without contacting Beazer/TRMI for approval”.

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- **BNSF Railway Services and Technical Specifications Manual, section 3.7 (most current revision)** – “All data, instrument output (inclusive of electronic media), logbooks, reports, hardcopy and electronic copy of all data packages delivered, and applicable peripheral documentation, including but not limited to, financial documents and invoices generated by each laboratory must be stored in an organized, categorized, inventoried fashion for 5 years after completion of the BNSF contract; at BNSF’s request, any and all data must be submitted to BNSF or its designated consultant/authorized representative at any point prior to the completion or at completion of the contract. Overwriting or disposal of any electronic media prior to this expiration period is strictly prohibited. Information sufficient to generate a Level 4 data package (if not initially requested) must be maintained...After the 5 year storage period, the laboratory must contact BNSF to determine if data should be properly disposed of, maintained for an extended period, or shipped to BNSF for storage. No data can be disposed of without contacting BNSF for approval”.
- **BP LaMP Technical Requirements Document, section 4.3 (most current revision)** – “All data, instrument output (inclusive of electronic media), controlled logbooks, reports, hardcopy and electronic copy of all data packages delivered, and all applicable peripheral documentation, including but not limited to financial documents and invoices generated by each LaMP laboratory, must be stored in an organized, categorized, inventoried fashion for 7 years after the completion of a project, or, at BP’s request, any and all data must be submitted to BP and/or designated/authorized representative at any point prior to the completion or at completion of the contract”.
- **Canadian National Railway (CNRR) Services and Technical Specifications Manual, section 3.7 (most current revision)** – “All data, instrument output (inclusive of electronic media), logbooks, reports, hardcopy and electronic copy of all data packages delivered, and applicable peripheral documentation, including but not limited to, financial documents and invoices generated by each laboratory must be stored in an organized, categorized, inventoried fashion for 7 years after completion of the CN contract; at CN’s request, any and all data must be submitted to CN or its designated consultant/authorized representative at any point prior to the completion or at completion of the contract. Overwriting or disposal of any electronic media prior to this expiration period is strictly prohibited. Information sufficient to generate a Level 3 data package (if not initially requested) must be maintained...After the 7 year storage period, the laboratories must contact CN to determine if data should be properly disposed of, maintained for an extended period, or shipped to CN for storage. No data can be disposed of without contacting CN for approval”.
- **ConocoPhillips Technical Specifications Manual, section 3.7 (most current revision)** – “All data, instrument output (inclusive of electronic media), logbooks, reports, hardcopy and electronic copy of all data packages delivered, and applicable peripheral documentation, including but not limited to, financial documents and invoices generated by each laboratory must be stored in an organized, categorized, inventoried fashion for 10 years after completion of the ConocoPhillips contract; at ConocoPhillips’ request, any and all data must be submitted to ConocoPhillips and/or its designated/authorized representative at any point prior to the completion or at completion of the contract. Overwriting or disposal of any electronic media is strictly

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prohibited. Information sufficient to generate an “after the fact” Tier III data package (if not initially requested) must be maintained ...After the 10 year storage period, the laboratory must contact ConocoPhillips to determine if data should be properly disposed of, maintained for an extended period, or shipped to ConocoPhillips for storage. No data can be disposed of without contacting ConocoPhillips for approval”.

- **GE Minimum Standards document, section 3.8 (most current revision)** – “All data, instrument output (inclusive of electronic media), logbooks, reports, hardcopy and electronic copy of all data packages delivered, and applicable peripheral documentation, including but not limited to, financial documents and invoices generated by each laboratory must be stored in an organized, categorized, inventoried fashion for 5 years after completion of a project; at GE’s request, any and all data must be submitted to GE and/or its designated consultant/authorized representative at any point prior to the completion or at completion of the contract. Overwriting or disposal of any electronic media prior to this expiration period is strictly prohibited. Information sufficient to generate a Level 3 data package (if not initially requested) must be maintained...After the 5 year storage period, the laboratories must contact GE to determine if data should be properly disposed of, kept for an extended period, or shipped to GE for storage. No data can be disposed without contacting GE for approval”.
- **Norfolk Southern Services and Technical Specifications Manual, section 3.7 (most current revision)** – “All data, instrument output (inclusive of electronic media), logbooks, reports, hardcopy and electronic copy of all data packages delivered, and applicable peripheral documentation, including but not limited to, financial documents and invoices generated by each laboratory must be stored in an organized, categorized, inventoried fashion for 7 years after completion of the Norfolk Southern contract; at Norfolk Southern’s request, any and all data must be submitted to Norfolk Southern or its designated consultant/authorized representative at any point prior to the completion or at completion of the contract. Overwriting or disposal of any electronic media prior to this expiration period is strictly prohibited. Information sufficient to generate a Level 4 data package (if not initially requested) must be maintained...After the 7 year storage period, the laboratory must contact Norfolk Southern to determine if data should be properly disposed of, maintained for an extended period, or shipped to Norfolk Southern for storage. No data can be disposed of without contacting Norfolk Southern for approval”.
- **Sunoco Technical Specifications Manual, section 3.7 (most current revision)** – “All data, instrument output (inclusive of electronic media), logbooks, reports, hardcopy and electronic copy of all data packages delivered, and applicable peripheral documentation, including but not limited to, financial documents and invoices generated by each laboratory must be stored in an organized, categorized, inventoried fashion for 7 years after completion of the Sunoco contract; at Sunoco’s request, any and all data must be submitted to Sunoco and/or its designated consultant/authorized representative at any point prior to the completion or at completion of the contract. Overwriting or disposal of any electronic media prior to this expiration period is strictly prohibited. Information sufficient to generate a Level 3 data package (if not initially requested) must be maintained...After the 7 year storage period, the laboratory must

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contact Sunoco to determine if data should be properly disposed of, maintained for an extended period, or shipped to Sunoco for storage. No data can be disposed of without contacting Sunoco for approval”.

- **Tetra Tech Fox Rover Cleanup Project contract, page 94 (most current revision)** – “SUBCONTRACTOR shall maintain in a safe place at the Site one record copy of all Remediation correspondence, Drawings, Specifications, Change Orders, Change Directives, Submittals, Field Orders and written interpretations and clarifications, and all records documenting the costs (except for records pertaining to or evidencing income, profit, or other corporate or project financial results or returns and costs encompassed within unit rate, lump sum or other such stipulated payment items) and services performed and materials or parts supplied as part of the Remediation (“Remediation Records”). All Remediation Records shall be kept in accordance with generally accepted accounting principles and all applicable requirements of the IRS and Laws. The Remediation Records shall be kept in good order and annotated to show changes made during performance of the Remediation and will be available to TTECI for reference. Upon completion of the Remediation, all of the Remediation Records, including a reproducible set of record Drawings, will be delivered to TTECI at SUBCONTRACTOR’s cost. The Remediation Records will be kept and maintained for a period of 10 years from the date of the final payment as set forth herein. The Remediation Records may be kept by electronic storage with TTECI’s written consent, which shall not be unreasonably withheld, conditioned or delayed; should TTECI fail to provide such consent, TTECI will be responsible for the cost of keeping and maintaining such records in hard-copy format”.
- **United Technologies Corporation (UTC) Analytical Minimum Standards, section 3.5 (most current revision)** – “All data, instrument output (inclusive of electronic media), logbooks, reports, hardcopy and electronic copies of all data packages delivered, and applicable peripheral documentation, including but not limited to, financial documents and invoices generated by each preferred vendor, must be stored in an organized, categorized, inventoried fashion for 5 years after completion of a project; at UTC’s or designated consultant’s request, any and all data must be submitted to UTC or designated consultant at any point prior to the completion or at completion of the contract. Overwriting or disposal of any electronic media prior to this expiration period is strictly prohibited. Information sufficient to generate a Level 3 data package (if not initially requested) must be maintained. All electronic and hardcopy data must be stored in an easily accessible climate-controlled environment. Preferred vendors must exercise “best practices” in terms of frequent, redundant electronic backup procedures on proper long-term storage media to ensure that all electronic data representing UTC sample analyses will be maintained for the required storage period. Electronic data must be stored in a secure, limited-access area with redundant copies stored in fireproof vaults and/or stored off-site of the preferred vendor facility. After the 5-year storage period, the preferred vendors must contact UTC to determine if data can be properly disposed of, maintained for an extended period, or shipped to UTC for storage. No data can be disposed of without contacting UTC or designated consultant for approval.”
- **U.S. Steel Technical Specifications Manual, section 3.7 (most current revision)** –

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6.6 Liability for Data

If a Pace laboratory goes out of business or closes for any reason, the Pace Corporate Quality office shall ensure that all records (or copies of such records) are transferred to clients according to their instructions. Liability for such records will be described in applicable contracts.


7.0 RESPONSIBILITIES

- All employees of Pace® Analytical Services that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement (R&A) in their training file for the version(s) of the SOP that were in effect during the time the employee performed the activity.
- Local quality personnel are responsible for tracking the currency of the R&A on this SOP for employees at the locations they are assigned to and for notifying the General Manager (GM), however named, when R&A are overdue or outstanding. The GM and the employee’s direct supervisor are responsible for ensuring the employee completes the R&A assignments as required.
- The supervisors and managers of Pace® Analytical Services, however named, are responsible for training employees on the procedures in this SOP, implementing the SOP in the work area, and monitoring on-going adherence to the SOP the work area(s) they oversee.
- All employees of Pace® Analytical Services are responsible for following the procedures in this SOP. Unauthorized deviations or departures from this SOP are not allowed except with documented approval from the local Quality Manager and only when those deviations do not violate the Pace® Code of Ethics or Professional Conduct (COR-POL-0004) or associated policy and procedure(s). Hand-edits or manual change to the SOP are not permitted. If a change is desired or necessary, Pace® employees must follow the procedures for document revision specified in corporate SOPs ENV-SOP-CORQ-0015 *Document Management* and ENV-SOP-CORQ-0016 *SOP for Creation of SOP and SWI*.
- Local quality personnel are responsible for monitoring conformity to this SOP during routine internal audits of work areas that utilize this SOP and for communicating gaps and deviations found during monitoring to the work area supervisor, who is responsible for correction of the situation.

8.0 ATTACHMENTS

The distribution of printed or electronic copies of this document outside of the PAS electronic document management system (eDMS) is not controlled or tracked. PAS employees must ensure any copy used for daily work, if not accessed directly from PAS eDMS, is the current version of the document.

Process Standard Operating Procedure (SOP): Pace® Analytical Services

	ENV-SOP-MIN4-0184 v02_Data and Records Archival	
	Effective Date: 01/03/2023	COPYRIGHT© 2019, 2021, 2022 Pace®

Not applicable to this SOP.

9.0 REFERENCES

- ENV-SOP-CORQ-0015, *Document Management*, current version.
- ENV-SOP-CORQ-0016, *SOP for SOP and SWI*, current version.
- ENV-TMP-CORQ-0007, *Quality Manual Template*, current version.
- COR-POL-0004, *Code of Ethics and Professional Conduct*, current version.
- Laboratory Quality Manual, ENV-MAN-MIN4-0001, current version.
- TNI Standard, *Management and Technical Requirements for Laboratories Performing Environmental Analyses*, EL-V1-2016-Rev.2.1.
- Department of Defense (DoD) Quality Systems Manual- most current version.

10.0 REVISION HISTORY

Revisions Made from Prior Version

Section	Description of Change
ALL	Converted to new Corp process SOP template.
6.3.4(1)	Replaced MasterControl with Qualtrax.
6.3.4(2)	Deleted information for Pace-Billings; and Added information for Pace-Duluth and Virginia.

Document Succession: This version replaces the following documents:

Document Number & Version	Document Title	Effective Date:
ENV-SOP-MIN4-0184 v01	Data and Records Archival	09/09/2021

The distribution of printed or electronic copies of this document outside of the PAS electronic document management system (eDMS) is not controlled or tracked. PAS employees must ensure any copy used for daily work, if not accessed directly from PAS eDMS, is the current version of the document.

Appendix C _____

Pace Analytical Services Accreditations

MIN4_OR P_052523-50756-2 NELAP

Pace Cert MIN4_WI_083123-50767-2



Oregon Environmental Laboratory Accreditation Program



NELAP Recognized

Pace Analytical Services, LLC - Minneapolis MN

MN300001

1700 Elm Street SE
Minneapolis, MN 55414

IS GRANTED APPROVAL BY ORELAP UNDER THE 2016 TN1 STANDARDS, TO PERFORM ANALYSES ON ENVIRONMENTAL SAMPLES IN MATRICES AS LISTED BELOW :

Air	Drinking Water	Non-Potable Water	Solids and Chemical Waste	Tissue
		Chemistry	Chemistry	Chemistry

AND AS RECORDED IN THE LIST OF APPROVED ANALYTES, METHODS, ANALYTICAL TECHNIQUES, AND FIELDS OF TESTING ISSUED CONCURRENTLY WITH THIS CERTIFICATE AND REVISED AS NECESSARY.

ACCREDITED STATUS DEPENDS ON SUCCESSFUL ONGOING PARTICIPATION IN THE PROGRAM AND CONTINUED COMPLIANCE WITH THE STANDARDS.

CUSTOMERS ARE URGED TO VERIFY THE LABORATORY'S CURRENT ACCREDITATION STATUS IN OREGON.

Travis Bartholomew
Oregon State Public Health Laboratory
ORELAP Program Manager
7202 NE Evergreen Parkway, Suite 100
Hillsboro, OR 97124

EFFECTIVE DATE : 5/26/2022
EXPIRATION DATE : 5/25/2023
Certificate No : MN300001 - 015





OREGON

Environmental Laboratory Accreditation Program

ORELAP Fields of Accreditation



Pace Analytical Services, LLC - Minneapolis MN

1700 Elm Street SE
Minneapolis, MN 55414

ORELAP ID: MN300001

EPA CODE: MN00064

Certificate: MN300001 - 015

Issue Date: 5/26/2022 Expiration Date: 5/25/2023

As of 5/26/2022 this list supersedes all previous lists for this certificate number.

Matrix	Reference	Analyte Code	Analyte	Method Code	Description
Biological Tissue					
	PACE ENV-SOP-MIN4-0178 03			60046017	Pace Analytical - Determination of Selected 36 Per- and Polyfluoroalkane Substances (PFAS) by LC/MS/MS (Isotope Dilution)
		9490	11-chloreicosafuoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)		
		6948	1H, 1H, 2H, 2H-Perfluorodecanesulfonic acid (8:2 FTS)		
		9616	1H, 1H, 2H, 2H-perfluorododecane sulfonic acid (10:2 FTS)		
		6946	1H, 1H, 2H, 2H-Perfluorohexanesulfonic acid (4:2 FTS)		
		6947	1H, 1H, 2H, 2H-Perfluorooctanesulfonic acid (6:2 FTS)		
		6951	4,8-Dioxa-3H-perfluorononanoic acid (DONA)		
		6952	9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS)		
		9395	N-Ethylperfluorooctane sulfonamide (EtFOSAm)		
		9431	N-Ethylperfluorooctane sulfonamido ethanol (EtFOSE)		
		4846	N-Ethylperfluorooctanesulfonamidoacetic acid (NEtFOSAA)		
		4847	N-Methylperfluorooctane sulfonamido acetic acid (NMeFOSAA)		
		6949	N-Methylperfluorooctane sulfonamido ethanol (MeFOSE)		
		9433	N-Methylperfluorooctanesulfonamide (MeFOSA)		
		6918	Perfluorobutane sulfonic acid (PFBS)		
		6915	Perfluorobutanoic acid (PFBA)		
		6920	Perfluorodecane sulfonic acid (PFDS)		



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Environmental Laboratory Accreditation Program

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Pace Analytical Services, LLC - Minneapolis MN

1700 Elm Street SE
Minneapolis, MN 55414

ORELAP ID: MN300001

EPA CODE: MN00064

Certificate: MN300001 - 015

Issue Date: 5/26/2022 Expiration Date: 5/25/2023

As of 5/26/2022 this list supersedes all previous lists for this certificate number.

Matrix	Reference	Analyte Code	Analyte	Method Code	Description
		6905	Perfluorodecanoic acid (PFDA)		
		6923	Perfluorododecane sulfonic acid (PFDoS)		
		6903	Perfluorododecanoic acid (PFDoA)		
		9470	Perfluoroheptane sulfonic acid (PFHpS)		
		6908	Perfluoroheptanoic acid (PFHpA)		
		6901	Perfluorohexadecanoic acid (PFHxDA)		
		6927	Perfluorohexane sulfonic acid (PFHxS)		
		6913	Perfluorohexanoic acid (PFHxA)		
		6929	Perfluorononane sulfonic acid (PFNS)		
		6906	Perfluorononanoic acid (PFNA)		
		6916	Perfluorooctadecanoic acid (PFODA)		
		6917	Perfluorooctane sulfonamide (PFOSAm)		
		6931	Perfluorooctane sulfonic acid (PFOS)		
		6912	Perfluorooctanoic acid (PFOA)		
		6934	Perfluoropentane sulfonic acid (PFPeS)		
		6914	Perfluoropentanoic acid (PFPeA)		
		6902	Perfluorotetradecanoic acid (PFTDA)		
		9563	Perfluorotridecanoic acid (PFTrDA)		
		6904	Perfluoroundecanoic acid (PFUnA)		

Non-Potable Water

NWTPH-Dx	90018409	Oregon DEQ TPH Diesel Range
	9369	Diesel range organics (DRO)
	9499	Motor Oil
	9506	Residual Range Organics (RRO)
PACE ENV-SOP-MIN4-0178 03	60046017	Pace Analytical - Determination of Selected 36 Per- and Polyfluoroalkane Substances (PFAS) by LC/MS/MS (Isotope Dilution)
	9490	11-chloreicosafuoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)



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Environmental Laboratory Accreditation Program

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Pace Analytical Services, LLC - Minneapolis MN

1700 Elm Street SE
Minneapolis, MN 55414

ORELAP ID: MN300001

EPA CODE: MN00064

Certificate: MN300001 - 015

Issue Date: 5/26/2022 Expiration Date: 5/25/2023

As of 5/26/2022 this list supersedes all previous lists for this certificate number.

Matrix	Reference	Analyte Code	Analyte	Method Code	Description
	6948	1H, 1H, 2H, 2H-	Perfluorodecanesulfonic acid (8:2 FTS)		
	9616	1H, 1H, 2H, 2H-	perfluorododecane sulfonic acid (10:2 FTS)		
	6946	1H, 1H, 2H, 2H-	Perfluorohexanesulfonic acid (4:2 FTS)		
	6947	1H, 1H, 2H, 2H-	Perfluorooctanesulfonic acid (6:2 FTS)		
	6951	4,8-Dioxa-3H-	perfluorononanoic acid (DONA)		
	6952	9-chlorohexadecafluoro-3-oxanone-1-	sulfonic acid (9Cl-PF3ONS)		
	9460	Hexafluoropropylene oxide dimer acid	(HFPO-DA)		
	9395	N-Ethylperfluorooctane sulfonamide	(EtFOSAm)		
	9431	N-Ethylperfluorooctane sulfonamido ethanol	(EtFOSE)		
	4846	N-Ethylperfluorooctanesulfonamidoacetic acid	(NEtFOSAA)		
	4847	N-Methylperfluorooctane sulfonamido acetic acid	(NMeFOSAA)		
	6949	N-Methylperfluorooctane sulfonamido ethanol	(MeFOSE)		
	9433	N-Methylperfluorooctanesulfonamide	(MeFOSA)		
	6918	Perfluorobutane sulfonic acid	(PFBS)		
	6915	Perfluorobutanoic acid	(PFBA)		
	6920	Perfluorodecane sulfonic acid	(PFDS)		
	6905	Perfluorodecanoic acid	(PFDA)		
	6923	Perfluorododecane sulfonic acid	(PFDoS)		
	6903	Perfluorododecanoic acid	(PFDoA)		
	9470	Perfluoroheptane sulfonic acid	(PFHpS)		



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Environmental Laboratory Accreditation Program

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Pace Analytical Services, LLC - Minneapolis MN

1700 Elm Street SE
Minneapolis, MN 55414

ORELAP ID: MN300001

EPA CODE: MN00064

Certificate: MN300001 - 015

Issue Date: 5/26/2022 Expiration Date: 5/25/2023

As of 5/26/2022 this list supersedes all previous lists for this certificate number.

Matrix	Reference	Analyte Code	Analyte	Method Code	Description
		6908	Perfluoroheptanoic acid (PFHpA)		
		6901	Perfluorohexadecanoic acid (PFHxDA)		
		6927	Perfluorohexane sulfonic acid (PFHxS)		
		6913	Perfluorohexanoic acid (PFHxA)		
		6929	Perfluorononane sulfonic acid (PFNS)		
		6906	Perfluorononanoic acid (PFNA)		
		6916	Perfluorooctadecanoic acid (PFODA)		
		6917	Perfluorooctane sulfonamide (PFOSAm)		
		6931	Perfluorooctane sulfonic acid (PFOS)		
		6912	Perfluorooctanoic acid (PFOA)		
		6934	Perfluoropentane sulfonic acid (PFPeS)		
		6914	Perfluoropentanoic acid (PFPeA)		
		6902	Perfluorotetradecanoic acid (PFTDA)		
		9563	Perfluorotridecanoic acid (PFTrDA)		
		6904	Perfluoroundecanoic acid (PFUnA)		

Solids

NWTPH-Dx		90018409	Oregon DEQ TPH Diesel Range		
		9369	Diesel range organics (DRO)		
		9499	Motor Oil		
NWTPH-Gx		90018603	Oregon DEQ TPH Gasoline Range Organics by GC/FID-PID Purge & Trap		
		9408	Gasoline range organics (GRO)		
PACE ENV-SOP-MIN4-0178 03		60046017	Pace Analytical - Determination of Selected 36 Per- and Polyfluoroalkane Substances (PFAS) by LC/MS/MS (Isotope Dilution)		
		9490	11-chloreicosafuoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)		
		6948	1H, 1H, 2H, 2H-Perfluorodecanesulfonic acid (8:2 FTS)		
		9616	1H, 1H, 2H, 2H-perfluorododecane		



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Environmental Laboratory Accreditation Program

ORELAP Fields of Accreditation



Pace Analytical Services, LLC - Minneapolis MN

1700 Elm Street SE
Minneapolis, MN 55414

ORELAP ID: MN300001

EPA CODE: MN00064

Certificate: MN300001 - 015

Issue Date: 5/26/2022 Expiration Date: 5/25/2023

As of 5/26/2022 this list supersedes all previous lists for this certificate number.

Matrix	Reference	Analyte Code	Analyte	Method Code	Description
			sulfonic acid (10:2 FTS)		
	6946	1H, 1H, 2H, 2H-	Perfluorohexanesulfonic acid (4:2 FTS)		
	6947	1H, 1H, 2H, 2H-	Perfluorooctanesulfonic acid (6:2 FTS)		
	6951		4,8-Dioxa-3H-perfluorononanoic acid (DONA)		
	6952		9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS)		
	9460		Hexafluoropropylene oxide dimer acid (HFPO-DA)		
	9395		N-Ethylperfluorooctane sulfonamide (EtFOSAm)		
	9431		N-Ethylperfluorooctane sulfonamido ethanol (EtFOSE)		
	4846		N-Ethylperfluorooctanesulfonamidoacetic acid (NEtFOSAA)		
	4847		N-Methylperfluorooctane sulfonamido acetic acid (NMeFOSAA)		
	6949		N-Methylperfluorooctane sulfonamido ethanol (MeFOSE)		
	9433		N-Methylperfluorooctanesulfonamide (MeFOSA)		
	6918		Perfluorobutane sulfonic acid (PFBS)		
	6915		Perfluorobutanoic acid (PFBA)		
	6920		Perfluorodecane sulfonic acid (PFDS)		
	6905		Perfluorodecanoic acid (PFDA)		
	6923		Perfluorododecane sulfonic acid (PFDoS)		
	6903		Perfluorododecanoic acid (PFDoA)		
	9470		Perfluoroheptane sulfonic acid (PFHpS)		
	6908		Perfluoroheptanoic acid (PFHpA)		
	6901		Perfluorohexadecanoic acid (PFHxDA)		



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Environmental Laboratory Accreditation Program

ORELAP Fields of Accreditation



Pace Analytical Services, LLC - Minneapolis MN

1700 Elm Street SE
Minneapolis, MN 55414

ORELAP ID: MN300001

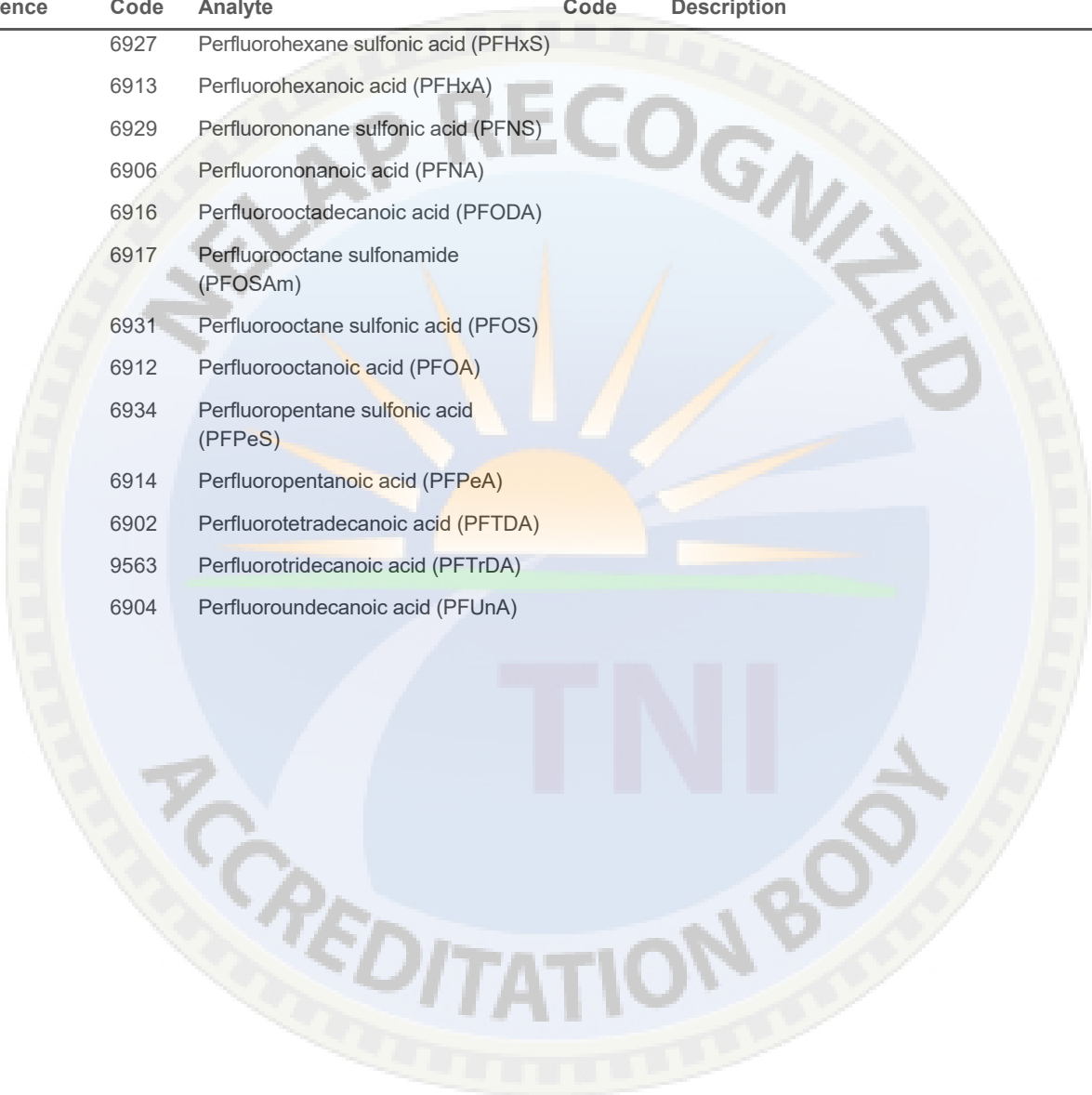
EPA CODE: MN00064

Certificate: MN300001 - 015

Issue Date: 5/26/2022 Expiration Date: 5/25/2023

As of 5/26/2022 this list supersedes all previous lists for this certificate number.

Matrix Reference	Analyte Code	Analyte	Method Code	Description
	6927	Perfluorohexane sulfonic acid (PFHxS)		
	6913	Perfluorohexanoic acid (PFHxA)		
	6929	Perfluorononane sulfonic acid (PFNS)		
	6906	Perfluorononanoic acid (PFNA)		
	6916	Perfluorooctadecanoic acid (PFODA)		
	6917	Perfluorooctane sulfonamide (PFOSAm)		
	6931	Perfluorooctane sulfonic acid (PFOS)		
	6912	Perfluorooctanoic acid (PFOA)		
	6934	Perfluoropentane sulfonic acid (PFPeS)		
	6914	Perfluoropentanoic acid (PFPeA)		
	6902	Perfluorotetradecanoic acid (PFTDA)		
	9563	Perfluorotridecanoic acid (PFTrDA)		
	6904	Perfluoroundecanoic acid (PFUnA)		





Accredited Laboratory

A2LA has accredited

PACE ANALYTICAL SERVICES, LLC

Minneapolis, MN

for technical competence in the field of

Environmental Testing

In recognition of the successful completion of the A2LA evaluation process that includes an assessment of the laboratory's compliance with ISO/IEC 17025:2017, the 2016 and 2009 TNI Environmental Testing Laboratory Standard, and the requirements of the Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in version 5.3 of the DoD Quality System Manual for Environmental Laboratories (QSM), accreditation is granted to this laboratory to perform recognized EPA methods as defined on the associated A2LA Environmental Scope of Accreditation. This accreditation demonstrates technical competence for this defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated April 2017).



Presented this 25th day of October 2021.

A blue ink signature of a man, written over a horizontal line.

Vice President, Accreditation Services
For the Accreditation Council
Certificate Number 2926.01
Valid to October 31, 2023

For the tests to which this accreditation applies, please refer to the laboratory's Environmental Scope of Accreditation.



SCOPE OF ACCREDITATION TO ISO/IEC 17025:2017

PACE ANALYTICAL SERVICES, LLC.
 1700 Elm Street SE, Suite 200
 Minneapolis, MN 55414
 Liz Stacks Phone: 612-607-6352

ENVIRONMENTAL

Valid To: October 31, 2023

Certificate Number: 2926.01

In recognition of the successful completion of the A2LA evaluation process, accreditation is granted to this laboratory to perform the following tests on dietary supplements, food products, and animal feed stocks:

Chemical Tests – Non-environmental testing

<u>Test</u>	<u>Test Method(s)</u>
PCB Congeners	EPA 1668A
Dioxins and Furans	EPA 1613B EPA 8290A

Environmental Tests

In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with ISO IEC 17025:2017, the 2016 and 2009 TNI Environmental Testing Laboratory Standard, and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in version 5.3 of the DoD Quality Systems Manual for Environmental Laboratories) accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

Testing Technologies: Gas Chromatography/Mass Spectrometry, High Resolution Gas Chromatography/Mass Spectrometry

<u>Parameter/Analyte</u>	<u>Potable Water</u>	<u>Nonpotable Water</u>	<u>Solid Hazardous Waste</u>	<u>Tissue</u>
<u>Extractable Organics</u>				
2,3,7,8-TCDD	-----	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A
2,3,7,8-TCDF	-----	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A
1,2,3,7,8-PeCDF	-----	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A
2,3,4,7,8-PeCDF	-----	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A
1,2,3,7,8-PeCDD	-----	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A
1,2,3,4,7,8-HxCDF	-----	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A

<u>Parameter/Analyte</u>	<u>Potable Water</u>	<u>Nonpotable Water</u>	<u>Solid Hazardous Waste</u>	<u>Tissue</u>
1,2,3,6,7,8-HxCDF	-----	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A
2,3,4,6,7,8-HxCDF	-----	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A
1,2,3,7,8,9-HxCDF	-----	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A
1,2,3,4,7,8-HxCDD	-----	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A
1,2,3,6,7,8-HxCDD	-----	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A
1,2,3,7,8,9-HxCDD	-----	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A
1,2,3,4,6,7,8-HpCDF	-----	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A
1,2,3,4,7,8,9-HpCDF	-----	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A
1,2,3,4,6,7,8-HpCDD	-----	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A
OCDF	-----	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A
OCDD	-----	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A
Total HpCDD	-----	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A
Total HpCDF	-----	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A
Total HxCDD	-----	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A
Total HxCDF	-----	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A
Total PeCDD	-----	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A
Total PeCDF	-----	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A
Total TCDD	-----	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A
Total TCDF	-----	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A	EPA 1613B EPA 8290/8290A

<u>Parameter/Analyte</u>	<u>PCB</u>	<u>Nonpotable Water</u>	<u>Solid Hazardous Waste</u>	<u>Tissue</u>
Extractable Organics				
PCB Congeners				
2-Chlorobiphenyl	PCB-1	EPA 1668A	EPA 1668A	EPA 1668A
3-Chlorobiphenyl	PCB-2	EPA 1668A	EPA 1668A	EPA 1668A
4-Chlorobiphenyl	PCB-3	EPA 1668A	EPA 1668A	EPA 1668A
2,2'-Dichlorobiphenyl	PCB-4	EPA 1668A	EPA 1668A	EPA 1668A
2,6-Dichlorobiphenyl	PCB-10	EPA 1668A	EPA 1668A	EPA 1668A
2,5-Dichlorobiphenyl	PCB-9	EPA 1668A	EPA 1668A	EPA 1668A

Parameter/Analyte	PCB	Nonpotable Water	Solid Hazardous Waste	Tissue
2,4-Dichlorobiphenyl	PCB-7	EPA 1668A	EPA 1668A	EPA 1668A
2,3'-Dichlorobiphenyl	PCB-6	EPA 1668A	EPA 1668A	EPA 1668A
2,3-Dichlorobiphenyl	PCB-5	EPA 1668A	EPA 1668A	EPA 1668A
2,4'-Dichlorobiphenyl	PCB-8	EPA 1668A	EPA 1668A	EPA 1668A
3,5-Dichlorobiphenyl	PCB-14	EPA 1668A	EPA 1668A	EPA 1668A
3,3'-Dichlorobiphenyl	PCB-11	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(13/12)	PCB-(13/12)	EPA 1668A	EPA 1668A	EPA 1668A
4,4'-Dichlorobiphenyl	PCB-15	EPA 1668A	EPA 1668A	EPA 1668A
2,2',6-Trichlorobiphenyl	PCB-19	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(30/18)	PCB-(30/18)	EPA 1668A	EPA 1668A	EPA 1668A
2,2',4-Trichlorobiphenyl	PCB-17	EPA 1668A	EPA 1668A	EPA 1668A
2,3',6-Trichlorobiphenyl	PCB-27	EPA 1668A	EPA 1668A	EPA 1668A
2,3,6-Trichlorobiphenyl	PCB-24	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3-Trichlorobiphenyl	PCB-16	EPA 1668A	EPA 1668A	EPA 1668A
2,4',6-Trichlorobiphenyl	PCB-32	EPA 1668A	EPA 1668A	EPA 1668A
2',3,5-Trichlorobiphenyl	PCB-34	EPA 1668A	EPA 1668A	EPA 1668A
2,3,5-Trichlorobiphenyl	PCB-23	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(26/29)	PCB-(26/29)	EPA 1668A	EPA 1668A	EPA 1668A
2,3',4-Trichlorobiphenyl	PCB-25	EPA 1668A	EPA 1668A	EPA 1668A
2,4',5-Trichlorobiphenyl	PCB-31	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(28/20)	PCB-(28/20)	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(21/33)	PCB-(21/33)	EPA 1668A	EPA 1668A	EPA 1668A
2,3,4'-Trichlorobiphenyl	PCB-22	EPA 1668A	EPA 1668A	EPA 1668A
3,3',5-Trichlorobiphenyl	PCB-36	EPA 1668A	EPA 1668A	EPA 1668A
3,4',5-Trichlorobiphenyl	PCB-39	EPA 1668A	EPA 1668A	EPA 1668A
3,4,5-Trichlorobiphenyl	PCB-38	EPA 1668A	EPA 1668A	EPA 1668A
3,3',4-Trichlorobiphenyl	PCB-35	EPA 1668A	EPA 1668A	EPA 1668A
3,4,4'-Trichlorobiphenyl	PCB-37	EPA 1668A	EPA 1668A	EPA 1668A
2,2',6,6'-Tetrachlorobiphenyl	PCB-54	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(50/53)	PCB-(50/53)	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(45/51)	PCB-(45/51)	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,6'-Tetrachlorobiphenyl	PCB-46	EPA 1668A	EPA 1668A	EPA 1668A
2,2',5,5'-Tetrachlorobiphenyl	PCB-52	EPA 1668A	EPA 1668A	EPA 1668A
2,3',5',6-Tetrachlorobiphenyl	PCB-(73/43)	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(69/49)	PCB-(69/49)	EPA 1668A	EPA 1668A	EPA 1668A
2,2',4,5-Tetrachlorobiphenyl	PCB-48	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(44/47/65)	PCB-(44/47/65)	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(59/62/75)	PCB-(59/62/75)	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,4'-Tetrachlorobiphenyl	PCB-42	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(41/40/71)	PCB-(41/40/71)	EPA 1668A	EPA 1668A	EPA 1668A
2,3,4',6-Tetrachlorobiphenyl	PCB-64	EPA 1668A	EPA 1668A	EPA 1668A
2,3',5,5'-Tetrachlorobiphenyl	PCB-72	EPA 1668A	EPA 1668A	EPA 1668A
2,3',4,5'-Tetrachlorobiphenyl	PCB-68	EPA 1668A	EPA 1668A	EPA 1668A
2,3,3',5-Tetrachlorobiphenyl	PCB-57	EPA 1668A	EPA 1668A	EPA 1668A
2,3,3',5'-Tetrachlorobiphenyl	PCB-58	EPA 1668A	EPA 1668A	EPA 1668A

Parameter/Analyte	PCB	Nonpotable Water	Solid Hazardous Waste	Tissue
2,3',4,5-Tetrachlorobiphenyl	PCB-67	EPA 1668A	EPA 1668A	EPA 1668A
2,3,4',5-Tetrachlorobiphenyl	PCB-63	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(61/70/74/76)	PCB-(61/70/74/76)	EPA 1668A	EPA 1668A	EPA 1668A
2,3',4,4'-Tetrachlorobiphenyl	PCB-66	EPA 1668A	EPA 1668A	EPA 1668A
2,3,3',4-Tetrachlorobiphenyl	PCB-55	EPA 1668A	EPA 1668A	EPA 1668A
2,3,3',4'-Tetrachlorobiphenyl	PCB-56	EPA 1668A	EPA 1668A	EPA 1668A
2,3,4,4'-Tetrachlorobiphenyl	PCB-60	EPA 1668A	EPA 1668A	EPA 1668A
3,3',5,5'-Tetrachlorobiphenyl	PCB-80	EPA 1668A	EPA 1668A	EPA 1668A
3,3',4,5'-Tetrachlorobiphenyl	PCB-79	EPA 1668A	EPA 1668A	EPA 1668A
3,3',4,5-Tetrachlorobiphenyl	PCB-78	EPA 1668A	EPA 1668A	EPA 1668A
3,4,4',5-Tetrachlorobiphenyl	PCB-81	EPA 1668A	EPA 1668A	EPA 1668A
3,3',4,4'-Tetrachlorobiphenyl	PCB-77	EPA 1668A	EPA 1668A	EPA 1668A
2,2',4,6,6'-Pentachlorobiphenyl	PCB-104	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,6,6'-Pentachlorobiphenyl	PCB-96	EPA 1668A	EPA 1668A	EPA 1668A
2,2',4,5',6-Pentachlorobiphenyl	PCB-103	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,5,6'-Pentachlorobiphenyl	PCB-94	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,5',6-Pentachlorobiphenyl	PCB-95	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(100/93/102/98)	PCB-(100/93/102/98)	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(88/91)	PCB-(88/91)	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,3',6-Pentachlorobiphenyl	PCB-84	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,4,6'-Pentachlorobiphenyl	PCB-89	EPA 1668A	EPA 1668A	EPA 1668A
2,3',4,5',6-Pentachlorobiphenyl	PCB-121	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,5,5'-Pentachlorobiphenyl	PCB-92	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(113/90/101)	PCB-(113/90/101)	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,3',5-Pentachlorobiphenyl	PCB-83	EPA 1668A	EPA 1668A	EPA 1668A
2,2',4,4',5-Pentachlorobiphenyl	PCB-99	EPA 1668A	EPA 1668A	EPA 1668A
2,3,3',5,6-Pentachlorobiphenyl	PCB-112	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(108/119/86/97/125/87)	PCB-(108/119/86/97/125/87)	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(117/116/85)	PCB-(117/116/85)	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(110/115)	PCB-(110/115)	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,3',4-Pentachlorobiphenyl	PCB-82	EPA 1668A	EPA 1668A	EPA 1668A
2,3,3',5,5'-Pentachlorobiphenyl	PCB-111	EPA 1668A	EPA 1668A	EPA 1668A
2,3',4,5,5'-Pentachlorobiphenyl	PCB-120	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(107/124)	PCB-(107/124)	EPA 1668A	EPA 1668A	EPA 1668A
2,3,3',4,6-Pentachlorobiphenyl	PCB-109	EPA 1668A	EPA 1668A	EPA 1668A
2,3',4,4',5'-Pentachlorobiphenyl	PCB-123	EPA 1668A	EPA 1668A	EPA 1668A
2,3,3',4,5-Pentachlorobiphenyl	PCB-106	EPA 1668A	EPA 1668A	EPA 1668A
2,3',4,4',5-Pentachlorobiphenyl	PCB-118	EPA 1668A	EPA 1668A	EPA 1668A
2,3,3',4',5'-Pentachlorobiphenyl	PCB-122	EPA 1668A	EPA 1668A	EPA 1668A
2,3,4,4',5-Pentachlorobiphenyl	PCB-114	EPA 1668A	EPA 1668A	EPA 1668A

Parameter/Analyte	PCB	Nonpotable Water	Solid Hazardous Waste	Tissue
2,3,3',4,4'-Pentachlorobiphenyl	PCB-105	EPA 1668A	EPA 1668A	EPA 1668A
3,3',4,5,5'-Pentachlorobiphenyl	PCB-127	EPA 1668A	EPA 1668A	EPA 1668A
3,3',4,4',5-Pentachlorobiphenyl	PCB-126	EPA 1668A	EPA 1668A	EPA 1668A
2,2',4,4',6,6'-Hexachlorobiphenyl	PCB-155	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,5,6,6'-Hexachlorobiphenyl	PCB-152	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,4',6,6'-Hexachlorobiphenyl	PCB-150	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,3',6,6'-Hexachlorobiphenyl	PCB-136	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,4,6,6'-Hexachlorobiphenyl	PCB-145	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,4',5,6'-Hexachlorobiphenyl	PCB-148	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(151/135)	PCB-(151/135)	EPA 1668A	EPA 1668A	EPA 1668A
2,2',4,4',5,6'-Hexachlorobiphenyl	PCB-154	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,4,5',6-Hexachlorobiphenyl	PCB-144	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(147/149)	PCB-(147/149)	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(134/143)	PCB-(134/143)	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(139/140)	PCB-(139/140)	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,3',4,6-Hexachlorobiphenyl	PCB-131	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,4,5,6-Hexachlorobiphenyl	PCB-142	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,3',4,6'-Hexachlorobiphenyl	PCB-132	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,3',5,5'-Hexachlorobiphenyl	PCB-133	EPA 1668A	EPA 1668A	EPA 1668A
2,3,3',5,5',6-Hexachlorobiphenyl	PCB-165	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,4',5,5'-Hexachlorobiphenyl	PCB-146	EPA 1668A	EPA 1668A	EPA 1668A
2,3,3',4,5',6-Hexachlorobiphenyl	PCB-161	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(153/168)	PCB-(153/168)	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,4,5,5'-Hexachlorobiphenyl	PCB-141	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,3',4,5'-Hexachlorobiphenyl	PCB-130	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,4,4',5-Hexachlorobiphenyl	PCB-137	EPA 1668A	EPA 1668A	EPA 1668A
2,3,3',4',5',6-Hexachlorobiphenyl	PCB-164	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(138/163/129)	PCB-(138/163/129)	EPA 1668A	EPA 1668A	EPA 1668A
2,3,3',4,5,6-Hexachlorobiphenyl	PCB-160	EPA 1668A	EPA 1668A	EPA 1668A
2,3,3',4,4',6-Hexachlorobiphenyl	PCB-158	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(128/166)	PCB-(128/166)	EPA 1668A	EPA 1668A	EPA 1668A
2,3,3',4,5,5'-Hexachlorobiphenyl	PCB-159	EPA 1668A	EPA 1668A	EPA 1668A
2,3,3',4',5,5'-Hexachlorobiphenyl	PCB-162	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(156/157)	PCB-(156/157)	EPA 1668A	EPA 1668A	EPA 1668A
3,3',4,4',5,5'-Hexachlorobiphenyl	PCB-169	EPA 1668A	EPA 1668A	EPA 1668A
2,3',4,4',5,5'-Hexachlorobiphenyl	PCB-167	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,4',5,6,6'-Heptachlorobiphenyl	PCB-188	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,3',5,6,6'-Heptachlorobiphenyl	PCB-179	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,4,4',6,6'-Heptachlorobiphenyl	PCB-184	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,3',4,6,6'-Heptachlorobiphenyl	PCB-176	EPA 1668A	EPA 1668A	EPA 1668A

Parameter/Analyte	PCB	Nonpotable Water	Solid Hazardous Waste	Tissue
2,2',3,4,4',5,6'-Heptachlorobiphenyl	PCB-186	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,4,5,6,6'-Heptachlorobiphenyl	PCB-178	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,3',5,5',6-Heptachlorobiphenyl	PCB-175	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,3',4,5',6-Heptachlorobiphenyl	PCB-187	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,4',5,5',6-Heptachlorobiphenyl	PCB-182	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(183/185)	PCB-(183/185)	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,3',4,5,6'-Heptachlorobiphenyl	PCB-174	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,3',4,5',6'-Heptachlorobiphenyl	PCB-177	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,4,4',5,6-Heptachlorobiphenyl	PCB-181	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(171/173)	PCB-(171/173)	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,3',4,5,5'-Heptachlorobiphenyl	PCB-172	EPA 1668A	EPA 1668A	EPA 1668A
2,3,3',4,5,5',6-Heptachlorobiphenyl	PCB-192	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(180/193)	PCB-(180/193)	EPA 1668A	EPA 1668A	EPA 1668A
2,3,3',4,4',5',6-Heptachlorobiphenyl	PCB-191	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,3',4,4',5-Heptachlorobiphenyl	PCB-170	EPA 1668A	EPA 1668A	EPA 1668A
2,3,3',4,4',5,6-Heptachlorobiphenyl	PCB-190	EPA 1668A	EPA 1668A	EPA 1668A
2,3,3',4,4',5,5-Heptachlorobiphenyl	PCB-189	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,3',5,5',6,6'-Octachlorobiphenyl	PCB-202	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,3',4,5',6,6'-Octachlorobiphenyl	PCB-201	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,4,4',5,6,6'-Octachlorobiphenyl	PCB-204	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(197/200)	PCB-(197/200)	EPA 1668A	EPA 1668A	EPA 1668A
PCB-(198/199)	PCB-(198/199)	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,3',4,4',5,6'-Octachlorobiphenyl	PCB-196	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,4,4',5,5',6-Octachlorobiphenyl	PCB-203	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,3',4,4',5,6-Octachlorobiphenyl	PCB-195	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,3',4,4',5,5'-Octachlorobiphenyl	PCB-194	EPA 1668A	EPA 1668A	EPA 1668A
2,3,3',4,4',5,5',6-Octachlorobiphenyl	PCB-205	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl	PCB-208	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl	PCB-207	EPA 1668A	EPA 1668A	EPA 1668A
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	PCB-206	EPA 1668A	EPA 1668A	EPA 1668A
Decachlorobiphenyl	PCB-209	EPA 1668A	EPA 1668A	EPA 1668A

Parameter/Analyte	Tissue
<u>Per- and Polyfluoroalkyl Substances (PFAS)</u>	
10:2 Fluorotelomer sulfonic acid (10:2 FTS)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
4,8-Dioxa-3H-perfluorononanoic acid (DONA)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
4:2 Fluorotelomer sulfonic acid (4:2 FTS)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
6:2 Fluorotelomer sulfonic acid (6:2 FTS)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3

<u>Parameter/Analyte</u>	<u>Tissue</u>
8:2 Fluorotelomer sulfonic acid (8:2 FTS)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
N-Ethylperfluorooctane sulfonamido acetic acid (NEtFOSAA)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
N-Ethylperfluorooctane sulfonamide (EtFOSAm)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
N-Ethylperfluorooctane sulfonamide ethanol (EtFOSE)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
N-Methyl perfluorooctane sulfonamido acetic acid (NMeFOSAA)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
N-Methylperfluorooctane sulfonamide (MeFOSA)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
N-Methylperfluorooctane sulfonamido ethanol (MeFOSE)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
Perfluorobutane sulfonic acid (PFBS)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
Perfluorobutanoic acid (PFBA)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
Perfluorodecane sulfonic acid (PFDS)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
Perfluorodecanoic acid (PFDA)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
Perfluorododecane sulfonic acid (PFDoS)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
Perfluorododecanoic acid (PFDOA)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
Perfluoroheptane sulfonic acid (PFHpS)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
Perfluoroheptanoic acid (PFHpA)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
Perfluorohexadecanoic acid (PFHxDA)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
Perfluorohexane sulfonic acid (PFHxS)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
Perfluorohexanoic acid (PFHxA)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
Perfluorononane sulfonic acid (PFNS)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
Perfluorononanoic acid (PFNA)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
Perfluorooctandecanoic acid (PFODA)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
Perfluorooctane sulfonamide (PFOSAm)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
Perfluorooctane sulfonic acid (PFOS)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
Perfluorooctanoic acid (PFOA)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3

<u>Parameter/Analyte</u>	<u>Tissue</u>
Perfluoropentane sulfonic acid (PFPeS)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
Perfluoropentanoic acid (PFPeA)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
Perfluorotetradecanoic acid (PFTDA)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
Perfluorotridecanoic acid (PFTrDA)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3
Perfluoroundecanoic acid (PFUnDA)	PFAS by LCMSMS Compliant with Table B-15 of the DoD/DOE QSM V5.3

In addition, in recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with ISO IEC 17025:2017, the 2016 and 2009 TNI Environmental Testing Laboratory Standard), accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

Testing Technologies: Gas Chromatography-Flame Ionization Detector, Gas Chromatography-Mass Spectrometry, Gas Chromatography-Photo Ionization Detector, Inductively Coupled Plasma-Mass Spectrometry, Inductively Coupled Plasma-Mass Spectrometry, Manual Cold Vapor Atomic Absorption, Colorimetric, Electrometric

<u>Parameter/Analyte</u>	<u>Air</u>
<u>Volatile Organic Compounds</u>	
1,1,1-Trichloroethane	EPA TO15-1999/TO15 SIM
1,1,2,2-Tetrachloroethane	EPA TO15-1999/TO15 SIM
1,1,2-Trichloroethane	EPA TO15-1999/TO15 SIM
1,1-Dichloroethane	EPA TO15-1999/TO15 SIM
1,1-Dichloroethene	EPA TO15-1999/TO15 SIM
1,2,3-Trimethylbenzene	EPA TO15-1999
1,2,4-Trichlorobenzene	EPA TO15-1999
1,2,4-Trimethylbenzene	EPA TO15-1999/TO-3
1,2-Dibromoethane	EPA TO15-1999/TO15 SIM
1,2-Dichlorobenzene	EPA TO15-1999
1,2-Dichloroethane	EPA TO15-1999/TO15 SIM
1,2-Dichloropropane	EPA TO15-1999/TO15 SIM
1,3,5-Trimethylbenzene	EPA TO15-1999/TO-3
1,3-Butadiene	EPA TO15-1999/TO15 SIM
1,3-Dichlorobenzene	EPA TO15-1999
1,4-Dichlorobenzene	EPA TO15-1999
1,4-Dioxane	EPA TO15-1999
2,2,4-Trimethylpentane	EPA TO15-1999
4-Isopropyltoluene (p-Cymene)	EPA TO15-1999
Benzene	EPA TO15-1999/TO15 SIM/TO-3
Benzylchloride	EPA TO15-1999
Bromomethane (Methyl bromide)	EPA TO15-1999
Carbon disulfide	EPA TO15-1999
Carbon tetrachloride	EPA TO15-1999/TO15 SIM
Carbon dioxide	EPA TO15-1999/Method 3C
Carbon monoxide	Method 3C

<u>Parameter/Analyte</u>	<u>Air</u>
Chlorobenzene	EPA TO15-1999
Chloroethane (Ethyl Chloride)	EPA TO15-1999
Chloroform	EPA TO15-1999/TO15 SIM
Chloromethane (Methyl Chloride)	EPA TO15-1999
cis-1,2-Dichloroethene	EPA TO15-1999/TO15 SIM
cis-1,3-Dichloropropene	EPA TO15-1999/TO15 SIM
Di-isopropyl ether (DIPE)	EPA TO15-1999
Ethane	EPA TO-3
Ethene	EPA TO-3
Ethylbenzene	EPA TO15-1999/TO-3
Ethyl-t-butyl ether (ETBE)	EPA TO15-1999
Hexachloro-1,3-butadiene	EPA TO15-1999
Isopentane	EPA TO15-1999
Isopropylbenzene (Cumene)	EPA TO15-1999
m&p-Xylene	EPA TO15-1999/TO15 SIM /TO-3
Methane	EPA TO-3/Method 3C
Methyl methacrylate	EPA TO15-1999
Methylcyclohexane	EPA TO15-1999
Methylene Chloride	EPA TO15-1999
Nitrogen	Method 3C
Oxygen	Method 3C
o-xylene	EPA TO15-1999/TO-3
Propylene (Methylethylene)	EPA TO15-1999
Styrene	EPA TO15-1999
Tetrachloroethene	EPA TO15-1999/TO15 SIM
Toluene	EPA TO15-1999/TO-3
trans-1,3-Dichloropropene	EPA TO15-1999/TO15 SIM
Trichloroethene	EPA TO15-1999
2-Butanone (methylethylketone - MEK)	EPA TO15-1999
4-Ethyltoluene	EPA TO15-1999
Acetone	EPA TO15-1999/TO15 SIM
Acrolein (Propenal)	EPA TO15-1999/TO15 SIM
Acrylonitrile	EPA TO15-1999/TO15 SIM
Allyl Chloride (3-Chloropropane)	EPA TO15-1999/TO15 SIM
Bromodichloromethane	EPA TO15-1999/TO15 SIM
Bromoform	EPA TO15-1999
Cyclohexane	EPA TO15-1999
Dibromochloromethane	EPA TO15-1999
Ethanol	EPA TO15-1999
Ethyl acetate	EPA TO15-1999
Methyl butyl ketone (2-Hexanone)	EPA TO15-1999
Methyl isobutyl ketone (4-Methyl-2-pentanone, MIBK)	EPA TO15-1999
Methyl-tert-butyl ether (MTBE)	EPA TO15-1999/TO-3
Naphthalene	EPA TO15-1999
n-Butylbenzene	EPA TO15-1999
n-Heptane	EPA TO15-1999
n-Hexane	EPA TO15-1999/TO-3

<u>Parameter/Analyte</u>	<u>Air</u>
n-Propylbenzene	EPA TO15-1999
2-Propanol (IPA)	EPA TO15-1999
sec-Butylbenzene	EPA TO15-1999
Tetrahydrofuran	EPA TO15-1999
tert-Amyl methyl ether (TAME)	EPA TO15-1999
tert-Butyl alcohol (TBA)	EPA TO15-1999
tert-Butylbenzene	EPA TO15-1999
trans-1,2-Dichloroethene	EPA TO15-1999/TO15 SIM
Vinyl acetate	EPA TO15-1999
Vinyl bromide (Bromoethane)	EPA TO15-1999
Vinyl Chloride	EPA TO15-1999/TO15 SIM
Xylene (total)	EPA TO15-1999
THC as Gas	EPA TO15-1999/EPA TO-3
Dichlorotetrafluoroethane (Freon-114)	EPA TO15-1999
Trichlorotrifluoroethane (Freon-113)	EPA TO15-1999
Chlorodifluoromethane (Freon-22)	EPA TO15-1999
Dichlorodifluoromethane (Freon-12)	EPA TO15-1999
Trichlorofluoromethane (Freon-11)	EPA TO15-1999
<u>Extractable Organics</u>	
2,3,7,8-TCDD	Method 23/TO-9
2,3,7,8-TCDF	Method 23/TO-9
1,2,3,7,8-PeCDF	Method 23/TO-9
2,3,4,7,8-PeCDF	Method 23/TO-9
1,2,3,7,8-PeCDD	Method 23/TO-9
1,2,3,4,7,8-HxCDF	Method 23/TO-9
1,2,3,6,7,8-HxCDF	Method 23/TO-9
2,3,4,6,7,8-HxCDF	Method 23/TO-9
1,2,3,7,8,9-HxCDF	Method 23/TO-9
1,2,3,4,7,8-HxCDD	Method 23/TO-9
1,2,3,6,7,8-HxCDD	Method 23/TO-9
1,2,3,7,8,9-HxCDD	Method 23/TO-9
1,2,3,4,6,7,8-HpCDF	Method 23/TO-9
1,2,3,4,7,8,9-HpCDF	Method 23/TO-9
1,2,3,4,6,7,8-HpCDD	Method 23/TO-9
OCDF	Method 23/TO-9
OCDD	Method 23/TO-9
Total HpCDD	Method 23/TO-9
Total HpCDF	Method 23/TO-9
Total HxCDD	Method 23/TO-9
Total HxCDF	Method 23/TO-9
Total PeCDD	Method 23/TO-9
Total PeCDF	Method 23/TO-9
Total TCDD	Method 23/TO-9
Total TCDF	Method 23/TO-9

<u>Parameter/Analyte</u>	<u>Nonpotable Water</u>	<u>Solid Hazardous Waste</u>
<u>Metals</u>		
Aluminum	EPA 6010D EPA 6020B	EPA 6010D EPA 6020B
Antimony	EPA 6010D EPA 6020B	EPA 6010D EPA 6020B
Arsenic	EPA 6010D EPA 6020B	EPA 6010D EPA 6020B
Barium	EPA 6010D EPA 6020B	EPA 6010D EPA 6020B
Beryllium	EPA 6010D EPA 6020B	EPA 6010D EPA 6020B
Bismuth	EPA 6020B	EPA 6020B
Boron	EPA 6010D EPA 6020B	EPA 6010D EPA 6020B
Cadmium	EPA 6010D EPA 6020B	EPA 6010D EPA 6020B
Calcium	EPA 6010D EPA 6020B	EPA 6010D EPA 6020B
Chromium	EPA 6010D EPA 6020B	EPA 6010D EPA 6020B
Cobalt	EPA 6010D EPA 6020B	EPA 6010D EPA 6020B
Copper	EPA 6010D EPA 6020B	EPA 6010D EPA 6020B
Iron	EPA 6010D EPA 6020B	EPA 6010D EPA 6020B
Lead	EPA 6010D EPA 6020B	EPA 6010D EPA 6020B
Lithium	EPA 6020B	EPA 6020B
Magnesium	EPA 6010D EPA 6020B	EPA 6010D EPA 6020B
Manganese	EPA 6010D EPA 6020B	EPA 6010D EPA 6020B
Mercury	EPA 7470A	EPA 7471B
Molybdenum	EPA 6010D EPA 6020B	EPA 6010D EPA 6020B
Nickel	EPA 6010D EPA 6020B	EPA 6010D EPA 6020B
Phosphorus	EPA 6010D	EPA 6010D
Potassium	EPA 6010D EPA 6020B	EPA 6010D EPA 6020B
Selenium	EPA 6010D EPA 6020B	EPA 6010D EPA 6020B
Silica	EPA 6020B	EPA 6020B
Silicon	EPA 6010D EPA 6020B	EPA 6010D EPA 6020B
Silver	EPA 6010D EPA 6020B	EPA 6010D EPA 6020B

<u>Parameter/Analyte</u>	<u>Nonpotable Water</u>	<u>Solid Hazardous Waste</u>
Sodium	EPA 6010D EPA 6020B	EPA 6010D EPA 6020B
Strontium	EPA 6010D EPA 6020B	EPA 6010D EPA 6020B
Thallium	EPA 6010D EPA 6020B	EPA 6010D EPA 6020B
Tin	EPA 6010D EPA 6020B	EPA 6010D EPA 6020B
Titanium	EPA 6010D EPA 6020B	EPA 6010D EPA 6020B
Uranium	EPA 6010D EPA 6020B	EPA 6010D EPA 6020B
Vanadium	EPA 6010D EPA 6020B	EPA 6010D EPA 6020B
Zinc	EPA 6010D EPA 6020B	EPA 6010D EPA 6020B
<u>Inorganic</u>		
Chloride	EPA 300.0 EPA 9056A	-----
Chemical Oxygen Demand – COD	EPA 410.4 SM 5220D	-----
Cyanide	SM 4500 CN-E	-----
Hardness	SM 2340B	-----
Nitrate	EPA 300.0 EPA 9056A	-----
Nitrate-Nitrate	EPA 353.2	-----
Nitrite	EPA 300.0 EPA 9056A	-----
Oil and Grease	EPA 1664B	EPA 9071B
pH	SM 4500 H+B	EPA 9045D
Total Petroleum Hydrocarbons - TPH	EPA 1664B	EPA 9071B
Alkalinity	SM 2320B	-----
Ammonia	EPA 350.1	-----
Conductivity	EPA 120.1	-----
Fluoride	EPA 300.0 EPA 9056A	-----
Paint Filters	-----	EPA 9095B
Sulfate	EPA 300.0 EPA 9056A	-----
Total Phosphorus	SM 4500 P-F	-----
Settleable Solids	SM 2540F	-----
Total Dissolved Solids	SM 2540C	-----
Total Suspended Solids	SM 2540D	-----
Turbidity	EPA 180.1	-----
Ferrous Iron	SM 3500 Fe-B	-----
<u>Organic</u>		
Diesel Range Organics - DRO	EPA 8015D	EPA 8015D
Gasoline Range Organics - GRO	EPA 8015D	EPA 8015D

<u>Parameter/Analyte</u>	<u>Nonpotable Water</u>	<u>Solid Hazardous Waste</u>
Aldrin	EPA 8081B	EPA 8081B
alpha-BHC	EPA 8081B	EPA 8081B
beta-BHC	EPA 8081B	EPA 8081B
gamma-BHC (Lindane)	EPA 8081B	EPA 8081B
alpha-Chlordane	EPA 8081B	EPA 8081B
gamma-Chordane	EPA 8081B	EPA 8081B
4,4'-DDD	EPA 8081B	EPA 8081B
4,4'-DDE	EPA 8081B	EPA 8081B
4,4'-DDT	EPA 8081B	EPA 8081B
Dieldrin	EPA 8081B	EPA 8081B
Endosulfan I	EPA 8081B	EPA 8081B
Endosulfan II	EPA 8081B	EPA 8081B
Endosulfan sulfate	EPA 8081B	EPA 8081B
Endrin	EPA 8081B	EPA 8081B
Endrin aldehyde	EPA 8081B	EPA 8081B
Endrin ketone	EPA 8081B	EPA 8081B
Heptachlor	EPA 8081B	EPA 8081B
Heptachlor epoxide	EPA 8081B	EPA 8081B
Methoxychlor	EPA 8081B	EPA 8081B
Toxaphene	EPA 8081B	EPA 8081B
Chlordane (Technical)	EPA 8081B	EPA 8081B
PCB-1016 (Aroclor 1016)	EPA 8082A	EPA 8082A
PCB-1221 (Aroclor 1221)	EPA 8082A	EPA 8082A
PCB-1232 (Aroclor 1232)	EPA 8082A	EPA 8082A
PCB-1242 (Aroclor 1242)	EPA 8082A	EPA 8082A
PCB-1248 (Aroclor 1248)	EPA 8082A	EPA 8082A
PCB-1254 (Aroclor 1254)	EPA 8082A	EPA 8082A
PCB-1260 (Aroclor 1260)	EPA 8082A	EPA 8082A
PCB-1262 (Aroclor 1262)	EPA 8082A	EPA 8082A
PCB-1268 (Aroclor 1268)	EPA 8082A	EPA 8082A
1,2-Dibromo-3-chloropropane	EPA 8011	-----
1,2-Dibromoethane (EDB)	EPA 8011	-----
1,2,4-Trichlorobenzene	EPA 8270E	EPA 8270E
1,2-Dichlorobenzene	EPA 8270E	EPA 8270E
1,3-Dichlorobenzene	EPA 8270E	EPA 8270E
1,4-Dichlorobenzene	EPA 8270E	EPA 8270E
1,4-Dioxane	EPA 8270E SIM	-----
1-Methylnaphthalene	EPA 8270E EPA 8270E SIM	EPA 8270E EPA 8270E SIM
1-Nitropyrene	EPA 8270E SIM	EPA 8270E SIM
2,4,5-Trichlorophenol	EPA 8270E	EPA 8270E
2,4,6-Trichlorophenol	EPA 8270E	EPA 8270E
2,4-Dichlorophenol	EPA 8270E	EPA 8270E
2,4-Dimethylphenol	EPA 8270E	EPA 8270E
2,4-Dinitrotoluene	EPA 8270E	EPA 8270E
2,4-Dinitrophenol	EPA 8270E	EPA 8270E

Parameter/Analyte	Nonpotable Water	Solid Hazardous Waste
2,6-Dinitrotoluene	EPA 8270E	EPA 8270E
2-Chloronaphthalene	EPA 8270E EPA 8270E SIM	EPA 8270E EPA 8270E SIM
2-Chlorophenol	EPA 8270E	EPA 8270E
2-Methylnaphthalene	EPA 8270E EPA 8270E SIM	EPA 8270E EPA 8270E SIM
2-Methylphenol(o-Cresol)	EPA 8270E	EPA 8270E
2-Nitroaniline	EPA 8270E	EPA 8270E
2-Nitrofluorene	EPA 8270E SIM	EPA 8270E SIM
2-Nitrophenol	EPA 8270E	EPA 8270E
3&4-Methylphenol	EPA 8270E	EPA 8270E
3,3'-Dichlorobenzidine	EPA 8270E	EPA 8270E
3-Methylcholanthrene	EPA 8270E SIM	EPA 8270E SIM
3-Nitroaniline	EPA 8270E	EPA 8270E
4,6-Dinitro-2-methylphenol	EPA 8270E	EPA 8270E
4-Bromophenylphenyl ether	EPA 8270E	EPA 8270E
4-Chloro-3-methylphenol	EPA 8270E	EPA 8270E
4-Chlorophenylphenyl ether	EPA 8270E	EPA 8270E
4-Nitroaniline	EPA 8270E	EPA 8270E
4-Nitrophenol	EPA 8270E	EPA 8270E
4-Nitropyrene	EPA 8270E SIM	EPA 8270E SIM
5-Methylchrysene	EPA 8270E SIM	EPA 8270E SIM
5-Nitroacenaphthene	EPA 8270E SIM	EPA 8270E SIM
6-Nitrochrysene	EPA 8270E SIM	EPA 8270E SIM
7,12-Dimethylbenz(a)anthracene	EPA 8270E SIM	EPA 8270E SIM
7H-Dibenzo(c,g)carbazole	EPA 8270E SIM	EPA 8270E SIM
Acenaphthene	EPA 8270E EPA 8270E SIM	EPA 8270E EPA 8270E SIM
Acenaphthylene	EPA 8270E EPA 8270E SIM	EPA 8270E EPA 8270E SIM
Anthracene	EPA 8270E EPA 8270E SIM	EPA 8270E EPA 8270E SIM
Benzo(a)anthracene	EPA 8270E EPA 8270E SIM	EPA 8270E EPA 8270E SIM
Benzo(a)pyrene	EPA 8270E EPA 8270E SIM	EPA 8270E EPA 8270E SIM
Benzo(e)pyrene	EPA 8270E SIM	EPA 8270E SIM
Benzo(g,h,i)perylene	EPA 8270E EPA 8270E SIM	EPA 8270E EPA 8270E SIM
bis(2-Chloroethoxy)methane	EPA 8270E SIM	EPA 8270E SIM
bis(2-Chloroethyl)ether	EPA 8270E SIM	EPA 8270E SIM
bis(2-Chloroisopropyl)ether	EPA 8270E SIM	EPA 8270E SIM
bis(2-Ethylhexyl)phthalate	EPA 8270E SIM	EPA 8270E SIM
Butylbenzylphthalate	EPA 8270E SIM	EPA 8270E SIM
Carbazole	EPA 8270E EPA 8270E SIM	EPA 8270E EPA 8270E SIM

<u>Parameter/Analyte</u>	<u>Nonpotable Water</u>	<u>Solid Hazardous Waste</u>
Chrysene	EPA 8270E EPA 8270E SIM	EPA 8270E EPA 8270E SIM
Dibenz(a,h)acridine	EPA 8270E SIM	EPA 8270E SIM
Dibenz(a,h)anthracene	EPA 8270E EPA 8270E SIM	EPA 8270E EPA 8270E SIM
Dibenz(a,j)aridine	EPA 8270E SIM	EPA 8270E SIM
Dibenzo(a,e)pyrene	EPA 8270E SIM	EPA 8270E SIM
Dibenzo(a,h)pyrene	EPA 8270E SIM	EPA 8270E SIM
Dibenzo(a,i)pyrene	EPA 8270E SIM	EPA 8270E SIM
Dibenzo(a,l)pyrene	EPA 8270E SIM	EPA 8270E SIM
Dibenzofuran	EPA 8270E EPA 8270E SIM	EPA 8270E EPA 8270E SIM
Diethylphthalate	EPA 8270E	EPA 8270E
Dimethylphthalate	EPA 8270E	EPA 8270E
di-n-Butylphthalate	EPA 8270E	EPA 8270E
di-n-Octylphthalate	EPA 8270E	EPA 8270E
Fluoranthene	EPA 8270E EPA 8270E SIM	EPA 8270E EPA 8270E SIM
Fluroene	EPA 8270E EPA 8270E SIM	EPA 8270E EPA 8270E SIM
Hexachloro-1,3-butadiene	EPA 8270E	EPA 8270E
Hexachlorobenzene	EPA 8270E	EPA 8270E
Hexachlorocyclopentadiene	EPA 8270E	EPA 8270E
Hexachloroethane	EPA 8270E	EPA 8270E
Indeno(1,2,3-cd)pyrene	EPA 8270E EPA 8270E SIM	EPA 8270E EPA 8270E SIM
Isophorone	EPA 8270E	EPA 8270E
Naphthalene	EPA 8270E EPA 8270E SIM	EPA 8270E EPA 8270E SIM
Nitrobenzene	EPA 8270E	EPA 8270E
n-Nitroso-di-n-propylamine	EPA 8270E	EPA 8270E
n-Nitrosodiphenylamine	EPA 8270E	EPA 8270E
Pentachlorophenol	EPA 8270E EPA 8270E SIM	EPA 8270E
Perylene	EPA 8270E SIM	EPA 8270E SIM
Phenanthrene	EPA 8270E EPA 8270E SIM	EPA 8270E EPA 8270E SIM
Phenol	EPA 8270E	EPA 8270E
Pyrene	EPA 8270E EPA 8270E SIM	EPA 8270E EPA 8270E SIM
Quinoline	EPA 8270E SIM	EPA 8270E SIM
1,1,1,2-Tetrachloroethane	EPA 8260D	EPA 8260D
1,1,1-Trichloroethane	EPA 8260D	EPA 8260D
1,1,2,2-Tetrachloroethane	EPA 8260D	EPA 8260D
1,1,2-Trichloroethane	EPA 8260D	EPA 8260D
1,1,2-Trichlorotrifluoroethane	EPA 8260D	EPA 8260D
1,1-Dichloroethane	EPA 8260D	EPA 8260D

<u>Parameter/Analyte</u>	<u>Nonpotable Water</u>	<u>Solid Hazardous Waste</u>
1,1-Dichloroethene	EPA 8260D	EPA 8260D
1,1-Dichloropropene	EPA 8260D	EPA 8260D
1,2,3-Trichlorobenzene	EPA 8260D	EPA 8260D
1,2,3-Trichloropropane	EPA 8260D EPA 8260D SIM	EPA 8260D
1,2,3-Trimethylbenzene	EPA 8260D	-----
1,2,4-Trichlorobenzene	EPA 8260D	EPA 8260D
1,2,4-Trimethylbenzene	EPA 8260D	EPA 8260D
1,2-Dibromo-3-chloropropane	EPA 8260D EPA 8260D SIM	EPA 8260D
1,2-Dibromoethane (EDB)	EPA 8260D EPA 8260D SIM	EPA 8260D
1,2-Dichlorobenzene	EPA 8260D	EPA 8260D
1,2-Dichloroethane	EPA 8260D EPA 8260D SIM	EPA 8260D
1,2-Dichloropropane	EPA 8260D	EPA 8260D
1,3,5-Trimethylbenzene	EPA 8260D	EPA 8260D
1,3-Butdiene	EPA 8260D	EPA 8260D
1,3-Dichlorobenzene	EPA 8260D	EPA 8260D
1,3-Dichloropropane	EPA 8260D	EPA 8260D
1,4-Dichlorobenzene	EPA 8260D	EPA 8260D
1,4-Dioxane	EPA 8260D EPA 8260D SIM	EPA 8260D
2,2-Dichloropropane	EPA 8260D	EPA 8260D
2-Butanone (MEK)	EPA 8260D	EPA 8260D
2-Chloroethylvinyl ether	EPA 8260D	EPA 8260D
2-Chlorotoluene	EPA 8260D	EPA 8260D
2-Hexanone	EPA 8260D	EPA 8260D
2-Methylnaphthalene	EPA 8260D	EPA 8260D
4-Chlorotoluene	EPA 8260D	EPA 8260D
4-Methyl-2-pentanone (MIBK)	EPA 8260D	EPA 8260D
Acetone	EPA 8260D	EPA 8260D
Acrolein	EPA 8260D	EPA 8260D
Acrylonitrile	EPA 8260D	EPA 8260D
Allyl Chloride	EPA 8260D	EPA 8260D
Benzene	EPA 8260D	EPA 8260D
Bromobenzene	EPA 8260D	EPA 8260D
Bromochloromethane	EPA 8260D	EPA 8260D
Bromodichloromethane	EPA 8260D	EPA 8260D
Bromoform	EPA 8260D	EPA 8260D
Bromomethane	EPA 8260D	EPA 8260D
Carbon disulfide	EPA 8260D	EPA 8260D
Carbon tetrachloride	EPA 8260D EPA 8260D SIM	EPA 8260D
Chlorobenzene	EPA 8260D	EPA 8260D
Chloroethane	EPA 8260D	EPA 8260D
Chloroform	EPA 8260D	EPA 8260D
Chloromethane	EPA 8260D	EPA 8260D

<u>Parameter/Analyte</u>	<u>Nonpotable Water</u>	<u>Solid Hazardous Waste</u>
cis-1,2-Dichloroethene	EPA 8260D	EPA 8260D
cis-1,3-Dichloropropene	EPA 8260D EPA 8260D SIM	EPA 8260D
Cyclohexane	EPA 8260D	EPA 8260D
Dibromochloromethane	EPA 8260D EPA 8260D SIM	EPA 8260D
Dibromomethane	EPA 8260D	EPA 8260D
Dichlorodifluoromethane	EPA 8260D	EPA 8260D
Dichlorofluoromethane	EPA 8260D	EPA 8260D
Diethyl ether (Ethyl ether)	EPA 8260D	EPA 8260D
Di-isopropyl ether	EPA 8260D	EPA 8260D
Ethylbenzene	EPA 8260D	EPA 8260D
Hexachloro-1,3-butadiene	EPA 8260D EPA 8260D SIM	EPA 8260D
Iodomethane	EPA 8260D	EPA 8260D
Isopropylbenzene (Cumene)	EPA 8260D	EPA 8260D
m&p-Xylene	EPA 8260D	EPA 8260D
Methyl-tert-butyl ether (MTBE)	EPA 8260D	EPA 8260D
Methylene Chloride	EPA 8260D	EPA 8260D
Naphthalene	EPA 8260D	EPA 8260D
n-Butanol	EPA 8260D	-----
n-Butylbenzene	EPA 8260D	EPA 8260D
n-Hexane	EPA 8260D	EPA 8260D
n-Propylbenzene	EPA 8260D	EPA 8260D
o-Xylene	EPA 8260D	EPA 8260D
p-Isopropyltoluene	EPA 8260D	EPA 8260D
sec-Butylbenzene	EPA 8260D	EPA 8260D
Styrene	EPA 8260D	EPA 8260D
tert-Butylbenzene	EPA 8260D	EPA 8260D
Tetrachloroethene	EPA 8260D	EPA 8260D
Tetrahydrofuran	EPA 8260D	EPA 8260D
Toluene	EPA 8260D	EPA 8260D
trans-1,2-Dichloroethene	EPA 8260D	EPA 8260D
trans-1,3-Dichloropropene	EPA 8260D EPA 8260D SIM	EPA 8260D
trans-1,4-Dichloro-2-butene	EPA 8260D	EPA 8260D
Trichloroethene	EPA 8260D EPA 8260D SIM	EPA 8260D
Trichlorofluoromethane	EPA 8260D	EPA 8260D
Vinyl acetate	EPA 8260D	EPA 8260D
Vinyl chloride	EPA 8260D EPA 8260D SIM	EPA 8260D
Xylene (Total)	EPA 8260D	EPA 8260D
Gasoline Range Organics - GRO	AK101	AK101
Diesel Range Organics - DRO	AK102	AK102
Residual Range Organics	AK103	AK103
Ethane	RSK-175	-----
Ethene	RSK-175	-----

<u>Parameter/Analyte</u>	<u>Nonpotable Water</u>	<u>Solid Hazardous Waste</u>
Methane	RSK-175	-----
n-Propane	RSK-175	-----
TCLP	EPA 1311	EPA 1311
SPLP	EPA 1312	EPA 1312
Waste Extraction Test (WET)	CCR Chapter 11, Article 5, Appendix II	CCR Chapter 11, Article 5, Appendix II

<u>Test Method(s)</u>	<u>Matrix</u>	<u>Extraction Method(s)</u>
8015D DRO, 8081B, 8082A, 8270E, 8270E SIM	Water	EPA 3510C
8270E SIM	Water	EPA 3511
8270E	Soil	EPA 3546
8015D DRO, 8081B, 8082A, 8270E, 8270E SIM	Solid	EPA 3550C
8260D	Solid	EPA 5035A/5030B
8015D GRO	Solid	EPA 5030B
6010D, 6020B	Water	EPA 3010A/3020A
6010D, 6020B	Solid	EPA 3050B
6010D, 6020B, 7470A, 7471B, 8081B, 8260D, 8270E	Solid/Liquid	EPA 1311 TCLP/1312 SPLP
6010B, 6010D	Solid/Liquid	CCR Chapter 11, Article 5, Appendix II

*Standard Methods (SM) refers to the current online edition.

<u>Parameter/Analyte</u>	<u>Potable Water</u>
2,3,7,8-TCDD	EPA 1613B

WYOMING STORAGE TANK

In addition, in recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with ISO IEC 17025:2017 and to the Wyoming Storage Tank Remediation Laboratory Accreditation Program, accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below on water and solids:

<u>Parameter</u>	<u>Analyte</u>	<u>Method(s)</u>	<u>NPW</u>	<u>SCM</u>
Extractable Organics	Diesel Range Organics C10-C32	EPA 8015C	X	X
Metals	Cadmium	EPA 6010D	X	X
Metals	Chromium	EPA 6010D	X	X
Metals	Lead	EPA 6010D	X	X
Purgeable Organics	Gasoline Range Organics C6-C10	EPA 8015B/8015C	X	X
Purgeable Organics	1,2-Dibromomethane	EPA 8011	X	-----

The State of Wisconsin Department of Natural Resources

has granted

Accreditation under NR 149

to

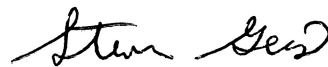
***Pace Analytical Services, LLC-Minneapolis
MN***

FID: 999407970

The laboratory is accredited to perform environmental sample analysis in support of covered environmental programs per matrix for the combination of analyte and technology or analyte and method as specified in the attached Scopes of Accreditation.

Printed on: August 13, 2022

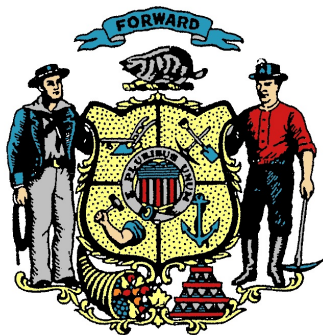
Expires on: August 31, 2023



Steven Geis, Chief
Certification Services



Preston D. Cole Secretary
Department of Natural Resources



This certificate does not guarantee validity of data generated, but indicates the methodology, equipment, quality control practices, records, and proficiency of the laboratory have been reviewed and found to satisfy the requirements of chapter NR 149, Wisconsin Administrative Code.



Wisconsin Scope of Accreditation

Laboratory FID: 999407970
 CERTIFICATION
 Aqueous (Non-potable Water)

Pace Analytical Services, LLC-Minneapolis MN
1700 SE Elm Street
Minneapolis, MN 55414

Printed on: 8/13/2022
Expires on: 8/31/2023

Colorimetric or Turbidimetric	Ammonia as N
	Chemical Oxygen Demand (COD)
	Chloride
	Nitrate
	Nitrate + Nitrite
	Nitrite
	Orthophosphate
	Sulfate
	Turbidity
Electrometric Assays (ISE)	pH
	Specific Conductance
Gravimetric Assays - Residue (solids)	Residue, Filterable (TDS)
	Residue, Nonfilterable (TSS)
	Residue, Total (Total Solids)
	Residue, Volatile (TVS)
	Residue, Volatile, Nonfilterable (TVSS)
Extraction/Gravimetric Assays - Oil & Grease as HEM	Oil & Grease as HEM
	Oil & Grease as HEM, Silica Gel Treated (SGT)
Ion Chromatography (IC)	Bromide
	Chloride
	Nitrate

This laboratory is accredited under NR 149 to perform environmental sample analysis in support of covered environmental programs for the combination of analyte or analyte group and technology in the aqueous and non-aqueous matrices and analyte or analyte group and method in the drinking water matrix as specified in this Scope of Accreditation.



Wisconsin Scope of Accreditation

Laboratory FID: 999407970
CERTIFICATION
Aqueous (Non-potable Water)

Ion Chromatography (IC)	Nitrite
	Sulfate
Titrimetric or Potentiometric Titration Assays	Alkalinity
Cold Vapor Atomic Absorption Spectrophotometry (CVAA)	Mercury
Inductively Coupled Plasma Emission Spectrophotometry (ICP)	Aluminum
	Antimony
	Arsenic
	Barium
	Beryllium
	Boron
	Cadmium
	Calcium
	Chromium, Total
	Cobalt
	Copper
	Hardness, Total as CaCO₃
	Iron
	Lead
	Magnesium
	Manganese
	Molybdenum
Nickel	

This laboratory is accredited under NR 149 to perform environmental sample analysis in support of covered environmental programs for the combination of analyte or analyte group and technology in the aqueous and non-aqueous matrices and analyte or analyte group and method in the drinking water matrix as specified in this Scope of Accreditation.



Wisconsin Scope of Accreditation

Laboratory FID: 999407970
CERTIFICATION
Aqueous (Non-potable Water)

Inductively Coupled Plasma Emission Spectrophotometry (ICP)	Potassium
	Selenium
	Silver
	Sodium
	Thallium
	Tin
	Titanium
	Vanadium
	Zinc
Inductively Coupled Plasma-Mass Spectrometry (ICP/MS)	Aluminum
	Antimony
	Arsenic
	Barium
	Beryllium
	Bismuth
	Boron
	Cadmium
	Calcium
	Chromium, Total
	Cobalt
	Copper
	Iron

This laboratory is accredited under NR 149 to perform environmental sample analysis in support of covered environmental programs for the combination of analyte or analyte group and technology in the aqueous and non-aqueous matrices and analyte or analyte group and method in the drinking water matrix as specified in this Scope of Accreditation.



Wisconsin Scope of Accreditation

Laboratory FID: 999407970
 CERTIFICATION
 Aqueous (Non-potable Water)

Inductively Coupled Plasma-Mass Spectrometry (ICP/MS)	Lead
	Lithium
	Magnesium
	Manganese
	Molybdenum
	Nickel
	Potassium
	Selenium
	Silicon
	Silver
	Sodium
	Strontium
	Thallium
	Tin
	Titanium
	Vanadium
Zinc	
Gas Chromatography (GC)	## PCB as AROCLORS (group)
	## PESTICIDES, ORGANOCHLORINE (group)
	Diesel Range Organics (DRO)
	Gasoline Range Organics (GRO)
Gas Chromatography-Mass Spectrometry (GC/MS)	## BNA - SEMIVOLATILE ORGANICS (group)

This laboratory is accredited under NR 149 to perform environmental sample analysis in support of covered environmental programs for the combination of analyte or analyte group and technology in the aqueous and non-aqueous matrices and analyte or analyte group and method in the drinking water matrix as specified in this Scope of Accreditation.



Wisconsin Scope of Accreditation

Laboratory FID: 999407970
CERTIFICATION
Aqueous (Non-potable Water)

Page 5 of 5

Gas Chromatography-Mass Spectrometry (GC/MS)	## PAH (group)
	## VOC - VOLATILE ORGANICS (group)
High Resolution Gas Chromatography-Mass Spectrometry (HRGC/MS)	## DIOXINS & FURANS (group)
	## PCB CONGENERS (group)
Liquid Chromatography-Mass Spectrometry (LC/MS)	## PFAS (group)

This laboratory is accredited under NR 149 to perform environmental sample analysis in support of covered environmental programs for the combination of analyte or analyte group and technology in the aqueous and non-aqueous matrices and analyte or analyte group and method in the drinking water matrix as specified in this Scope of Accreditation.



Wisconsin Scope of Accreditation

Laboratory FID: 999407970
CERTIFICATION
Drinking Water (Potable Water)

Page 1 of 1

Pace Analytical Services, LLC-Minneapolis MN
1700 SE Elm Street
Minneapolis, MN 55414

Printed on: 8/13/2022
Expires on: 8/31/2023

Primary Inorganics Contaminants (Non-Metals)	Nitrate - EPA 353.2
	Nitrite - EPA 353.2
Primary Inorganics Contaminants (Metals)	Copper - EPA 200.8
	Lead - EPA 200.8
	Mercury - EPA 245.1
SOC - Dioxin	2,3,7,8-TCDD (Dioxin) - EPA 1613
SOC - Miscellaneous	## PFAS (group) – EPA 537.1 (18)

This laboratory is accredited under NR 149 to perform environmental sample analysis in support of covered environmental programs for the combination of analyte or analyte group and technology in the aqueous and non-aqueous matrices and analyte or analyte group and method in the drinking water matrix as specified in this Scope of Accreditation.



Wisconsin Scope of Accreditation

Laboratory FID: 999407970
CERTIFICATION

Page 1 of 4

Non-Aqueous (Biosolids, Leachates, Soils, Tissues, & Wastes)

Pace Analytical Services, LLC-Minneapolis MN
1700 SE Elm Street
Minneapolis, MN 55414

Printed on: 8/13/2022
Expires on: 8/31/2023

Electrometric Assays (ISE)	pH
Cold Vapor Atomic Absorption Spectrophotometry (CVAA)	Mercury
Inductively Coupled Plasma Emission Spectrophotometry (ICP)	Aluminum
	Antimony
	Arsenic
	Barium
	Beryllium
	Boron
	Cadmium
	Calcium
	Chromium, Total
	Cobalt
	Copper
	Iron
	Lead
	Magnesium
	Manganese
	Molybdenum
	Nickel
Potassium	
Selenium	

This laboratory is accredited under NR 149 to perform environmental sample analysis in support of covered environmental programs for the combination of analyte or analyte group and technology in the aqueous and non-aqueous matrices and analyte or analyte group and method in the drinking water matrix as specified in this Scope of Accreditation.



Wisconsin Scope of Accreditation

Laboratory FID: 999407970
CERTIFICATION

Page 2 of 4

Non-Aqueous (Biosolids, Leachates, Soils, Tissues, & Wastes)

Inductively Coupled Plasma Emission Spectrophotometry (ICP)	Silver
	Sodium
	Thallium
	Tin
	Titanium
	Vanadium
	Zinc
Inductively Coupled Plasma-Mass Spectrometry (ICP/MS)	Aluminum
	Antimony
	Arsenic
	Barium
	Beryllium
	Bismuth
	Boron
	Cadmium
	Calcium
	Chromium, Total
	Cobalt
	Copper
	Iron
	Lead
	Lithium

This laboratory is accredited under NR 149 to perform environmental sample analysis in support of covered environmental programs for the combination of analyte or analyte group and technology in the aqueous and non-aqueous matrices and analyte or analyte group and method in the drinking water matrix as specified in this Scope of Accreditation.



Wisconsin Scope of Accreditation

Laboratory FID: 999407970
 CERTIFICATION

Non-Aqueous (Biosolids, Leachates, Soils, Tissues, & Wastes)

Inductively Coupled Plasma-Mass Spectrometry (ICP/MS)	Magnesium
	Manganese
	Molybdenum
	Nickel
	Potassium
	Selenium
	Silver
	Sodium
	Strontium
	Thallium
	Tin
	Titanium
	Vanadium
	Zinc
Gas Chromatography (GC)	## PCB as AROCLORS (group)
	## PESTICIDES, ORGANOCHLORINE (group)
	Diesel Range Organics (DRO)
	Gasoline Range Organics (GRO)
Gas Chromatography-Mass Spectrometry (GC/MS)	## BNA - SEMIVOLATILE ORGANICS (group)
	## PAH (group)
	## VOC - VOLATILE ORGANICS (group)
High Resolution Gas Chromatography-Mass Spectrometry (HRGC/MS)	## DIOXINS & FURANS (group)

This laboratory is accredited under NR 149 to perform environmental sample analysis in support of covered environmental programs for the combination of analyte or analyte group and technology in the aqueous and non-aqueous matrices and analyte or analyte group and method in the drinking water matrix as specified in this Scope of Accreditation.



Wisconsin Scope of Accreditation

Laboratory FID: 999407970
CERTIFICATION

Page 4 of 4

Non-Aqueous (Biosolids, Leachates, Soils, Tissues, & Wastes)

High Resolution Gas Chromatography-Mass Spectrometry (HRGC/MS)	## PCB CONGENERS (group)
Liquid Chromatography-Mass Spectrometry (LC/MS)	## PFAS (group)
Solid Waste Leaching Procedures	SPLP Extraction
Hazardous Waste Characteristics	TCLP Extraction

This laboratory is accredited under NR 149 to perform environmental sample analysis in support of covered environmental programs for the combination of analyte or analyte group and technology in the aqueous and non-aqueous matrices and analyte or analyte group and method in the drinking water matrix as specified in this Scope of Accreditation.

Appendix D _____

December 2022 Field Sampling Plan (Including applicable SOPs)

FIELD SAMPLING PLAN – PFAS SAMPLING

**Oconomowoc Electroplating Company, Inc. Superfund Site
W2573 Oak Street
Ashippun, Dodge County, Wisconsin 53003**

U.S. EPA ID: WID006100275

BRRTS#: 02-14-000905

WI FID: 114004220



HYDE ENVIRONMENTAL, INC.

FIELD SAMPLING PLAN – PFAS SAMPLING

**Oconomowoc Electroplating Company, Inc. Superfund Site
W2573 Oak Street
Ashippun, Dodge County, Wisconsin 53003**

U.S. EPA ID: WID006100275

BRRTS#: 02-14-000905

WI FID: 114004220

Prepared For:

Ms. Gwen Saliars
Wisconsin Dept. of Natural Resources
625 E. County Rd. Y
Oshkosh, WI 54901

Prepared By:

Hyde Environmental, Inc.
W175 N11163 Stonewood Drive, Suite 110
Germantown, WI 53022

December 05, 2022



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FIGURES

1. Location Map
2. Site Map
3. Schedule

ATTACHMENTS

- A. Aqua-Troll 500™ Operations Manual and Heron Dipper T™
Water-Level Meter Operations Manual
- B. U.S. EPA Region 5 Analytical Services Branch PFAS Sampling Fact
Sheet and PFAS-Specific Sampling Precautions, Procedures,
and Requirements document

FIELD SAMPLING PLAN – PFAS SAMPLING

**Oconomowoc Electroplating Company, Inc. Superfund Site
W2573 Oak Street
Ashippun, Dodge County, Wisconsin 53003**

**EPA ID: WID006100275
BRRTS#: 02-14-000905
WI FID: 114004220**

1.0 INTRODUCTION

This field sampling plan covers the collection of groundwater samples for analysis of Perfluorooctanoic Acid (PFOA) and Perfluorooctane Sulfonate (PFOS) compounds, collectively referred to as Per- and polyfluoroalkyl substances (PFAS), from 28 monitoring wells and eight (8) private potable wells at/near the former Oconomowoc Electroplating Company, Inc. (OECI) facility using U.S. EPA low-flow groundwater sampling techniques; *U.S. EPA Low Stress (Low Flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells (September 19, 2017)*, and following applicable Wisconsin Department of Natural Resources (WDNR) guidance.



2.0 SITE BACKGROUND

The OECI Site is located at W2573 Oak Street, Town of Ashippun, Dodge County, WI (**Figure 1**). The Site is located approximately 11 miles east of Watertown, seven (7) miles north of Oconomowoc, WI, and 28 miles west-northwest of the state's largest city, Milwaukee. The Site is in the Northeast Quarter (NE ¼) of the Southeast Quarter (SE ¼) of Section 30, Township 9 North, Range 17 East, Dodge County, WI. The Site includes approximately four (4) acres of the former electroplating facility and an additional 6.5 acres of wetland, including part of Davy Creek, a tributary of the nearby Rock River. The former OECI facility is bounded by Oak St. to the northeast; Eva St. to the northwest; Elm St. to the southwest; and a Town garage and park to the southeast. The former electroplating area is vacant, devoid of any structures, and generally grass and tree-covered. The Site is in a mixed-use neighborhood with commercial operations and railroad tracks to the northeast of the Site; single-family residents to the northeast; Village-owned buildings and the Ashippun Community Park to the southeast; and wetland, farmland, and a wastewater treatment plant to the southwest.

OECI operated an electroplating facility at the Site from 1957 until February 1991. Electroplating and finishing operations at the facility used nickel, chromium, zinc, copper brass, cadmium, and tin. Wastewater discharged from the Site contained cyanide, chromium, and acid and alkaline solutions. Degreasing at the Site resulted in the discharge of 1,1-dichloroethane (1,1-DCA), chloroform, 1,2-dichloroethane (1,2-DCA), and TCE. Between 1957 and 1972, untreated wastewater was discharged directly to the wetland south of the Site. Two (2) unlined settling lagoons were constructed on-Site in 1972, prior to the construction of an on-Site wastewater treatment plant, which was completed in 1973. The lagoons measured approximately 60 feet long by 40 wide and were approximately 5 feet deep. Concrete lined two (2) of the walls, and sloped gravel lined the others. The untreated plating sludge was known to have overflowed the lagoon banks, accumulating wastes in the adjacent wetlands between Davy Creek and the OECI facility.

In 1980, OECI contracted to remove approximately one million pounds (500 tons) of plating sludge from the lagoons. This amount only represented approximately two-thirds of the volume present in the lagoons at the time. OECI refused to remove the remainder of the wastes. In the late 1980s, U.S. EPA investigations of the Site revealed approximately 75,000 sq. ft. of wetland near the OECI facility was contaminated with metals and cyanide.

By 1990, the U.S. EPA completed a Remedial Investigation (RI) and Feasibility Study (FS). A ROD was first signed in September 1990 (later amended [AROD] in September 1991 with the addition of OU Five) and contained five (5) separate discrete actions or operable units (OUs). In general, these include the following:

OU 1 – Surface water, metal hydroxide sludge, and contaminated soil associated with the two lagoons.



FIELD SAMPLING PLAN – PFAS SAMPLING

Oconomowoc Electroplating Co., Inc., Ashippun, WI

December 05, 2022; Page 3 of 14

OU 2 – All other contaminated soil around the OECI facility not associated with the lagoons.

OU 3 – Contaminated groundwater associated with the Site.

OU 4 – The most highly contaminated sediments in Davy Creek and the wetlands.

OU 5 – Dismantle the abandoned facility and dispose of associated debris.

In the early 1990s, all OECI assets were removed, including a main process building, wastewater treatment building, waste lagoons, and other miscellaneous equipment, along with 650 cubic yards of lagoon sediments, 700 yards of contaminated soil, and approximately 6,000 cubic yards of contaminated sediments from the adjacent wetlands around Davy Creek. A groundwater pump and treat system, including a building combined with five (5) groundwater recovery wells, was installed at the Site. The system operated between 1997 and 2004. At the request of the U.S. EPA, groundwater treatment was discontinued because the system was deemed no longer effective. The recovery wells were abandoned in 2009 and the treatment building removed in early 2017.

In 2011, the ROD was amended to change the selected remedy for OU3 from groundwater extraction and treatment to either source area removal or in situ treatment, followed by MNA. Soil treatment of the area beneath the former OECI process building was completed with a zero valent iron product in 2013. The Site has been reviewed for treatment effectiveness by the U.S. EPA over five (5) 5-Year Reviews. The remedial actions for OUs 1, 2, 4, and 5 have been completed. Groundwater has been consistently monitored since 2004, with the last monitoring event completed in November 2021.



3.0 DEMONSTRATION OF QA/QC

Well sampling activities will be completed by Hyde Environmental; Inc. (Hyde) and field quality assurance/quality control (QA/QC) measures will be consistent with Hyde's *Quality Assurance Project Plan* (QAPP) dated December 7, 2022 and applicable Standard Operating Procedures (SOPs). The groundwater samples will be analyzed by Pace Analytical Services ("Pace"), whose Quality Assurance Manual is provided in Appendix B of Hyde's QAPP. Pace will produce data reports that are consistent with WDNR requirements, including:

- Cover Page
- Narrative
- Chain of Custody (COC) forms
- Analytical Results (Relational Laboratory Information Management System-RLMS)
- QC Summaries and Data (including method QC, instrument QC, and CLP forms)
- Raw Data
- Laboratory Logs (including internal COC, standard logs, instrument logs, other logs)
- Summary Table
- Laboratory QC Department Review
- Full CLP-Like Data Package

QA/QC measures related to data verification and validation are also described in Hyde's QAPP.

4.0 WELL SAMPLING PROCEDURE

Groundwater samples will be collected from 28 monitoring wells and eight (8) private potable wells. The groundwater samples collected from the monitoring wells and private potable wells will be analyzed for 33 PFAS compounds. The locations of all wells are shown on **Figure 2**.

Sampling methods and equipment will follow guidance in U.S. EPA low-flow groundwater sampling techniques; *U.S. EPA Low Stress (Low Flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells (September 19, 2017)*. Sampling activities, including decontamination of sampling equipment, will be conducted in the immediate area of the wells.

Prior to sampling each day, the multi-parameter meter (In-Situ AquaTroll 500™) and water-level meter (Heron Dipper T™) used in the following sampling activities will be calibrated per the procedures contained in their specific operation manuals. These operation manuals are included in **Attachment A**.



FIELD SAMPLING PLAN – PFAS SAMPLING

Oconomowoc Electroplating Co., Inc., Ashippun, WI

December 05, 2022; Page 5 of 14

Prior to purging and sampling, the depth to water will be measured in the monitoring wells. The depth to water in the monitoring wells will be measured manually, using an electric water level meter (Heron Dipper T™). The observer will measure the depth to water until two (2) consecutive measurements are obtained that agree within 0.01 feet. Measurements will be collected from the north side of the well casing, unless the previous measuring point is clearly marked on the well casing. The observer will record the year, month, day, hour, and minute of the measurements along with the depth to water below the measuring point, in feet. The water-level meter will be decontaminated prior to and after each use, as specified later in this section.

The depth to water will not be measured in the private potable wells.

A water quality analyzer (In-Situ AquaTroll 500™) and flow cell manufactured by In-Situ (or a comparable analyzer) will be employed to monitor water quality parameters. Groundwater will be purged from the well using a Geotech™ Model 900-1290 peristaltic pump or comparable pump. Downhole pump tubing will consist of food-grade 1/4-inch outside diameter polyethylene tubing, which will be dedicated to the particular well, attached to food-grade silicone tubing. The silicone tubing will be replaced with clean tubing between sampling locations. The end of the downhole tubing will be located within the screen interval and at a depth that will remain under water at all times. The mid-point or the lowest historical midpoint of the saturated screen length will be the target of the intake.

The monitoring wells will be purged using a low flow purging technique per EPA guidance. Care will be taken to ensure that water-level drawdown and aquifer disturbance is minimized during purging. Stabilization of indicator field parameters described below will be used to indicate that conditions are suitable for sampling to begin. Achievement of turbidity levels of less than 5 NTU, and stable drawdowns of less than 0.33 feet, while desirable, are not mandatory. Sample collection may still take place provided the indicator field parameter criteria in the procedure are met. It is recommended that low-flow sampling be conducted when the air temperature is above 32°F (0°C). If the procedure is used below 32°F, special precautions will need to be taken to prevent the groundwater from freezing in the equipment.

Using a flow-through cell, properly sized for immersion of the probes at pumping rates between 100 and 300 milliliters per minute (ml/min), measurements of pH, temperature, specific conductance, turbidity, and oxidation-reduction potential (ORP) will be made at intervals between five (5) and 10 minutes as direct readings, through a Bluetooth device connected to the In-Situ sonde. The well will be purged until stable readings of pH, temperature, specific conductance, dissolved oxygen, ORP, and turbidity are obtained. Parameters will be stabilized when three (3) consecutive readings, with interim periods of five (5) to 10 minutes, vary less than ± 0.1 pH unit, ± 3 percent S/cm for specific conductance, $\pm 0.5^\circ\text{C}$ for temperature, ± 10 millivolts (mV) for ORP, and ± 0.2 milligrams per liter for dissolved oxygen. Turbidity will be considered stabilized when variations less



FIELD SAMPLING PLAN – PFAS SAMPLING

Oconomowoc Electroplating Co., Inc., Ashippun, WI

December 05, 2022; Page 6 of 14

than ± 10 percent Nephelometric Turbidity Units (NTU) (when turbidity is greater than 10 NTUs) are observed.

If after two (2) hours of purging indicator field parameters have not stabilized, one of three (3) optional courses of action may be taken: a) continue purging until stabilization is achieved, b) discontinue purging, do not collect any samples, and record in log book that stabilization could not be achieved (documentation must describe attempts to achieve stabilization), c) discontinue purging, collect samples and provide full explanation of attempts to achieve stabilization (note: there is a risk that the analytical data obtained, especially metals and strongly hydrophobic organic analytes, may reflect a sampling bias and therefore, the data may not meet the data quality objectives of the sampling event).

If well drawdown is greater than 0.33 feet during purging, the pumping rate will be slowed to decrease drawdown. If a stabilized drawdown in the well cannot be maintained at 0.33 feet and the water level is approaching the top of the well screen, the flow rate shall be reduced, or the pump turned off for 15 minutes to allow for well recovery. If pumping at a lower rate draws water down to the top of the screened interval again, the pump shall be turned off and the well allowed to recover. If two (2) tubing volumes (including water in the flow cell) have been removed during previous purging, the well will be sampled the next time the pump is turned on. Measurements made during purging will be digitally recorded in an application specifically designed for the In-Situ sonde. Purge water volume will be measured by placing the purged water in a five-gallon bucket with graduated markings.

The groundwater sample will be collected directly from the pump tubing after the tubing has been disconnected from the flow-through cell.

The Project Manager or a designee will control field logbooks. The field logbook will receive a serialized number and be issued to the field sampler. The field logbook will be maintained by the field sampler to provide a daily record of significant events, observations, and measurements during the field investigation.

Information (except chain of custody forms) pertinent to sampling activities will be recorded in the logbooks. The books will be bound with consecutively numbered pages. Entries in the logbook will include, at a minimum, the following information:

- Name and title of author, date and time of entry, and physical/environmental conditions during field activity
- Purpose of sampling activity
- Location of sampling activity
- Name and address of field contact
- Name and title of field crew
- Name and title of visitors



FIELD SAMPLING PLAN – PFAS SAMPLING

Oconomowoc Electroplating Co., Inc., Ashippun, WI

December 05, 2022; Page 7 of 14

- Ambient weather conditions
- Sample media (e.g., groundwater, etc.)
- Sample collection method
- Number and volume of sample(s) collected
- Description of sampling point(s)
- Volume of groundwater removed before sampling
- Preservatives used
- Date and time of collection
- Sample identification number(s)
- Sample distribution (e.g., laboratory)
- Field observations
- Field measurements made, including water level
- Types of sampling and field equipment, manufacturers, and model numbers
- References for maps and photographs of the sampling location(s)
- Information pertaining to sample documentation such as:
 1. Bottle lot numbers
 2. Dates and methods of sample shipments
 3. Chain of custody record numbers

Original data recorded in field logbooks, sample labels, and chain-of-custody records will be written with waterproof black ink. The bottom of each page of a daily entry will be initialed and dated. The last page of a daily entry will be signed and dated. None of the accountable serialized documents will be destroyed. If an error is made on an accountable document assigned to one individual, that individual will make corrections by crossing a line through the error, initialing, and entering the correct information. Erroneous information will not be erased. Subsequent errors discovered on an accountable document will be corrected by the person who made the entry. Subsequent corrections will be initialed and dated.

Grab groundwater samples for PFAS analysis will be collected in 250-ml high density polyethylene or polypropylene containers (avoiding polytetrafluoroethylene [PTFE] containers) without preservative and allowing some headspace between the liquid and the lid of the container. The maximum holding time for PFAS in groundwater samples is 28 days from collection to sample extraction by the laboratory. The sample containers will be placed inside coolers and stored on ice at approximately 4 degrees centigrade (4°C). Samples will be shipped, as soon as possible, to the laboratory, using an overnight method of delivery.

Laboratory sample analysis for PFAS will follow the Wisconsin method guidance document, *Wisconsin PFAS Aqueous (Non-Potable Water) and Non-Aqueous Matrices Method Expectations, Version 12.16.2019, Per-and Polyfluorinated Alkyl Substances (PFAS) Analysis Using Isotope Dilution by LC/MS/MS*.



FIELD SAMPLING PLAN – PFAS SAMPLING

Oconomowoc Electroplating Co., Inc., Ashippun, WI

December 05, 2022; Page 8 of 14

Sampling technicians will wear disposable, powderless, nitrile gloves while handling decontaminated sampling equipment. Sampling personnel will change gloves when a breach in the glove is noticed and between wells, to prevent cross contamination.

The only non-dedicated piece of equipment that will come into contact with groundwater within the monitoring well, the water-level meter, will be decontaminated using the following procedure:

1. Clean with PFAS-free potable water and detergent (i.e. Alconox®) using a stiff brush to remove visible foreign materials;
2. Rinse three (3) times with PFAS-free distilled water;
3. Allow to air dry; and
4. Place in or wrap down-hole equipment with poly bags when not in use.

In addition to collecting one groundwater sample from each well, one duplicate groundwater sample (10% of wells or a minimum of one), one equipment blank sample (one per sampling event), and one trip blank (one for each sample cooler) will be collected for Quality Assurance/Quality Control. The trip blank samples will be of water matrix.

Pace Analytical Services uses control procedures to assess the validity of the analytical results. Determination of the validity of sample results is based on adherence to laboratory quality control procedures as described in Pace Analytical's Quality Manual, which is provided in Appendix B of Hyde's QAPP. The acceptance criteria are method-specific requirements. Pace Analytical Services will conduct matrix spike/matrix spike duplicate (MS/MSD) analysis on duplicate and field blank samples provided by Hyde. The control samples will be analyzed in the same manner as the field samples.

At a minimum, the following information will be placed on each sample container:

1. Sample name/location
2. Sample number
3. Name of collector
4. Date and time of sampling
5. Site location
6. Analysis to be performed, and
7. Preservative

Sample naming conventions will follow existing OECI Site location names.

Chain-of-custody documents will be completed and maintained for the sample containers from the time that the samples are collected until they are delivered to the lab. Tracking of receipt, storage, and handling will be documented and sample containers will be kept sealed and safeguarded while in the custody of sampling crews. The chain-of-custody forms will be



completed and placed in the respective shipping container, which then will be sealed and delivered to the courier service or vehicle that will transport them to the laboratory.

Two (2) seals, comprised of chain-of-custody tape, will be placed over the lid on the front and back of each shipping cooler, prior to shipment, to secure the lid and provide evidence that the samples have not been tampered with enroute to the laboratory. Clear tape will be placed over the seals, to ensure that they are not accidentally broken during shipment. Upon receipt of the cooler at the laboratory, the designated sample custodian will inspect the cooler. On the chain-of-custody form, the sample custodian will document the condition of the cooler and seal. The sample custodian will also document the date and time of receipt of the cooler, and sample receipt temperature.

The sample custodian will then check the contents of the cooler with those samples listed on the chain-of-custody form. If damage or discrepancies are noticed, they will be recorded in the “Remarks” column of the chain-of-custody form. The sample custodian then will date and sign the chain-of-custody form. Damage or discrepancies will be reported to the laboratory supervisor, who will inform the laboratory manager and quality assurance officer. The laboratory will notify the Hyde QA Manager, who will then notify the WDNR Project Manager.

5.0 SPECIAL CONSIDERATIONS WHEN SAMPLING FOR PFAS

This section discusses the processes and acceptable items and materials that should be used when sampling groundwater monitoring wells for PFAS. Many materials used in the course of environmental investigation can potentially contain PFAS. Obtain and review all Safety Data Sheets (SDSs) before considering materials for use during PFAS sampling. If PFAS are listed on the SDS, it is recommended that piece of equipment/supply not be utilized.

Potential sources for PFAS cross-contamination include items and materials used during sampling activities, such as sampling equipment, field clothing, personal protective equipment (PPE), sun and biological protection products, personal hygiene, personal care products (PCPs), and food packaging. Items to avoid include materials that contain fluoropolymers such as polytetrafluoroethylene (PTFE), that includes the trademarks Teflon® and Hostafion®, polyvinylidene fluoride (PVDF), that includes the trademark Kynar®, polychlorotrifluoroethylene (PCTFE), that includes the trademark Neoflon®, ethylene-tetrafluoro-ethylene (ETFE), that includes the trademark Tefzel®, and fluorinated ethylene propylene (FEP), that includes the trademarks Teflon® FEP and Hostafion® FEP.

Additional items to avoid during sampling include waterproof, water-resistant, or stain-resistant clothing/products, certain cosmetics and lotions, certain insect repellants and sunscreens, water or oil-resistant paper (e.g. fast food wrappers), latex gloves, plastic clipboards, binders, or hardcover spiral books, Post-it® notes, recycled paper products



(e.g. paper towels and notebook paper), chemical (blue) ice packs, disposable glass pipettes, aluminum foil, and Kim® wipes

The following considerations should be taken during sample collection to prevent contamination: keep dust and fibers out of sample bottles; sample cap should never be placed directly on the ground during sampling; do not sample without powderless nitrile gloves; regular/thick size markers (Sharpie® or otherwise) should be avoided (fine and ultra-fine point Sharpie® markers are acceptable to label the empty sample bottle while in the staging area provided the lid is on the sample bottle and gloves are changed following sample bottle labeling); preprinted labels from the laboratory may be used. Additional information, *U.S. EPA Region 5 Analytical Services Branch PFAS Sampling Fact Sheet* and *PFAS-Specific Sampling Precautions, Procedures, and Requirements* document, are included in **Attachment B**.

6.0 MANAGEMENT OF PURGE AND DECONTAMINATION FLUIDS

Purge water (water removed from the monitoring wells prior to and during sample collections) and decontamination fluids (fluids used to clean equipment), will be managed at the Site. Water removed from private wells will be discharged to the ground.

On-Site handling of investigation-derived wastes (IDW) at this Site is consistent with U.S. EPA's policies on management of IDW (U.S. EPA, 2020). The IDW will be collected in a five-gallon container(s) and transferred to a 55-gallon steel drum(s). At the completion of sampling, the water stored in the drum(s) will be sampled for disposal profiling purposes and ultimately transported off-site for disposal at a licensed landfill by a licensed waste hauler.

Personal protective equipment and decontamination equipment will be disposed as solid waste.



7.0 PERFORMANCE STANDARDS

The performance standards associated with collecting representative groundwater data include:

- Collection, storage, and delivery of groundwater samples, in accordance with WDNR and U.S. EPA guidelines and policies; and
- Analysis of groundwater samples at a laboratory specifically approved by WDNR for this project, and in accordance with WDNR guidelines and policies.

8.0 SCHEDULE

The starting point of the schedule, included as **Figure 3**, is based on the date of approval of the QAPP and this *Field Sampling Plan* by the U.S. EPA and WDNR.

9.0 DELIVERABLES

Hyde will submit a single digital copy of the final *PFAS Groundwater Monitoring Report* to WDNR through the submittal portal. The report will contain the following certification statement to be signed by the Project Manager from Hyde:

“To the best of my knowledge, after thorough investigation, I certify that the information contained in or accompanying this submission is true, accurate and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine or imprisonment for knowing violations.”



10.0 REFERENCES

Pace Analytical Services, *ENV-MAN-MIN4-0001, Pace Analytical, Quality Manual*

U.S. EPA low-flow groundwater sampling techniques; *U.S. EPA Low Stress (Low Flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells, September 19, 2017*

U.S. EPA Region 4 *Management of Investigated Derived Waste, May 8, 2020*

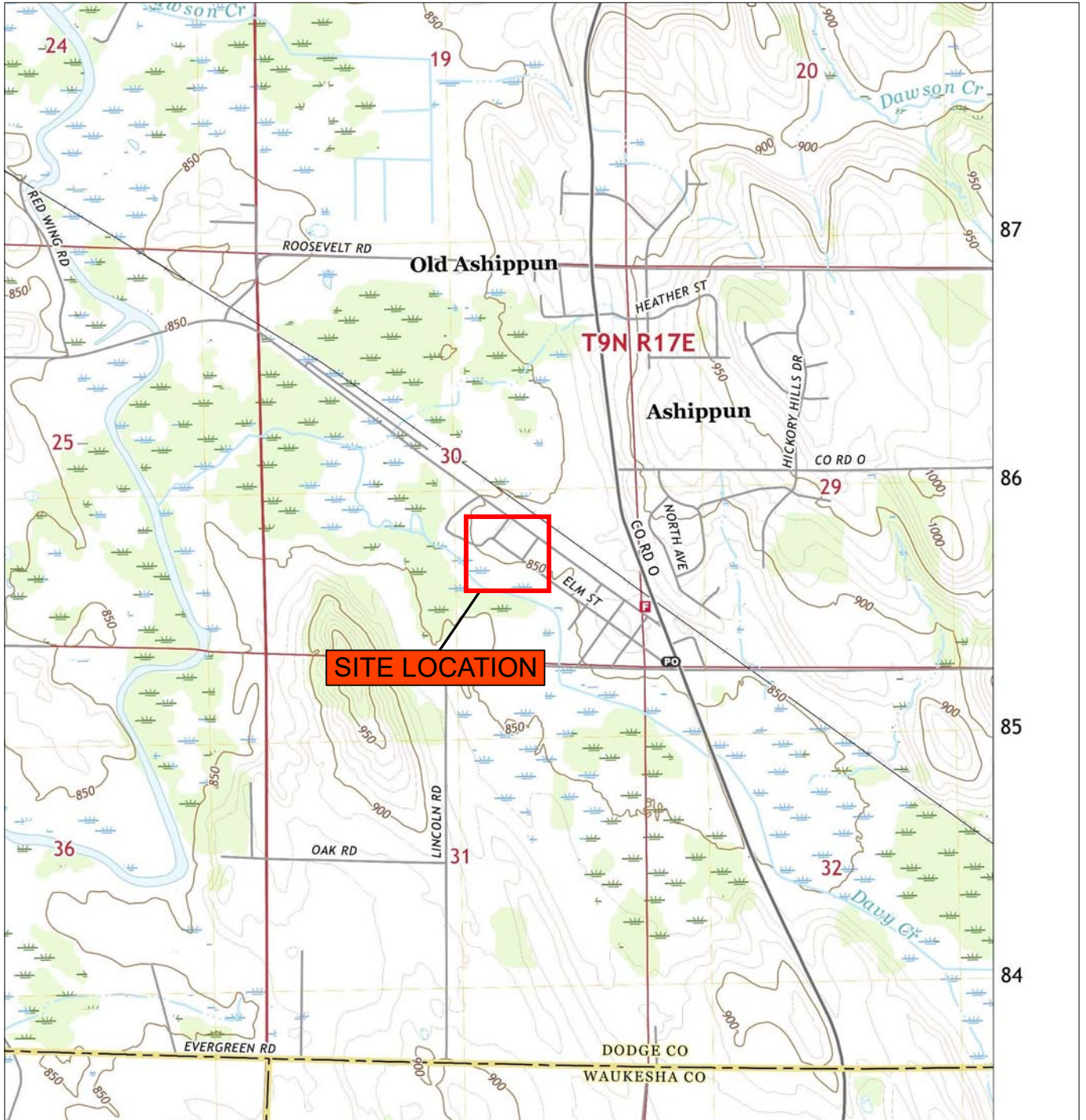
WDNR, Request for Bid, PFAS Groundwater Investigation, *Oconomowoc Electroplating Company, Inc. (OEI) Superfund Site, November 2021*

WDNR, *Wisconsin PFAS Aqueous (Non-Potable Water) and Non-Aqueous Matrices Method Expectations, Version 12.16.2019, Per- and Polyfluorinated Alkyl Substances (PFAS) Analysis Using Isotope Dilution by LC/MS/MS.*

WDNR, *Annual Groundwater Monitoring Report, November-December 2021 Sampling Events. Oconomowoc Electroplating Company, Inc. Superfund Site, Town of Ashippun, Wisconsin, Hyde Environmental, Inc., June 28, 2022.*



FIGURES



Base map from U.S.G.S. 7.5' IXONIA, 2022, WISCONSIN topographic quadrangle map.

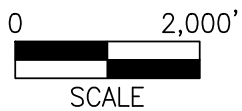
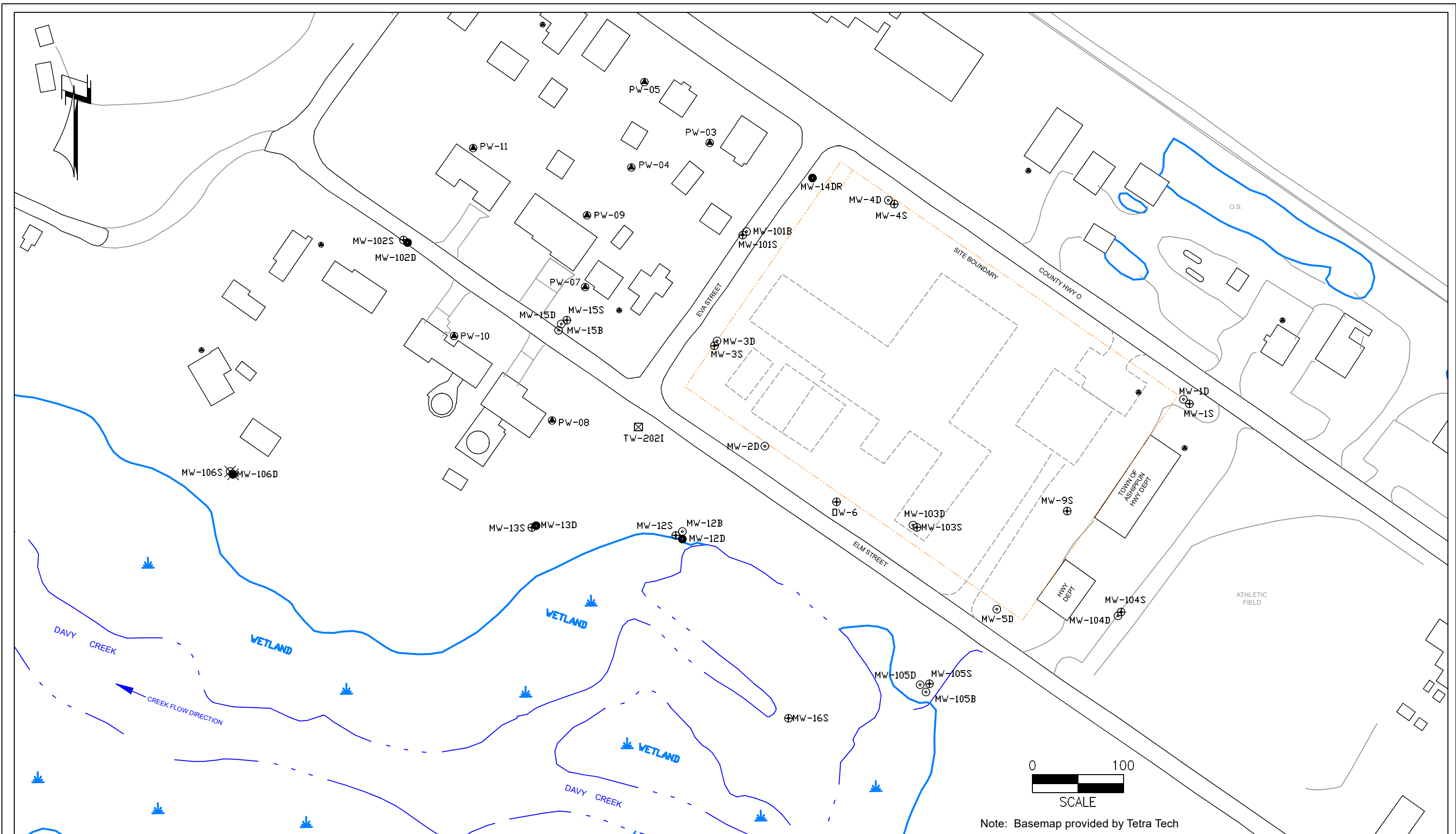


Figure 1
SITE LOCATION MAP
 Oconomowoc Electroplating Company, Inc.
 Ashippun, WI



- | | | | |
|-----------|--|-----------|--------------------------------------|
| ⊙ MW-105B | BEDROCK MONITORING WELL | ● PW-11 | RESIDENTIAL WELL |
| ● MW-105D | DEEP UNCONSOLIDATED MONITORING WELL | ⊙ MW-106D | DEEP UNCONSOLIDATED SENTINEL WELL |
| ⊕ MW-105S | SHALLOW UNCONSOLIDATED MONITORING WELL | ⊙ MW-106S | SHALLOW UNCONSOLIDATED SENTINEL WELL |



Figure 2
SAMPLE LOCATION MAP
 Oconomowoc Electroplating Company, Inc.
 Ashippun, WI



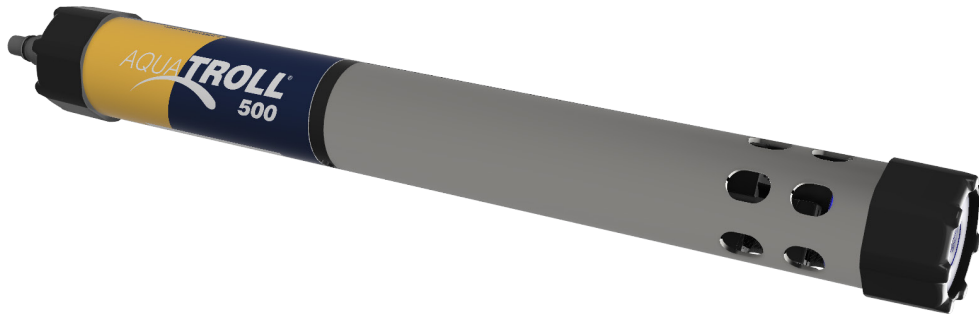
ATTACHMENT A

Aqua-Troll 500™ Operations Manual

and

Heron Dipper T™ Operations Manual

Operator's Manual



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Instrument Overview

Serial Number Location

The instrument serial number is on the product label affixed to the instrument body. Serial numbers for individual sensors are engraved on the sensor body.

Unpacking and Inspection

Your equipment was carefully inspected before shipping. Check the equipment for any physical damage sustained during shipment. Notify In-Situ and file a claim with the carrier if there is any such damage; do not attempt to deploy or operate the instrument.

! Save packing materials for future storage and shipping of your equipment.

Obtaining Repair Service

If you suspect your system is malfunctioning and repair is needed, you can help assure efficient servicing by following these guidelines:

1. Call or email In-Situ Technical Support. Have the product model and serial number available.
2. Be prepared to describe the problem, including how the product was used and the conditions noted at the time of the malfunction.
3. If Technical Support determines that service is needed, they will ask your company to fill out the RMA form and pre-approve a specified monetary amount for repair charges. When the form and pre-approval is received,
4. Technical Support will assign an RMA (Return Material Authorization) number.
5. Clean the product as described in the manual.
6. If the product contains a removable battery, remove and retain it unless you are returning the system for a refund or Technical Support states otherwise.
7. Carefully pack your product in its original shipping box, if possible.
8. Mark the RMA number clearly on the outside of the box.
9. Send the package, shipping prepaid, to:

In-Situ
ATTN: Repairs
221 East Lincoln Avenue
Fort Collins, CO 80524

The warranty does not cover damage during transit. In-Situ recommends insurance for all shipments. Warranty repairs will be shipped back prepaid.

Outside the U.S.

Contact your international In-Situ distributor for repair and service information.

Guidelines for Cleaning Returned Equipment

Please help us protect the health and safety of our employees by cleaning and decontaminating equipment that has been subjected to potential biological or health hazards, and labeling such equipment. Unfortunately, we cannot service your equipment without such notification. Please complete and sign the form on page 12 (or a similar statement certifying that the equipment has been cleaned and decontaminated) and send it to us with each instrument.

- We recommend the glassware cleaning product, Alconox, available from In-Situ and from laboratory supply companies.
- Clean all cables and remove all foreign matter.
- Clean the cable connectors with a clean, dry cloth. Do not submerge the connectors.
- Clean the instrument including the nosecone, cable head, and protective caps.

I If an instrument is returned to our Service Center for repair or recalibration without a statement that it has been cleaned and decontaminated, or if it is the opinion of our Service Representatives that the equipment presents a potential health or biological hazard, we reserve the right to withhold service until proper certification is obtained.

Decontamination & Cleaning Statement

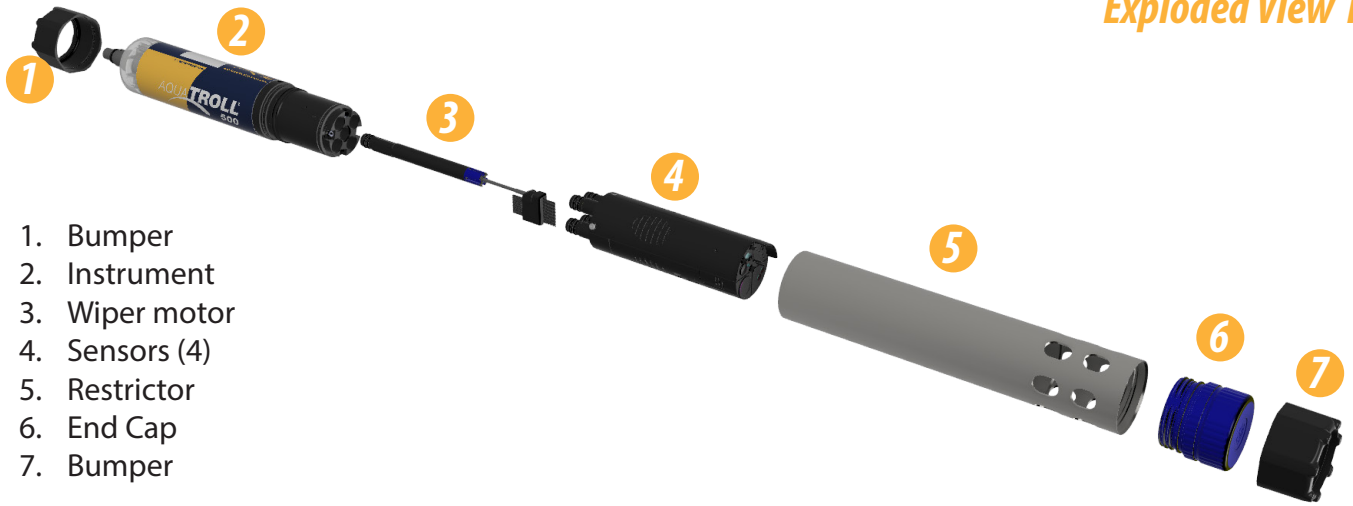
Company Name	Phone
Address	
City	State
Instrument Type	Serial Number
Contaminant(s) if known)	
Decontamination procedure(s) used	
Cleaning verified by	Title
Date	

Safety

- Do not submerge the Wireless TROLL Com or your mobile device in liquid.
- Ensure that sensors, or sensor plugs, are completely inserted into the ports, so that no liquid can enter the instrument.
- Ensure that the RDO Sensor Cap is pressed firmly over the sensor lens and is flush with the instrument before submerging in liquid.
- Replace the cable if insulation or connectors are damaged.
- Make sure the probe and sensor O-rings are clean and free of damage.

Instrument Components

Exploded View 1

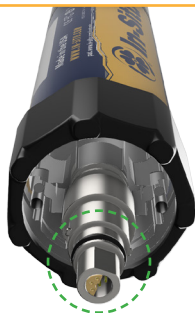


Exploded View 2



End View

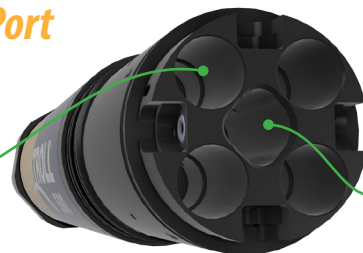
Flat edge of connector aligns with flat edge of Rugged Cable.



Sensor Port Detail

Sensor ports

Wiper motor port



Required Accessories

Communication Device

You will need a communication device to calibrate, configure and deploy the Aqua TROLL 500.



Wireless TROLL Com

Provides power to the Aqua TROLL 500.

Configure and deploy with a Bluetooth-enabled Android device.

Connects the Aqua TROLL 500 to a PC via USB or Bluetooth.

Cable



Rugged Twist-Lock Cable

Connects the Aqua TROLL 500 to a Wireless TROLL Com, USB TROLL Com or Cube/Tube.

Vented or non-vented.

Sensors



Available Sensors

1. Temperature
2. Conductivity/temperature
3. pH/ORP
4. RDO
5. Turbidity
6. Ammonium
7. Chloride
8. Nitrate

Software



Win-Situ 5 Software for PC

Calibrate, configure and take readings with the Aqua TROLL 500 from a PC.



VuSitu Mobile App

Calibrate, configure and deploy the Aqua TROLL 500 from a Bluetooth-enabled Android device.

Download it from www.in-situ.com.

Get it at play.google.com.

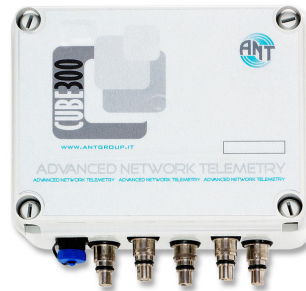
Telemetry



Tube 300

Power Aqua TROLL 500 in remote-monitoring applications

Send data to HydroVu or another FTP server.



Cube 300

Power up to five instruments in remote-monitoring applications

Send data to HydroVu or another FTP server.

* Cubes and Tubes are available in battery or solar-powered options.

Instrument Dimensions



LCD Screen

! View instrument status and access settings via the LCD screen. The sonde must be connected to a Wireless TROLL Com or other power source.

Accessing the LCD Menu



LCD screen will display sensor status on activation.



Hold instrument horizontally and slowly tap Aqua TROLL 500 logo 3-4 times to view the main menu.



Tilt instrument left or right to scroll through menu options.



Select an item when its background turns black by tapping the instrument once.



You can enable Bluetooth communication directly with the sonde via the Bluetooth menu option.

Possible Port Statuses



Sensors installed



Port plugs installed



Sensor/port error

Possible Power Statuses



Power level within specs



Power level NOT within specs

Possible Connected Statuses



Connected via Bluetooth



Connected via cable

Error Messages



Install All Sensors!

Port(s) empty



RDO Cap expired!

Cap expiration

System Components

Base Unit Components

	Part Number
RDO Sensor	0063450
Combination pH/ORP Sensor	0063470
Turbidity	0063480
Combination Conductivity/Temperature Sensor or standalone Temperature Sensor	0063460, 0063490
Ammonium Sensor	0033700
Chloride Sensor	0033720
Nitrate Sensor	0033710
Dual Stainless Titanium Storage Chamber	0079880
Sensor Port Plug	0063510
Rubber Bumpers (2)	0079880
Wiper or Wiper Port Plug	0063500, 0064630

Accessories purchased separately

Wireless TROLL Com for Android	0031240
Rugged Android Tablet	0064860

Cable

Stripped-and-tinned Cable with male connector	0053310
Twist-Lock Bulkhead Connector	0053240
Cable Extender	0051490
Large Desiccant (titanium connector)	0051810
Large Desiccant (ABS connector)	0053550
Small Desiccant (3 pack) - storage desiccant	0052230
Desiccant Refill Kit for Large or Outboard Desiccant	0029140

Calibration and Maintenance

RDO X Cap Replacement Kit	0079790
RDO Fast Cap	0066800
pH/ORP & ISE Replacement Reference Junction Kit	0078990
Wiper Brush Kit	0079810
Maintenance Kit	0078940
Copper Antifouling Guard	0076100
Quick-Cal Solution for calibrating DO , Conductivity, pH and ORP	0033250
Dissolved Oxygen Calibration Kit	0032110
DO Field Calibration Kit	0080830
Conductivity Calibration Kit (Full)	0032090
Conductivity Calibration Kit (Low)	0032630
Conductivity Calibration Kit (High)	0032640
pH Calibration Kit	0032080
pH/ORP Calibration Kit	0032120
pH & ISE Storage Solution	0065370
Individual Calibration Solutions	See website
Ammonium Calibration Kit (includes 1 liter each: 14 ppm, 140 ppm, 1400 ppm, DI water)	0032140
Chloride Calibration Kit (includes 1 liter each: 35.5ppm, 355 ppm, 3545 ppm, DI water)	0032150
Nitrate Calibration Kit (includes 1 liter each: 14 ppm, 140 ppm, 1400 ppm, DI water)	0032130

Spot Checking Configuration

Take live readings with an Aqua TROLL 500, Rugged Cable, Wireless TROLL Com and a Bluetooth-enabled Android device.



Wireless TROLL Com

Powers the Aqua TROLL 500 during live readings.

Communicates with the VuSitu mobile app via Bluetooth.

Rugged Cable

Connects the Wireless TROLL Com to the instrument.

Aqua TROLL 500

Takes live readings and transfers data to the Wireless TROLL Com via the Rugged Cable.

Getting Started (Spot-Checking)

Follow the steps below to set up and deploy the Aqua TROLL 500 when you intend to take live readings. See the next page for information about setting up and using the instrument in remote-monitoring applications.

1 **Unpack instrument.**

Remove sonde, sensors and maintenance supplies from box.

2 **Install RDO cap and pH/ORP sensor.**

- a. If your instrument includes a pH/ORP sensor, you'll need to install it prior to calibration and deployment.
- b. Install the RDO cap on the RDO sensor.

3 **Download and install software.**

- PC users visit www.in-situ.com
- Mobile device users: play.google.com

4 **Connect instrument to TROLL Com.**

- a. Attach the Rugged Cable to the TROLL Com and Aqua TROLL 500.
- b. Press power button on TROLL Com and pair with the VuSitu mobile app.

5 **Calibrate.**

Perform a single or multi-point calibration.

6 **Configure the instrument and take readings.**

- a. Create a site in VuSitu.
- b. Take readings in VuSitu's Snapshot or Live Readings mode.
- c. Save readings and share via email, SMS or cloud storage.

Installing Wiper Motor and Sensors



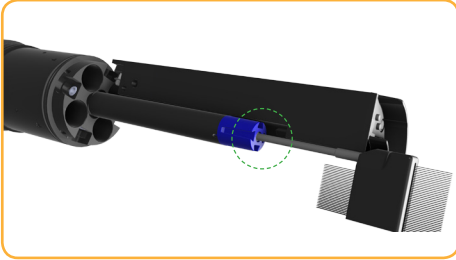
Remove restrictor.



Install wiper motor.



Install sensors in any order.



Align sensor with interlock groove in wiper motor.



Tighten set-screw at base of each sensor.



Unscrew end cap from restrictor.



Flip restrictor and install with restrictor holes near center of instrument for calibration.



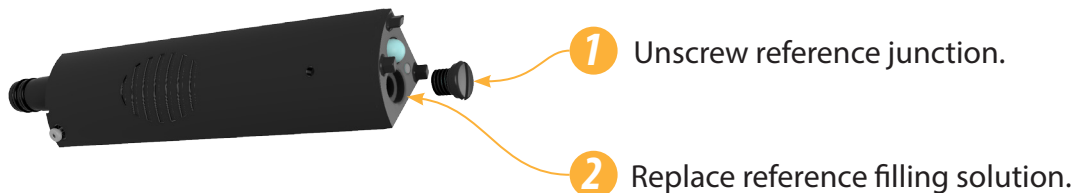
Flip restrictor and replace end cap before deployment.

Handling pH and Ion-Selective Electrode Sensors (ISEs)

- ! Salt may accumulate around the reference junctions of the ammonium, chloride, nitrate and pH sensors. Rinse with deionized water to remove any buildup.

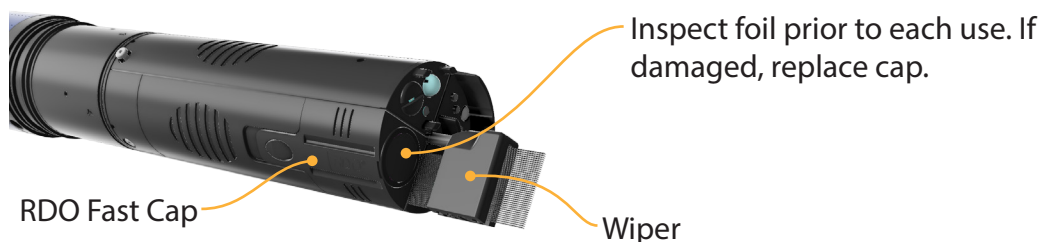


- ! Before using the pH or ISE sensors for the first time, replace the reference filling solution. Condition the sensors by soaking in calibration standard for 4-24 hours prior to deployment.



Using the RDO Sensor and RDO Fast Cap

- ! The wiper can severely reduce the life of the RDO Fast Cap. Wear will vary by application. Verify sensor performance prior to use and replace the Fast Cap if damaged.



Connecting the TROLL Com

Wireless TROLL Com

- ! You must connect the Aqua TROLL 500 to a Wireless TROLL Com to calibrate the instrument, configure or take live readings.



Attach Rugged Cable to the Wireless TROLL Com.



Attach opposite end of cable to the Aqua TROLL 500.



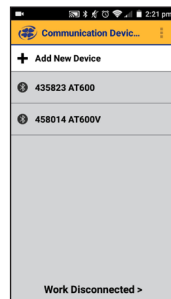
Press power button.

Pairing the Instrument with the VuSitu Mobile App

- ! Download and install the VuSitu mobile app from the Google Play store. Visit play.google.com on your Android device.



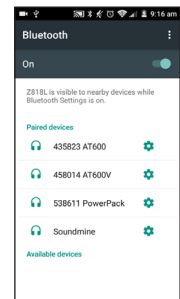
Turn on the Wireless TROLL Com and open VuSitu mobile app.



Select **Add New Device** when connecting for the first time.

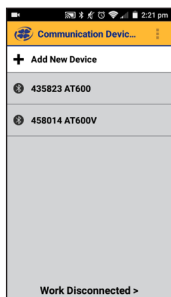


Locate the serial number under the yellow lid on the Wireless TROLL Com.

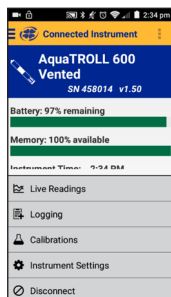


From device's Bluetooth Settings screen, tap serial number of Wireless TROLL COM.

Select **Choose or Add a Device**.



Tap mobile device's back button and tap serial number from list.



Tap mobile device's **Back** button to view Connected Instrument screen.

Navigating VuSitu

! After pairing a Wireless TROLL Com with VuSitu, the app will always display the Connected Instrument screen at launch. You can access all features of the app from this screen.

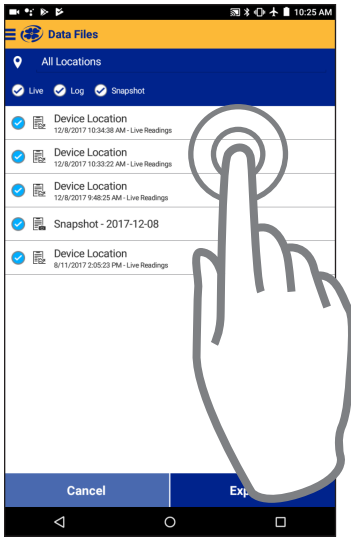
Connected Instrument Screen

The screenshot shows the 'Connected Instrument' screen for an Aqua TROLL 500. The top status bar shows the time as 4:27 PM. The app title is 'Connected Instrument'. Below the title, the instrument name 'Aqua TROLL 500' is displayed, along with its serial number 'SN 50002' and version 'v0.13'. The battery level is shown as 84% remaining. The instrument time is 4:27 PM on 1/15/2018. At the bottom, there is a menu with four options: 'Live Readings', 'Calibrations', 'Instrument Settings', and 'Disconnect'. Callout boxes provide the following descriptions for these options:

- Access menu.** (points to the hamburger menu icon)
- Access help information.** (points to the three-dot menu icon)
- Take single readings or continuously record at two-second intervals.** (points to 'Live Readings')
- Calibrate sensors.** (points to 'Calibrations')
- Access instrument clock and telemetry settings.** (points to 'Instrument Settings')
- Disconnect app from instrument.** (points to 'Disconnect')

Selecting with Long-press and Swipe

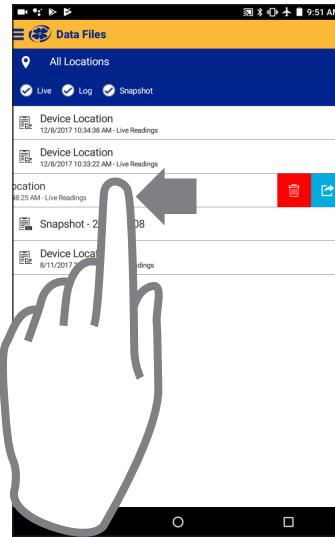
Long-Press



Press and hold any of the items in a list of files.

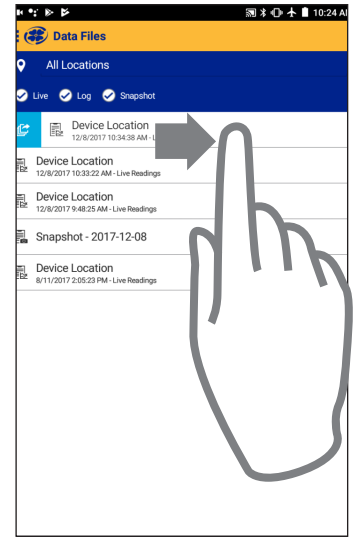
You can now select two or more items.

Swipe Left



Press an item and swipe left to reveal the delete and sharing icons.

Swipe Right



Press any item in a list and swipe right to reveal the sharing icon.

Calibrating the Sensors

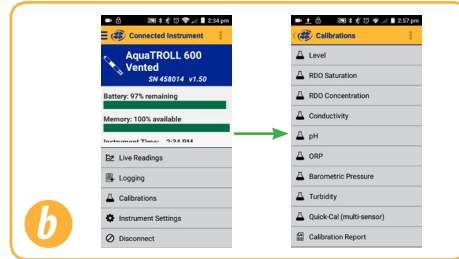
Solution-based calibration

Use the solution-based procedure described below to calibrate all sensors except RDO. You will need the following items.

- Calibration standard, or multiple standards for multi-point calibrations
- Wireless TROLL Com connected to the Aqua TROLL 500
- Bluetooth-enabled Android device



Connect the sonde to a Wireless TROLL Com and pair with VuSitu.



In VuSitu, click Calibrations from the Connected Instrument screen and choose sensor to calibrate.



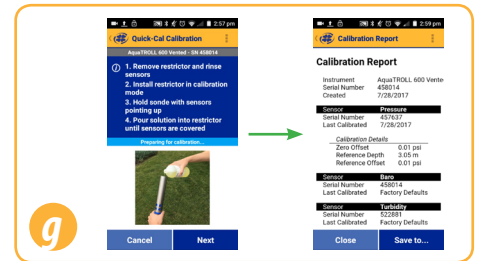
Remove cap from instrument and pour 10–20 ml of DI water into restrictor.



Gently shake the sonde in a circular motion to rinse the inside of restrictor and sensors.



Discard the DI water and repeat rinsing procedure two more times with 10–20 ml of your first calibration standard.



Follow the instructions in VuSitu to perform the calibration.

RDO 100% Saturation Calibration: Water Saturated Air

Use the procedure below to calibrate the Aqua TROLL 500 RDO sensor, or see the next section for an alternative method.



Place the restrictor in calibration mode (holes near center of instrument).



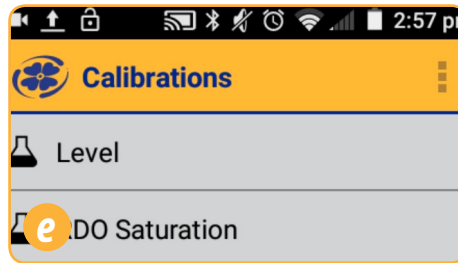
Saturate a small sponge with water.



Place sponge in restrictor.

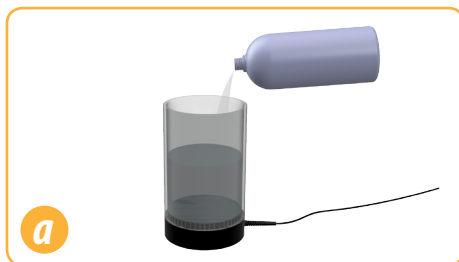


Reinstall the end cap and leave sponge in restrictor for five minutes.



Follow the instructions in VuSitu to finish calibration.

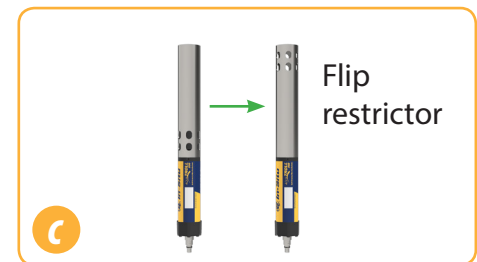
RDO 100% Saturation Calibration: Saturation Bubbler



Fill a 100% saturation bubbler two-thirds with tap water.



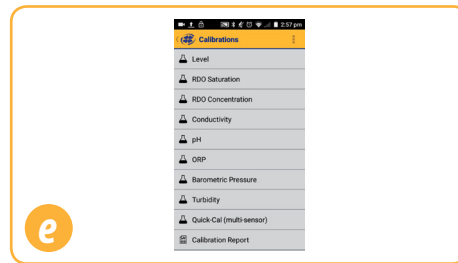
Turn on bubbler and allow 5-10 minutes for 100% saturation.



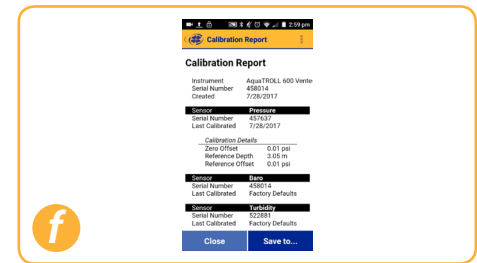
Put sonde into deployment mode by flipping restrictor 180 degrees.



Place sonde into bubbler.



Open the VuSitu mobile app and tap Calibrations > RDO Saturation.

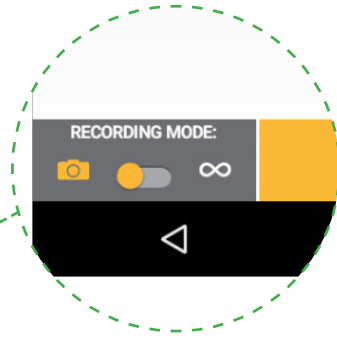
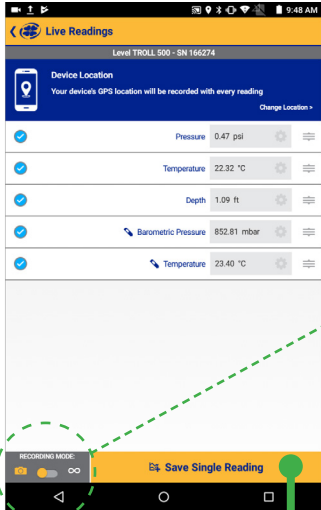


Follow instructions in VuSitu to finish calibration.

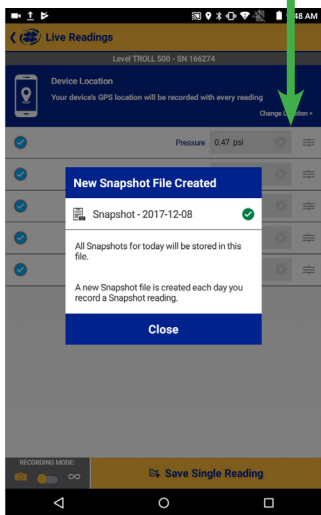
Live Readings in VuSitu

! To take live readings with the Aqua TROLL 500 and VuSitu mobile app, the sonde must be connected to a Wireless TROLL Com.

Snapshot Mode

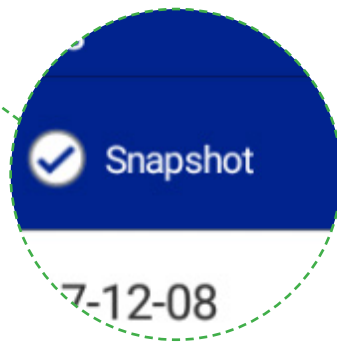
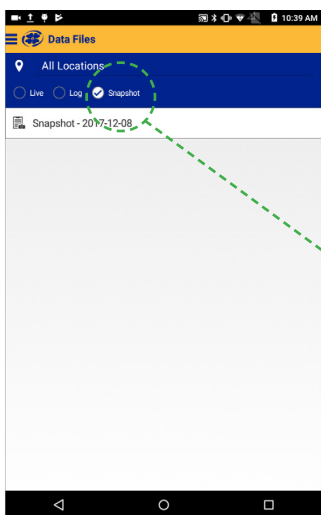


Take a single reading and save to Snapshot file.

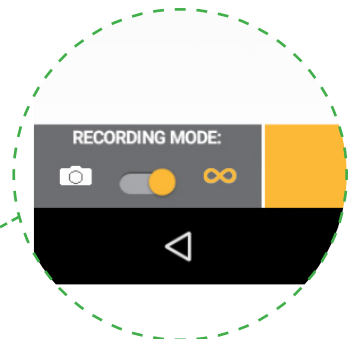
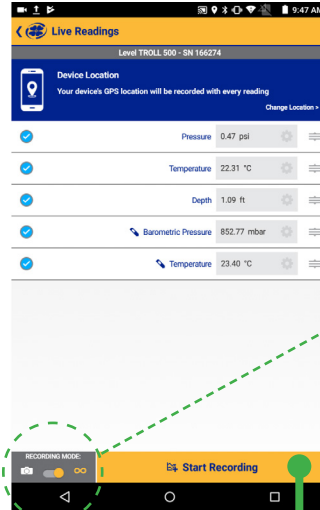


View Snapshot file from Menu > Data Files.

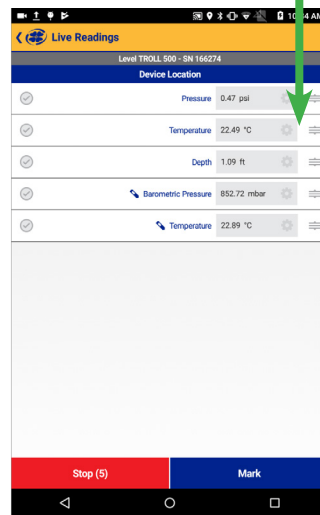
Check Snapshot option.



Live Readings Mode

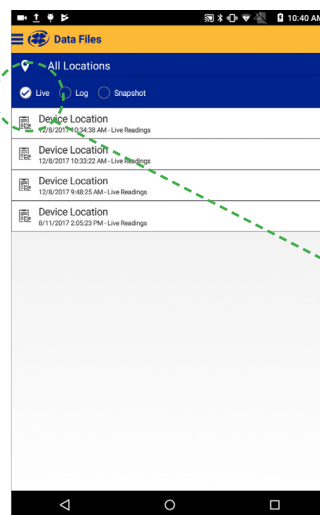


Take readings at two-second intervals.

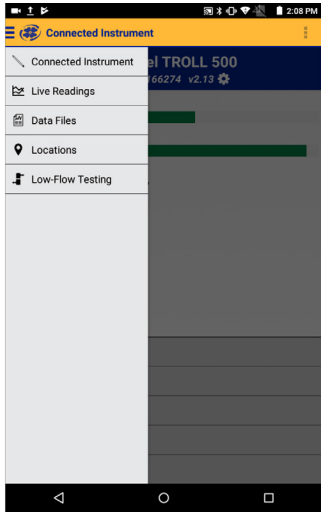


View file from Menu > Data Files.

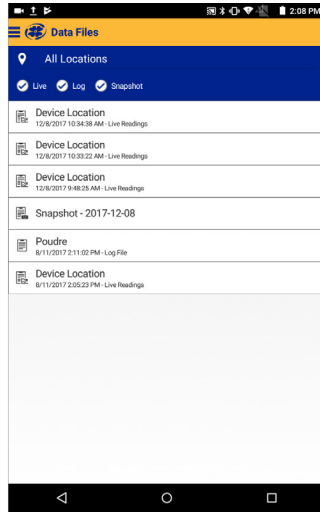
Check Live option.



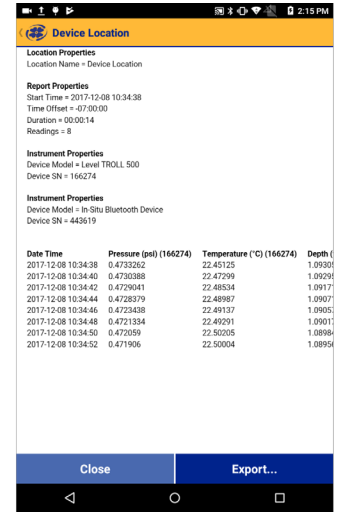
Exporting Data Files



Use the menu at the top left to access the Data Files screen.

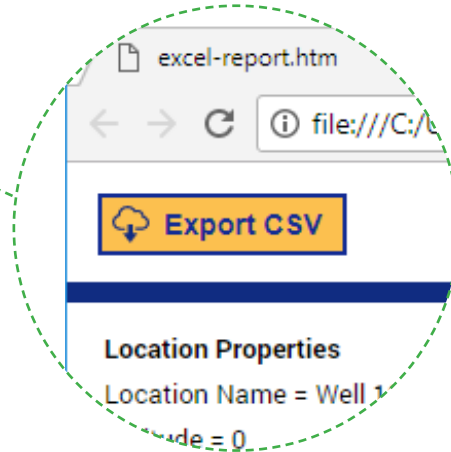
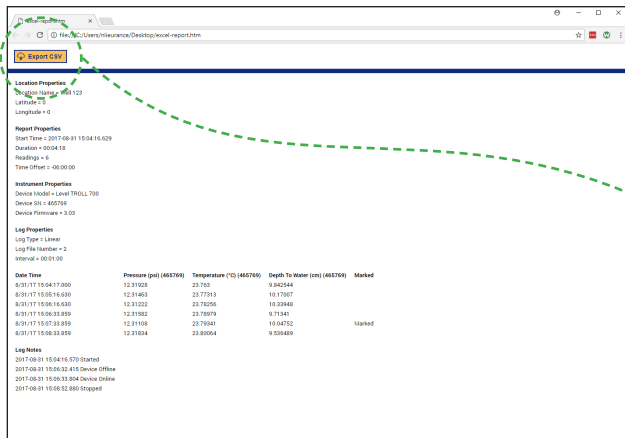


Tap one of the files to view and export.

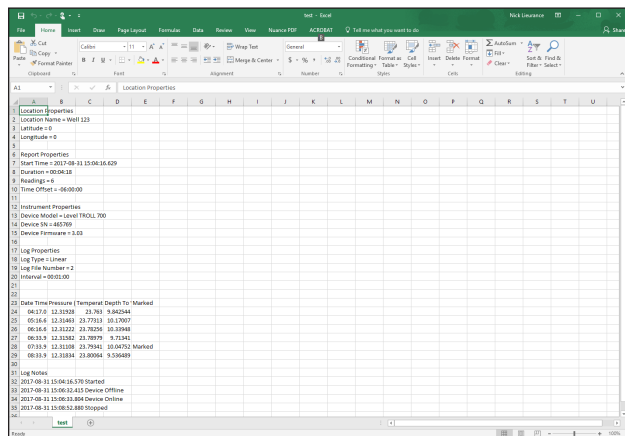


Tap **Export** to save the file and choose how you wish to share it.

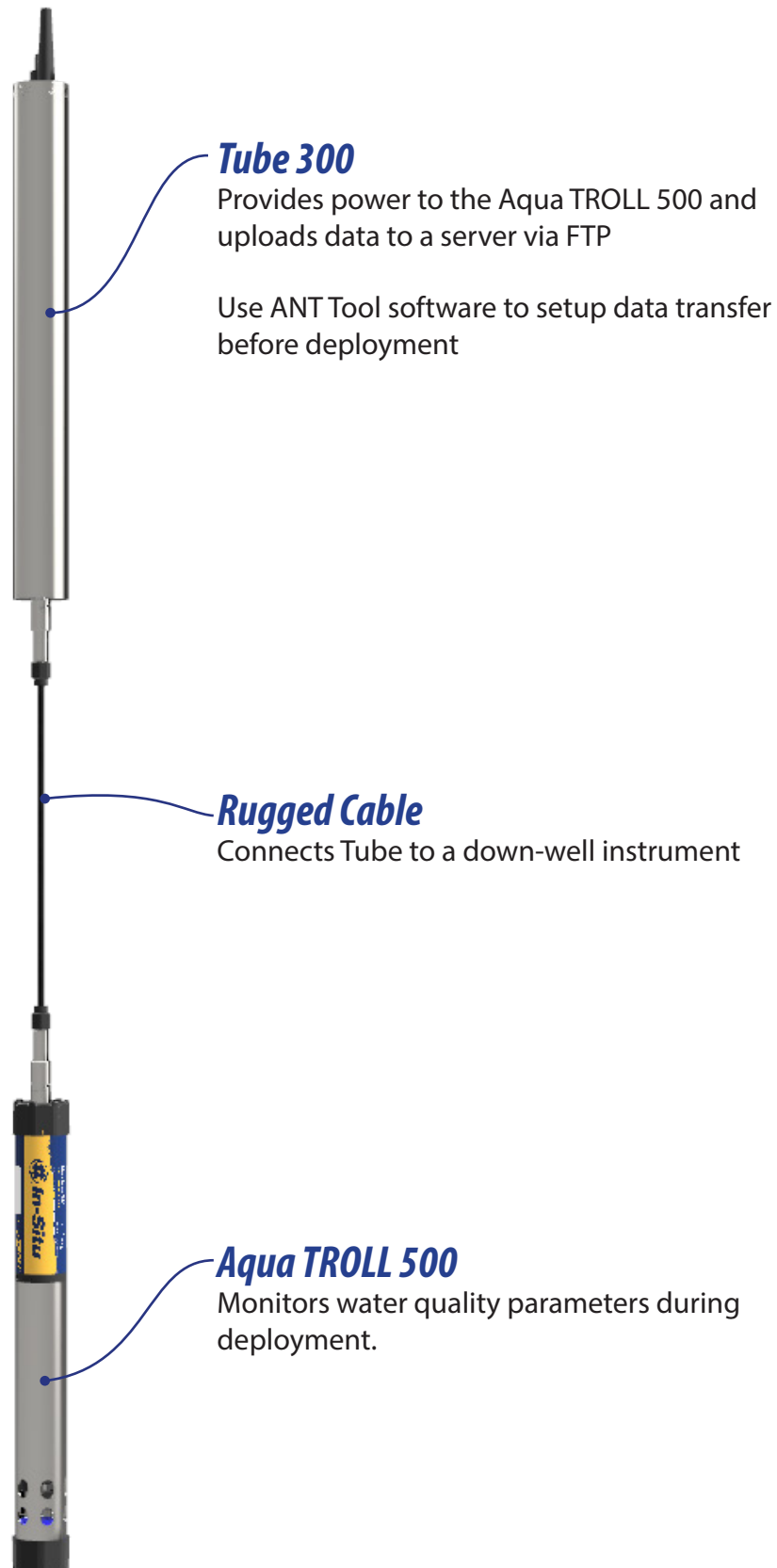
Viewing Data Files



Open a VuSitu data file in any web browser. Click the button at the top left to generate a CSV.



Remote-Monitoring Configuration (Telemetry)



Getting Started (Remote Monitoring)

1 **Unpack instrument.**

Remove sonde, sensors and maintenance supplies from box.

2 **Install RDO cap and pH/ORP sensor.**

- a. If your instrument includes a pH/ORP sensor, you'll need to install it prior to calibration and deployment.
- b. Install the RDO cap on the RDO sensor.

3 **Download and install software.**

- PC users visit www.in-situ.com to download Win-Situ 5 and the ANT Tube/Cube Tool
- Mobile device users: play.google.com

4 **Connect instrument to TROLL Com.**

- a. Connect the Aqua TROLL 500 to a Wireless TROLL Com with a Rugged Cable.
- b. Press the power button on the Wireless TROLL Com.

5 **Calibrate.**

Perform a single or multi-point calibration.

6 **Configure.**

See instructions on the next page to configure the telemetry device.

7 **Configure the Tube/Cube.**

Use the ANT Cube/Tube tool to set alarms and FTP information.

8 **Deploy.**

Place the instrument in the deployment location.

Configuring the Cube/Tube

! Before deploying the Aqua TROLL 500 in a remote-monitoring application, configure the Tube/Cube and the sonde.

1 **Download and install the ANT Tool.**

Visit www.in-situ.com/software and download the ANT Tube/Cube Tool.

2 **Connect the Aqua TROLL 500 to your PC.**

Connect the Cube/Tube to a PC with the setup cable.

3 **Configure Cube/Tube options.**

Use the ANT Tool to configure alarms and FTP information.

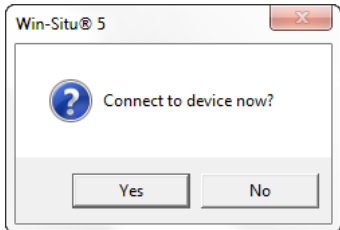
4 **Deploy the instrument.**

Connect Cube/Tube to the instrument with a Rugged Cable.
Place the tube and instrument in the deployment location.

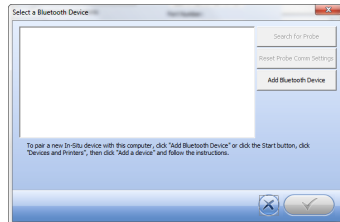
Connecting to Win-Situ on a PC

Connecting to Win-Situ via Bluetooth

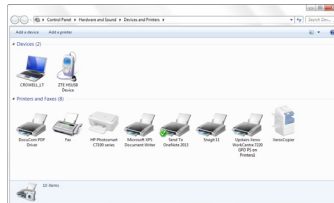
- ! Download and install Win-Situ by visiting www.in-situ.com. The Wireless TROLL Com must be connected to the sonde and powered on to connect the instrument to Win-Situ.



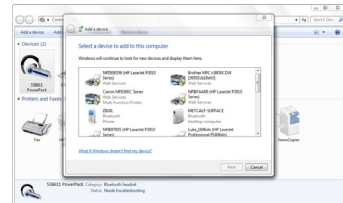
Open Win-Situ. Select **Yes** when asked to connect now.



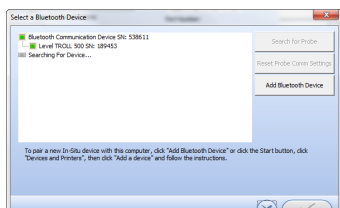
Click **Add Bluetooth Device** button.



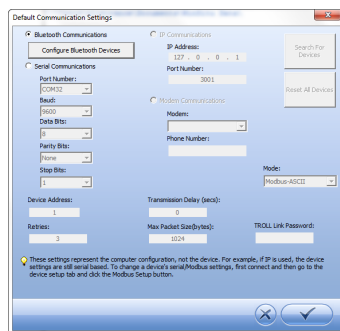
From Devices and Printers window, click **Add a device**.



Select your Wireless TROLL Com (PowerPack) from the list of Bluetooth-enabled devices. Click **Next**.



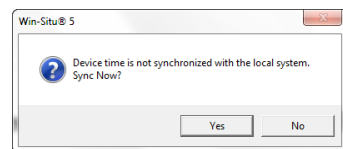
Make sure device is highlighted in Select a Bluetooth Device window and click check mark button.



Click check mark button on Default Communication Settings window to close.



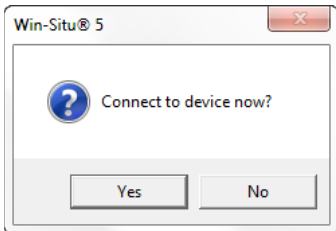
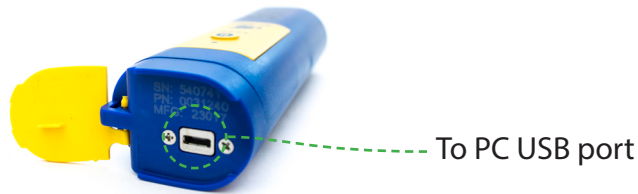
Click the yellow connect button at the bottom right of the screen.



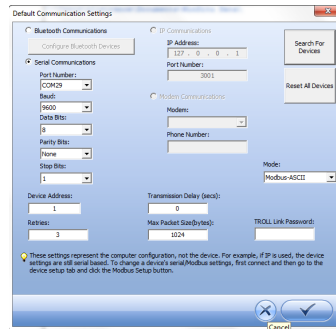
Click Yes if prompted to sync device time with local system.

Connecting to Win-Situ via USB

- ! You can connect a Wireless TROLL Com to a PC with the included USB cable. Plug the cable into the port at the top of the TROLL Com and the USB port on your PC.



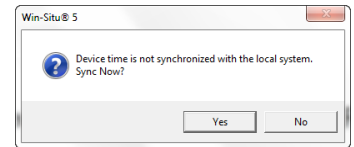
Open Win-Situ. Select **No** when asked to connect now.



Click Preferences > Com Settings from menu bar and choose correct com port. Select Serial Communications button. Click check mark button.



Click the yellow connect button at the bottom right of the screen.



Click Yes if prompted to sync device time with local system.

Navigating the Win-Situ Interface

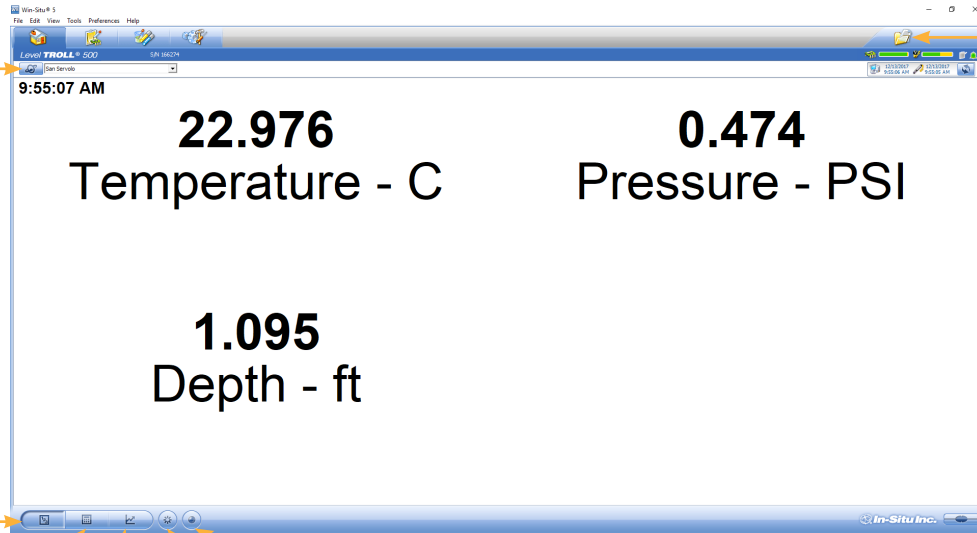
Home Tab



Display Sites list



View data files



Meter view



List view



Graph view



Record snapshot

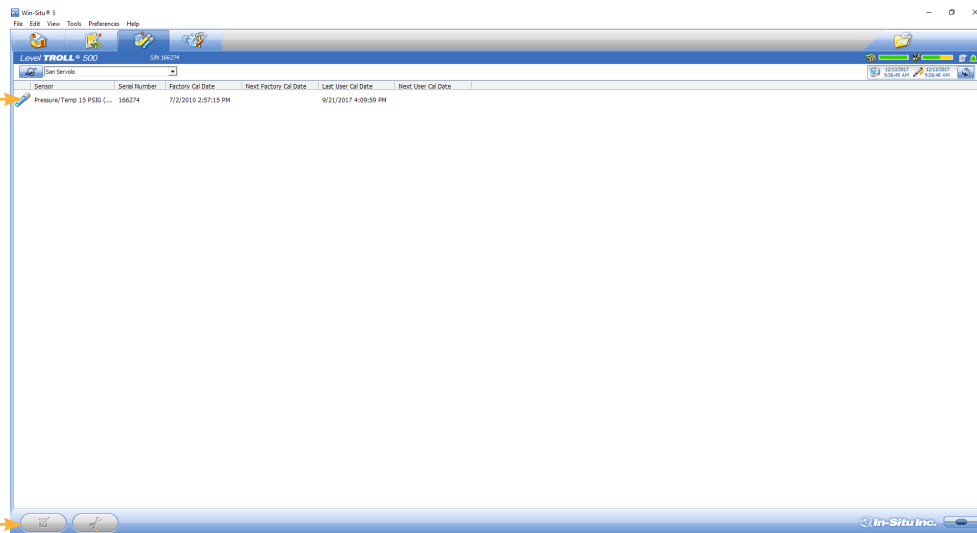


Record current values

Sensor Tab



Set up sensor (double-click)



Calibrate sensor



Set up sensor

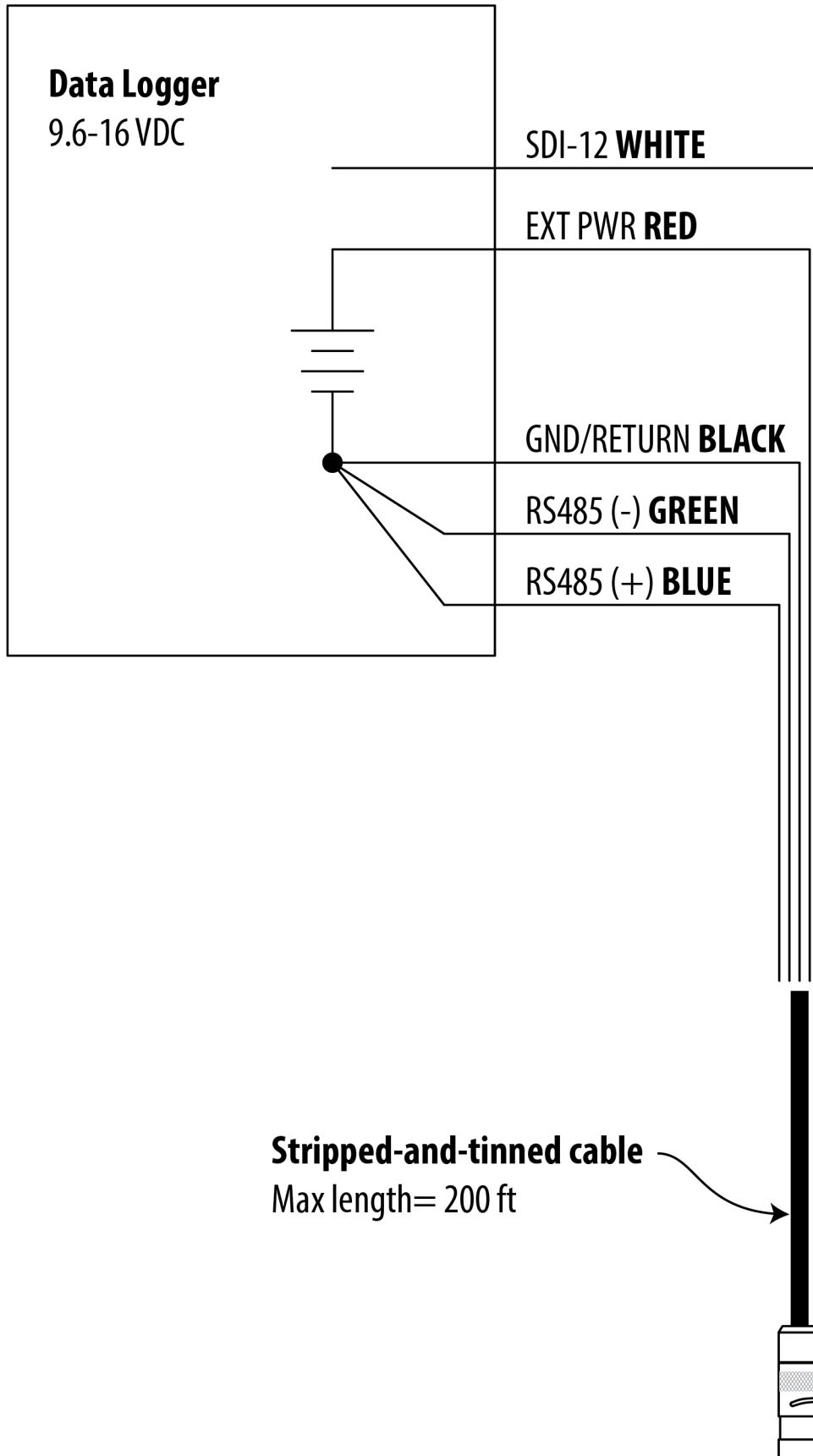
Device Setup Tab

The screenshot displays the Win-Studio software interface for device configuration. The main window shows the 'Device Setup' tab with several sub-sections: Device Information, Device Status, Power Management, and Device Firmware Update. Four orange arrows originate from the sub-tab labels at the bottom of the main window and point to four separate dialog boxes:

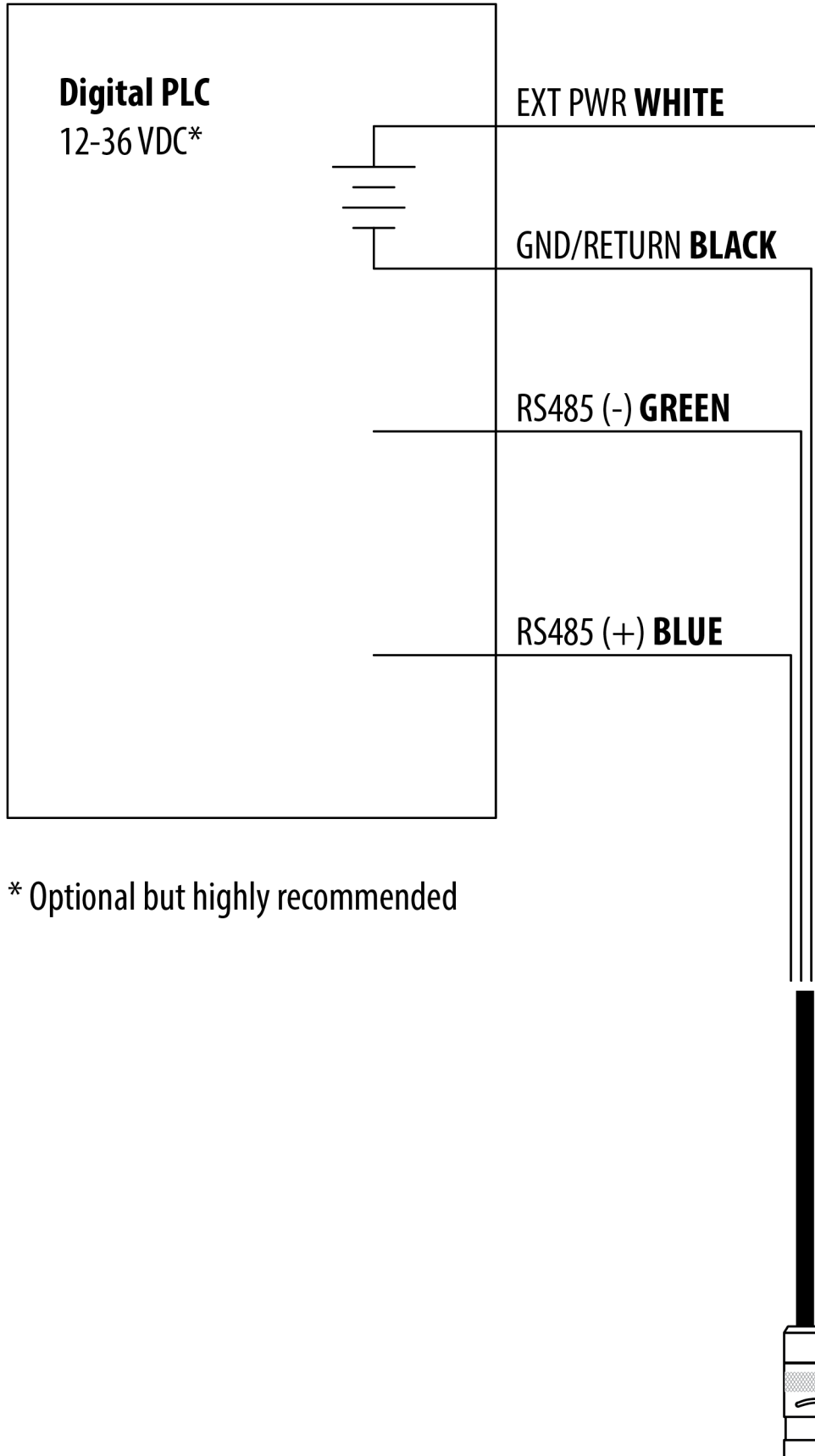
- Analog Setup:** Configures sensor parameters for 'Pres(G) 35ft'. It includes fields for 'Zero Scale (4mA)' (Value: 0) and 'Full Scale (20mA)' (Value: 15), both in PSI. It also has radio buttons for 'Disable Analog Output' and 'Enable Analog Output'.
- SDI-12 Setup:** Configures the SDI-12 protocol. It includes an 'Address Character' field (Value: 0), a sensor parameter dropdown (Pres(G) 35ft), and an 'Output Order' list containing 'Pres(G) 35ft - Pressure - PSI' and 'Pres(G) 35ft - Temperature - C'.
- Serial Communication Settings:** Configures the serial interface. It includes a 'Device Address' field (Value: 1), 'Serial Communications' settings (Baud: 19200, Data Bits: 8, Parity Bits: Even, Stop Bits: 1), and 'Timeout' settings (End of Message Timeout: 1000 ms, End of Session Timeout: 10000 ms). The Mode is set to 'Modbus-RTU'.
- Alarm Setup:** Configures alarm parameters for 'Pres(G) 35ft'. It includes 'Warning' and 'Alarm' sections, each with 'Enable Low' and 'Enable High' checkboxes and corresponding 'Low Setpoint', 'Low Reset', 'High Setpoint', and 'High Reset' fields, all in PSI.

Connecting the Aqua TROLL 500 to a PLC or Data Logger

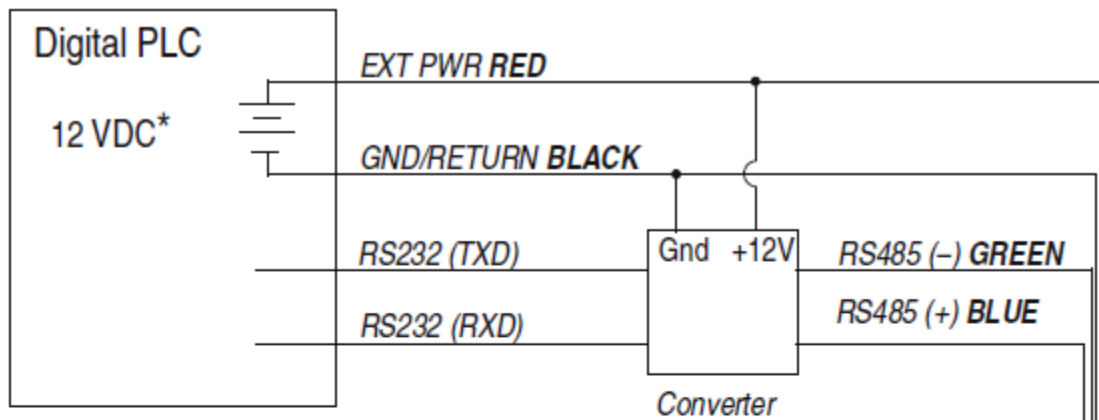
SDI-12 3 Wire



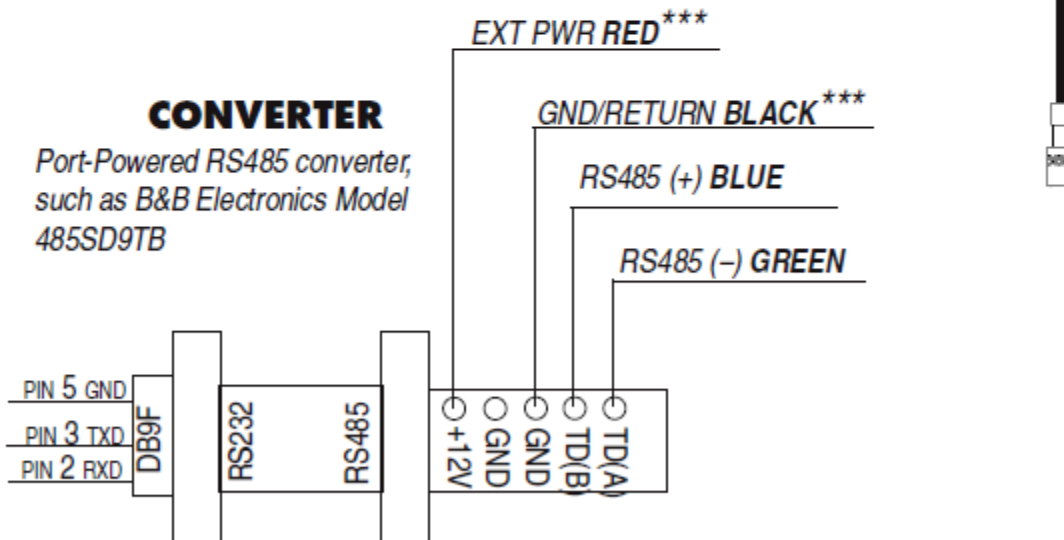
Modbus Master



Modbus Master with RS232 (Converter Required)



* Voltage limited by converter



***Required if port power is not available

Modbus PLC Interface

Overview

The Modbus PLC Interface is a simplified method of communicating with the Aqua TROLL 500 using the Modbus protocol. It reduces programming complexity and allows the user to remove sensors and reinstall them in different ports. Please observe the following limitations when using this interface:

- Only one sensor of any sensor model can be used in the sonde (for example: only one turbidity sensor can be installed).
- If a parameter is provided by more than one of the installed sensors, the interface will return the first value available.
- Firmware version 1.71 or later must be installed on the sonde.

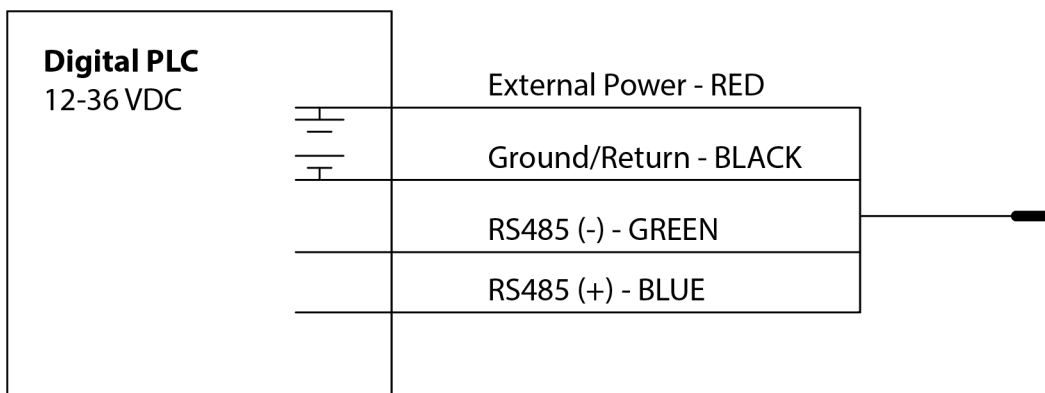
For information about the full Modbus capabilities of your sonde, see the Aqua TROLL 500/600 Interface Specification at www.in-situ.com/support/type/documentation.

Setting Up Instrument

1. Install the sensors and turn on the display by holding the instrument vertically.
 - a. Ensure the display turns on and check the LCD to ensure the sensors are working.
2. The setup below is using the instrument's factory default settings. Use WinSitu or VuSitu to reset the instrument to factory defaults if they have been changed.
 - a. Take note of any changes in default units setup.

Wiring the Modbus Master

Connect the Twist-Lock termination to the instrument and wire the stripped-and-tinned connection as shown below:



Programming the PLC

1. Setup the serial communication the following values:

Mode	Start Bit	Baud Rate	Data Bits	Parity	Stop Bit
RTU	1	19200	8	Even	1

2. Set the device address to: 1
3. Set the PLC to wake-up the device by sending any Modbus command.
 - a. This could be a carriage return, reading the slave id or reading any register.
4. Read the discovery register using Appendix A to trigger the instrument to scan the sensors.
 - a. The return value can be discarded.
 - b. Each register is a holding register. Some PLCs require you to add 40000 to the register number or address. For example: 9301 would be 49301.
 - c. Alternatively, you can prompt the instrument to discover its sensor mapping by connecting it to the VuSitu mobile app or Win-Situ software.
5. Select the register to read on the PLC using Appendix B
 - a. Some PLC devices use the register number directly in programming statements, others use register addresses, which are one less than the register number; the programmer must adhere to the PLC's programming style
 - b. Each register is a holding register. Some PLCs require you to add 40000 to the register number or address. For example: 5451 would be 45451.
6. Set the type of register to: 32-bit float
 - a. If asked by the PLC this is 2 registers
7. Set the byte order to: Big Endean (MSB)
 - a. This should be the default and may not be configurable on all PLCs

Reading Parameters

To determine the starting register number for a given parameter register block, first determine its parameter id by looking in the sensor's parameter tables. Then calculate the starting register number of the parameter block using the following equation.

$$\text{Starting Register} = (\text{Parameter Id} - 1) \times 7 + 5451$$

For example, for the Conductivity Sensor, the parameter id for specific conductivity is 10 (bit 9 will be set in register 6984 if it is available). The starting register number for the specific conductivity register block is thus $(10 - 1) \times 7 + 5451 = 5514$.

The starting register for each parameter points to a block of 7 registers that contain the following information.

Register Offset	Size (Registers)	Mode & Access Level (R/W)	Data Type	Description
0	2	R	float	The measured value from sensor
2	1	R	ushort	Data Quality Id: If this is 0 then there are no errors or warnings. See: Full System Specification
3	1	R/W	float	Units Id for the measured value. The default values are listed in the table below.
4	1	R	ushort	Parameter Id: The ID of the parameter for this location. See: Full System Specification
5	2	R/W	float	Off line sentinel value: The value that's returned on error or if the parameter isn't available. The default sentinel is 0.0

Care and Maintenance

Maintenance Schedule

For best results, send the instrument to the manufacturer for factory calibration every 12 to 18 months.

User-Serviceable Parts

The user-serviceable parts on the instrument include the O-rings, removable sensors, RDO Sensor Cap and pH/ORP/ISE reference junction filling solution.

O-rings

The instrument has several O-rings that can be maintained by the user in order to keep moisture from entering the instrument and damaging the electronics. Apply a very thin layer of vacuum grease to new O-rings upon installation. Check O-rings for cracks, chips, or discoloration and change when any of these conditions appear.

pH/ORP & ISE Sensor Replacement

To replace the pH/ORP or ISE sensor or to refill the reference junction, follow the instructions in the Instruction Sheet that is included with the replacement sensor.

RDO Sensor Cap Replacement

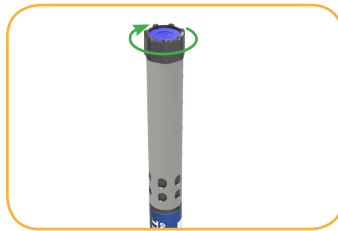
The RDO-X Sensor Cap has a 2-year typical life. The RDO Fast Cap has a 1-year typical life. Follow the instructions included in the RDO Sensor Cap Replacement Kit. Replacement caps are available from In-Situ Inc. or your authorized In-Situ distributor.

Instrument Storage

Short-term Storage (less than one week)



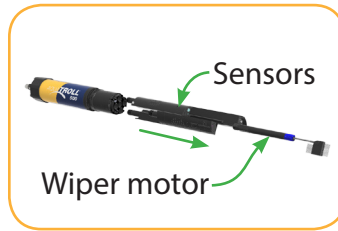
Place the restrictor in storage mode and pour ~15 mL of water, pH 4 buffer or pH/ISE storage solution over the sensors.



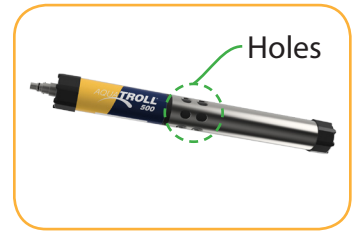
Screw the end cap onto the restrictor.

- ! pH/ORP sensor must remain wet during storage. ISE sensors may be stored dry but must be reconditioned prior to calibration and deployment.
-

Long-term Storage (more than one week)



Remove the restrictor, sensors and wiper motor.



Thread the restrictor back onto the sonde with the holes at the center of the instrument.



Add a small amount of pH storage solution or pH 4 calibration standard to the sponge inside sensor cap.



Replace caps at both ends of sensor. Use electrical tape to seal the cap onto the sensor to prevent leaks or the sponge drying out.

Cleaning the Sonde

Rinse the sonde thoroughly, clean with warm water and mild soap, then rinse the sonde again. Allow to air dry. Be sure not to allow water to enter into the connector.

Cleaning and Storing the pH/ORP Sensor

If the ORP platinum electrode is dull or dirty, it can be cleaned with a swab and methanol or isopropyl alcohol. Rub the electrode gently until it is shiny.

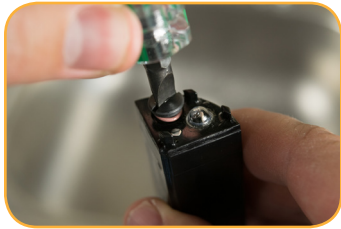
The pH sensor must be kept moist for the life of the sensor. The sensor fill solution has a shelf life of 2 years. Replace the fill solution every 5 to 6 months or when:

- The sensor fails to calibrate within the acceptable slope and offset range.
- Sensor readings vary.
- Readings during calibration at pH 7 are greater than +30 mV or less than -30 mV.
- Sensor is slow to respond.



If the sensor fails to calibrate after you replace the fill solution, replace the reference junction.

Replacing the Filling Solution



Remove sensor from sonde and unscrew reference junction.



Discard old solution.



Insert tube from filling solution bottle into sensor.



Squeeze solution into reservoir until full. Slowly remove tube.



Reinstall reference junction and wipe sensor body dry.



Soak sensor in tap water for at least 15 minutes.



If necessary, thoroughly clean the sensor connector to remove filling solution: Using a disposable pipette, fill the connector with isopropyl alcohol (70% to 100%), Shake to dry. Repeat 3 times. Dry overnight. When thoroughly dry, calibrate the sensor.

Replacing the Junction

Replace the junction when the sensor fails to calibrate with a reasonable slope and offset, even after you have replaced the filling solution.

- Unscrew the reference junction and discard.
- Replace the filling solution and screw in a new reference junction.
- Soak for 15 minutes, then calibrate the sensor.



Keep the junction damp at all times to avoid a lengthy rewetting process.

Cleaning

Begin with the most gentle cleaning method and continue to the other methods only if necessary. Do not directly wipe the glass bulb.

To clean the pH sensor, gently rinse with cold water. If further cleaning is required, consider the nature of the debris.

To remove crystalline deposits:

- Clean the sensor with warm water and mild soap.
- Soak the sensor in 5% HCl solution for 10 to 30 minutes.
- If deposits persist, alternate soaking in 5% HCl and 5% NaOH solutions.

To remove oily or greasy residue:

- Clean the sensor with warm water and mild soap.
- Methanol or isopropyl alcohol may be used for short soaking periods, up to 1 hour.
- Do not soak the sensor in strong solvents, such as chlorinated solvents, ethers, or ketones, such as acetone.

To remove protein-like material, or slimy film:

- Clean the sensor with warm water and mild soap.
- Soak the sensor in 0.1 M HCl solution for 10 minutes and then rinse with deionized water.

After performing any of these cleaning methods, rinse the sensor with water, then soak overnight in pH 4 buffer.

! After performing any of these cleaning methods, rinse the sensor with water, then soak overnight in pH 4 buffer.

Storage Recommendations

Prior to using the pH sensor after long-term storage, rinse the sensor with DI water and then soak it in pH 4 buffer for 1 or 2 hours. This will saturate the glass bulb with hydrogen ions and prepare it for use.

! Do not store the pH sensor in DI water because it will deplete the reference solution and drastically reduce the life of the sensor.

Cleaning and Storing the RDO Sensor

Routine Maintenance

1. Leave the sensor cap on.
2. Rinse the sensor with clean water.
3. Gently wipe with a soft cloth or brush if biofouling is present.
4. If extensive fouling or mineral buildup is present, soak the sensor in vinegar for 15 minutes, then soak in deionized water for 15 minutes.

! Do not use organic solvents—they will damage the sensor cap. Do not remove the sensor cap when rinsing or brushing.

6. After cleaning the sensor, perform a 2-point calibration.

Cleaning the optical window

1. Remove the cap.
2. Gently wipe the sensing window with the supplied lens cloth.

! Do not wet the lens with any liquid.

Storage

Prior to installation, store the sensor body and cap in the factory supplied containers.

Once installed on the sonde, the RDO sensor can be stored wet or dry depending on the sensor configuration of the sonde.

- ! Never store the RDO sensor without the sensor cap once it has been installed on the sonde.

Cleaning and Storing the Conductivity Sensor

Cleaning

Begin with the most gentle cleaning method and continue to the other methods only if necessary.

To clean the conductivity sensor face, gently rinse with clean, cold water. If further cleaning is required, consider the nature of the debris.

To remove crystalline deposits:

- Clean the sensor face with warm water and mild soap.
- Use a soft brush to gently clean the sensor pins and temperature button. Ensure removal of all debris around the base of the pins and button.
- If crystalline deposits persist, soak in 5% HCl for 10 to 30 minutes followed by warm soapy water and soft brushing.
- If deposits persist, alternate soaking in 5% HCl and 5% NaOH solutions followed by warm soapy water and soft brushing.

To remove oily or greasy residue:

- Clean the sensor face with warm water and mild soap.
- Using a soft brush, gently clean the sensor pins and temperature button. Ensure removal of all residue around the base of the pins and temperature button.
- Isopropyl alcohol may be used for short soaking periods, up to one hour.
- Do not soak in strong solvents such as chlorinated solvents, ethers or ketones (such as acetone).

To remove protein-like material, or slimy film:

- Clean the sensor face with warm water and mild soap.
- Using a soft brush, gently clean the sensor pins and temperature button. Ensure removal of all material/film around the base of the pins and temperature button.
- Soak the sensor in 0.10% HCl for 10 minutes and then rinse thoroughly with distilled water.

Storage

Prior to installation, store the sensor in the factory supplied container.

Once installed on the sonde, the Temperature Sensor and Conductivity Sensor can be stored wet or dry depending on the sensor configuration of the sonde. For the best accuracy over instrument life, keep the conductivity cell submersed in water for 24-48 hours prior to calibration and deployment.

Cleaning and Storing the Turbidity Sensor

Routine Maintenance

The optical windows should be clear of foreign material. To clear material gently rub the sensing windows using clean water and a soft cloth or swab. Do not use solvents on the sensor.

Storage

Prior to installation, store the sensor in the factory supplied container. Once installed on the sonde, the turbidity sensor can be stored wet or dry depending on the sensor configuration of the sonde.

Instrument Specifications

Operating temperature	-5° to 50° C (23° to 122° F)
Storage temperature	Components without fluid: -40° to 65° C (-40° to 149°F) pH/ORP probes: -5° to 65°C
Dimensions	Length: 46 cm (18.145") (includes connector) Diameter: 4.7 cm (1.860") With bail: 59cm (23.25")
Weight	0.978kg / 2.15 lbs. (includes instrument, sensors, restrictor and bumpers)
Wetted materials (sonde and sensors)	PC, PC alloy, Delrin, Santoprene, Inconel, Viton, Titanium, Platinum, Ceramic, Nylon, PVC, Graphite
Environmental rating	IP68 with all sensors and cable attached. IP67 with sensors removed, battery cover removed, or cable detached
Max pressure rating	Up to 150 PSI Ammonium/Nitrate up to 30PSI
Communication	RS485/MODBUS, Wireless TROLL Com, Bluetooth®
Reading rate	1 reading every 2 seconds
LCD screen	Integrated display shows status of sonde, sensor ports, power voltage and connectivity. BlueTooth may be disabled through the hidden menu
External power voltage External power current ¹	8-36 VDC (required for normal operation) Sleep: < 0.2 mA typical Measurement: 40 mA typical, 75 mA Max
Interface	Win-Situ 5 Software, VuSitu Mobile App on select mobile devices using Android 4.4 with Bluetooth 2.0
Cable	Vented or non-vented polyurethane or vented Tefzel®
Hex screw driver	0.050 in. (1.3 mm)
Software	Android: VuSitu through Google Play Windows: Win-Situ 5 Data Services: HydroVu
Interface	Android 4.4, requires BlueTooth 2.0
Certifications	CE, FCC, WEEE, RoHS Compliant

Sensor Specifications

Sensor	Shelf Life	Field Life	Recommended Calibration Frequency	Pressure Rating - PSI	Usable Depth		Operational Temperature Range
					Meters	Feet	
pH/ORP	15 months	1 year or greater	10 to 12 weeks	350	200	650	-5° to 50° C
RDO	NA	2 years or greater	12 months	350	200	650	-5° to 50° C
Conductivity	NA	2 years or greater	User calibration only if needed	350	200	650	-5° to 50° C
Temperature	NA	2 years or greater	NA	350	200	650	-5° to 50° C
Turbidity	NA	2 years or greater	User calibration only if needed	350	200	650	-5° to 50° C
Pressure	NA	2 years or greater	User calibration only if needed	12.8 42.7 108 285	9 30 76 200	30 100 250 650	-5° to 50° C
Barometric Pressure	NA	2 years or greater	User calibration only if needed	NA	NA	NA	-5° to 50° C
Ammonium	6 months	6 to 12 months	Monthly	30	25	70	0° to 40° C
Chloride	6 months	1 year or greater	Monthly	350	200	650	0° to 40° C
Nitrate	6 months	6 to 12 months	Monthly	30	25	70	0° to 40° C

Accuracy, Range & Resolution

Temperature ²	Accuracy	+/- 0.1° C
	Range	-5 to 50° C (23 to 122° F)
	Resolution/Precision	0.01° C
	Response Time	T63<2s, T90<15s, T95<30s
	Units of Measure	Celsius or Fahrenheit
	Method	EPA 170.1
Barometric Pressure (vented models only)	Accuracy	+/- 1.0 mBars
	Range	300 - 1100 mBars
	Resolution/Precision	0.1 mBar
	Response Time	T63<1s, T90<1s, T95<1s
	Units of Measure	Pressure: psi, kPa, bar, mbar, inHg, mmHg;
	Method	Silicon strain gauge
pH ³	Accuracy	±0.1 pH unit or better
	Range	0-14 pH
	Resolution/Precision	0.01 pH
	Response Time	T63<3s, T90<15s, T95<30s
	Units of Measure	pH, mV
	Method	Std. Methods 4500-H+, EPA 150.2
ORP ⁴	Accuracy	+/- 5 mV
	Range	±1400 mV
	Resolution/Precision	0.1 mV
	Response Time	T63<3s, T90<15s, T95<30s
	Units of Measure	mV
	Method	Std. Methods 2580

Conductivity ⁵	Accuracy	+/-0.5% of reading plus 1 µS/cm from 0 to 100,000 µS/cm; +/-1.0% of reading from 100,000 to 200,000 µS
	Range	0 to 350,000µS/cm 0-350 ppt 0-350 PSU
	Resolution/Precision	0.1 µS/cm 0.1 ppt 0.1 PSU
	Response Time	T63<1s, T90<3s, T95<5s
	Units of Measure	Actual conductivity (µS/cm, mS/cm); Specific conductivity (µS/cm, mS/cm); Salinity (PSU, ppt); Total dissolved solids (ppt, ppm); Resistivity (Ohms-cm); Density (g/cm ³)
	Method	Std. Methods 2510, EPA 120.1 Std. Methods 2520A
Rugged Dissolved Oxygen ⁶	Accuracy	±0.1mg/L +/-0.2mg/L +/-10% of reading
	Range	0 to 8 mg/L 8 to 20 mg/L 20 to 50 mg/L
	Resolution/Precision	0.01 mg/L
	Response Time	RDO-X: T63<15s, T90<45s, T95<60s Fast Cap: T63<15s, T90<45s, T95<60s
	Units of Measure	mg/L, %saturation, ppm
	Method	EPA-approved In-Situ Methods: 1002-8-2009, 1003-8-2009, 1004-8-2009
Turbidity ⁷	Accuracy	+/-2% of reading or +/-2 NTU, FNU, w.i.g.
	Range	0 – 4,000 NTU 0-1500 mg/L
	Resolution/Precision	0.01 NTU (0-1000); 0.1 NTU (1000-4000) 0.1 mg/L
	Response Time	T63<1s, T90<1s, T95<1s
	Units of Measure	NTU, FNU ppt, mg/L
	Method	ISO 7027
Ammonium ^{8,9}	Accuracy	±10% or ± 2mg/L, w.i.g.*
	Range	0-10,000 mg/L as N
	Resolution/Precision	0.01mg/L
	Response Time	T63<1s, T90<10s, T95<30s
	Units of Measure	mg/L, ppm, mV
	Method	

Unionized Ammonia, Total Ammonia	Accuracy	
	Range	0-10,000 mg/L as N
	Resolution/Precision	0.01mg/L
	Response Time	-
	Units of Measure	mg/L, ppm
	Method	-
Nitrate ⁸	Accuracy	±10% or ± 2mg/L, w.i.g.*
	Range	0-40,000 mg/L as N
	Resolution/Precision	0.01mg/L
	Response Time	T63<1s, T90<1s, T95<1s
	Units of Measure	mg/L, ppm, mV
	Method	Std. Methods 4500-NO3 D
Chloride ⁸	Accuracy	±10% or ± 2mg/L, w.i.g.*
	Range	0-150,000 mg/L - Cl-
	Resolution/Precision	0.01mg/L
	Response Time	T63<1s, T90<10s, T95<30s
	Units of Measure	mg/L, ppm, mV
	Method	Std. Methods 4500-Cl- D
Pressure ¹⁰	Accuracy	±0.1% full scale (FS)
	Range	Non-Vented or Vented 9.0 m (30 ft.) - Burst: 27 m (90 ft.) 30 m (100 ft.) - Burst: 40 m (130 ft.) 76 m (250 ft.) - Burst: 107 m (350 ft.) 100 m (325 ft.) - Burst: 200 m (650 ft.)
	Resolution/Precision	0.01% full scale
	Response Time	T63<1s, T90<1s, T95<1s
	Units of Measure	Pressure: psi, kPa, bar, mbar, inHg, mmHg; Level: in, ft., mm, cm, m; Level: in, ft., mm, cm, m
	Method	Piezoresistive; Ceramic

Warranty ¹¹	<p>2 year - Sonde, RDO and sensor cap, temperature/conductivity, temperature only, turbidity (excluding pH/ORP)</p> <p>1 year - pH/ORP, chloride ISE, accessories</p> <p>90 Days - Nitrate and Ammonium ISE sensors</p> <p>Other: see warranty policy (www.in-situ.com/warranty)</p>
Notes	<p>Specifications are subject to change without notice. Android is a trademark of Google, Inc. Bluetooth is a trademark of Bluetooth SIG, Inc. Delrin and Tefzel are trademarks of E.I. du Pont de Nemours & Co. Santoprene is a trademark of ExxonMobile. Inconel is a trademark of Special Metals Corporation. Viton is a registered trademark of DuPont Performance Elastomers L.L.C.</p>

¹ Dependent on display and wiping

² Typical system response with instrument, sensors and restrictor when changing approximately 15° C in moderate flow

³ Response time at thermal equilibrium

⁴ Accuracy from calibration standard @ 25C, response-at thermal equilibrium immediately following calibration in ZoBell's measuring from air to +400 mV

⁵ Accuracy at calibration points

⁶ RDO sensor full range 0-50mg/L, 0-500% sat. EPA-approved under the Alternate Test Procedure process

⁷ User defined reference

⁸ Between 2 calibration points immediately following proper conditioning and calibration. Varies on site conditions and environmental interferences. See sensor summary sheet for potential interferences

⁹ Average response, can be longer with increasing concentrations of ammonium

¹⁰ Typical performance across full temperature and pressure calibrated range

¹¹ Extended warranty option for sonde only (1-3 year extension for up to 5 years total)

Potential Interferents

pH

Sodium salts

Dissolved Oxygen

Temperature, atmospheric pressure, salinity, chlorinity

Ammonium

Cesium, Potassium, Thallium, pH, Silver, Lithium, Sodium

Nitrate

Perchlorate, Iodide, Chlorate, Cyanide, Bromide, Nitrite, Hydrogen Sulfide (bisulfite), Hydrogen Carbonate (bicarbonate), Carbonate, Chloride, Dihydrogen Phosphate, Hydrogen Phosphate, Phosphate, Acetate, Fluoride, Sulfate

Conductivity

Temperature

ORP

Ions that are stronger reducing agents than hydrogen or platinum, e.g., chromium, vanadium, titanium

Chloride

Hydroxide, Ammonia, Thiosulfate, Bromide, Sulfide, Iodide, Cyanide

Ammonium, Chloride and Nitrate Interferent Concentrations

Ammonium

The table below lists concentrations of possible interfering ions that cause 10% error at various levels (in ppm) of NH_4^+ .

Ion	100 ppm NH_4^+	10 ppm NH_4^+	1 ppm NH_4^+
Cesium (Cs^+)	100	10	1
Potassium (K^+)	270	27	2.7
Thallium (Tl^+)	3100	310	31
pH (H^+)	pH 1.6	pH 2.6	pH 3.6
Silver (Ag^+)	270,000	27,000	2,700
Lithium (Li^+)	35,000	3,500	350
Sodium (Na^+)	11,100	1,100	110

Chloride

The table below lists concentrations of possible interfering ions that cause 10% error at various levels (in ppm) of Cl⁻.

Ion	100 ppm Cl ⁻	10 ppm Cl ⁻	1 ppm Cl ⁻
Hydroxide (OH ⁻)	3,840	384	38.4
Ammonia (NH ₃)	6	0.6	0.06
Thiosulfate (S ₂ O ₃ ²⁻)	3	0.3	0.03
Bromide (Br ⁻)	0.68	0.068	6.8 x 10 ⁻³
Sulfide (S ²⁻)	9 x 10 ⁻⁴	9 x 10 ⁻⁶	9 x 10 ⁻⁷
Iodide (I ⁻)	1.8 x 10 ⁻⁴	1.8 x 10 ⁻⁵	1.8 x 10 ⁻⁶
Cyanide (CN ⁻)	1.5 x 10 ⁻⁵	1.5 x 10 ⁻⁶	1.5 x 10 ⁻⁷

Nitrate

The table below lists concentrations of possible interfering ions that cause 10% error at various levels (in ppm) of NO₃⁻.

Ion	100 ppm NO ₃ ⁻ as N	10 ppm NO ₃ ⁻ as N	1 ppm NO ₃ ⁻ as N
Perchlorate (ClO ₄ ⁻)	7 x 10 ⁻²	7 x 10 ⁻³	7 x 10 ⁻⁴
Iodide (I ⁻)	4	0.4	0.04
Chlorate (ClO ₃ ⁻)	30	3	0.3
Cyanide (CN ⁻)	20	2	0.2
Bromide (Br ⁻)	400	40	4
Nitrite (NO ₂ ⁻)	230	23	2
Hydrogen Sulfide (HS ⁻)	230	23	2
Bicarbonate (HCO ₃ ⁻)	440	440	44
Carbonate (CO ₃ ²⁻)	8,600	860	86
Chloride (Cl ⁻)	7,600	760	76
Dihydrogen Phosphate (H ₂ PO ₄ ⁻)	34,640	3,464	346
Hydrogen Phosphate (HPO ₄ ²⁻)	34,300	3,430	343

Phosphate (PO ₄ ³⁻)	33,900	3,390	339
Acetate (OAc ⁻)	104,200	10,420	1,042
Fluoride (F ⁻)	81,400	8,140	814
Sulfate (SO ₄ ²⁻)	685,600	68,570	6,857

Conductivity Sensor Specifications

Accuracy	±0.5% of reading plus 1 µS/cm from 0 to 100,000 µS/cm; ±1.0% of reading from 100,000 to 200,000 µS/cm
Range	0 to 350,000 µS/cm
Resolution	0.1 µS/cm
Sensor Type	Removeable
Response Time	T63<1s, T90<3s, T95<5s
Units of Measure	Actual conductivity: µS/cm, mS/cm Specific conductivity: µS/cm, mS/cm Salinity: PSU Total dissolved solids: ppt, ppm Resistivity: Ohms-cm Density: g/cm ³
Methodology	Std. Methods 2510, EPA 120.1

*Accuracy at calibration points. For greatest accuracy over instrument life, keep the conductivity cell submerged in water for 24-48 hours prior to calibration and deployment.

Total Dissolved Solids

TDS is derived from conductivity and temperature.

Range	0 to 350 ppt
Resolution	0.1 ppt
Units of Measure	ppt, ppm

Salinity

Salinity is derived from pH, conductivity and temperature.

Range	0 to 350 ppt
Resolution	0.1 PSU
Units of Measure	PSU, ppt
Methodology	Std. Methods 2520A

Dissolved Oxygen RDO Sensor Specifications

Accuracy	± 0.1 mg/L from 0 to 8 mg/L ± 0.2 mg/L from 8 to 20 mg/L $\pm 10\%$ of reading from 20 to 50 mg/L
Range	0 to 8 mg/L 8 to 20 mg/L 20 to 50 mg/L Full operating range: 0 to 50 mg/L; 0 to 500% saturation
Resolution	0.01 mg/L
Sensor Type	Removable with replaceable RDO-X Cap
Response Time	RDO-X Cap: T63<15s, T90<45s, T95<60s
Units of Measure	mg/L, % saturation, ppm
Methodology	EPA-approved In-Situ Methods (under the Alternate Test Procedure process): 1002-8-2009, 1003-8-2009, 1004-8-2009

Level, Depth and Pressure Sensor Specifications

Accuracy	Typical $\pm 0.1\%$ full scale (FS)
Range	Non-vented or Vented 9.0 m (30 ft) - Burst: 27 m (90 ft) 30 m (100 ft) - Burst: 40 m (130 ft) 76 m (250 ft) - Burst: 107 m (350 ft) 100 m (325 ft) - Burst:
Resolution	$\pm 0.01\%$ FS or better
Sensor Type	Fixed
Response Time	T63 < 1s, T90 < 1s, T95 < 1s
Units of Measure	Pressure: psi, kPa, bar, mbar, mmHg, inHg, cmH ₂ O, inH ₂ O Level: mm, cm, m, in, ft, cmH ₂ O, inH ₂ O
Methodology	Piezoresistive; ceramic

*Typical performance across full temperature and pressure calibrated range. Typical is defined as all values within 1 standard deviation.

ORP Sensor Specifications

Accuracy	± 5.0 mV @ 25° C
Range	$\pm 1,400$ mV
Resolution	0.1 mV
Sensor Type	Replaceable pH/ORP combo sensor
Response Time	T63 < 3s, T90 < 15s, T95 < 30s
Units of Measure	mV
Methodology	Std. Methods 2580

*Accuracy from standard at 25° C.

**At thermal equilibrium immediately following calibration, measuring from air to +400 mV

pH Sensor Specifications

Accuracy	± 0.1 pH units or better
Range	0 to 14 pH units
Resolution	0.01 pH unit
Sensor Type	Replaceable pH/ORP combo sensor

Response Time	T63<1s, T90<2s, T95<3s
Units of Measure	pH units
Methodology	Std. Methods 4500-H+, EPA 150.2

*At thermal equilibrium.

Temperature Sensor Specifications

Accuracy	±0.1° C
Range	-5 to 50° C (23 to 122° F)
Resolution	0.01° C
Sensor Type	Replaceable
Response Time	T63<2s, T90<15s, T95<30s
Units of Measure	° C, ° F
Methodology	EPA 170.1

Sensor only, when transferring from air to ambient water temperature. Typical system response time with all sensors and restrictor installed: T63<30s; T90<3.5m; T95,7.5m

Turbidity Sensor Specifications

Accuracy	±2% of reading or ±2 NTU or FNU, whichever is greater
Range	0 to 4,000 NTU
Resolution	0.01 NTU (0 to 1,000 NTU) 0.1 NTU (1,000 to 4,000 NTU)
Sensor Type	Replaceable
Response Time	T63<1s, T90<1s, T95<1s
Units of Measure	NTU, FNU
Methodology	ISO 7027

Total Suspended Solids

TSS is derived from turbidity.

Range	0 to 1,500 mg/L
Resolution	0.1 mg/L
Units of Measure	ppt, mg/L

User-defined reference.

1-970-498-1500

Ammonium Sensor Specifications

Accuracy	±10% or ±2 mg/L, w.i.g.*
Max Depth	25 m, 30 PSI
Range	0-10,000 mg/L as N
Resolution	0.01 mg/L
Sensor Type	Removable
Response Time*	T90 < 10sec, T95 < 30sec
Units of Measure	mg/L, ppm, mV
Methodology	Std. Methods 4500-NH ₃ D, EPA 350.3

*Between calibration points.

Ammonia (Un-ionized Ammonia and Total Ammonia)

Ammonia is derived from ammonium, pH, salinity and temperature.

Range	0 to 10,000 mg/L
Resolution	0.01 mg/L
Units of Measure	mg/L, ppm

Chloride Sensor Specifications

Accuracy	±10% or ±2 mg/L, w.i.g.*
Range	0-190,000 mg/L - CL
Resolution	0.01 mg/L
Sensor Type	Removable
Response Time*	T90 < 10sec, T95 < 30sec
Units of Measure	mg/L, ppm, mV
Methodology	Std. Methods 4500-Cl-D

*Between calibration points.

Nitrate Sensor Specifications

Accuracy	$\pm 10\%$ or ± 2 mg/L, w.i.g.*
Max Depth	25 m, 30 PSI
Range	0-10,000 mg/L as N
Resolution	0.01 mg/L
Sensor Type	Removable
Response Time*	T90 < 10sec, T95 < 30sec
Units of Measure	mg/L, ppm, mV
Methodology	Std. Methods 4500-NO 3D

*Between calibration points.

More Information

! To learn more about the Aqua TROLL 500, telemetry, software and other In-Situ products, see the resources listed below.

1 **Visit www.in-situ.com**

Find information about In-Situ water quality, water level, telemetry and other products. Download software, manuals and product instructions.

2 **View the [In-Situ YouTube channel](#).**

Get video instructions for the Aqua TROLL 500 and other instruments. Watch quickstart videos and other tutorials.

3 **Call [In-Situ's technical support team](#).**

For further instructions and help with technical questions, call the In-Situ support line.

Appendix

Appendix A: Sensor Discovery

The first register read in a PLC measurement sequence should be a 14-register block beginning with register number 6984. Reading these registers triggers the sonde to scan its sensor ports and update its sensor map. This guarantees the sonde has properly registered any changes to the sensor configuration a user may have made since the last measurement sequence. The bitwise contents of these registers indicate which parameter IDs (1 to 219) are currently available from the sonde according to the table below. Refer to Appendix B for a description of the parameter ids.

Parameter ID Map

Register	Bit				
	15	14	13...2	1	0
6984	16	15	14...3	2	1
6985	32	31	30...19	18	17
6986	48	47	46...35	34	33
6987	64	63	65...51	50	49
6988	80	79	78...67	66	65
6989	96	95	94...83	82	81
6990	112	111	110...99	98	97
6991	128	127	126...115	114	113
6992	144	143	142...131	130	129
6993	160	159	158...147	146	145
6994	176	175	174...163	162	161
6995	192	191	190...179	178	177
6996	208	207	206...195	194	193
6997	0	0	219...211	210	209

Appendix B: Parameter Numbers and Locations

ID	Parameter Name	Holding Register Number	Holding Register Address	Default Units
1	Temperature	5451	5450	1 = °C
2	Pressure	5458	5457	17 = PSI
3	Depth	5465	5464	38 = feet
4	Level, Depth to Water	5472	5471	38 = feet
5	Level, Surface Elevation	5479	5478	38 = feet
9	Actual Conductivity	5507	5506	65 = $\mu\text{S}/\text{cm}$
10	Specific Conductivity	5514	5513	65 = $\mu\text{S}/\text{cm}$
11	Resistivity	5521	5520	81 = ohm-cm
12	Salinity	5528	5527	97 = PSU
13	Total Dissolved Solids	5535	5534	114 = ppt
14	Density of Water	5542	5541	129 = g/cm^3
16	Barometric Pressure	5556	5555	22 = mmHg
17	pH	5563	5562	145 = pH
18	pH mV	5570	5569	162 = mV
19	ORP	5577	5576	162 = mV
20	Dissolved Oxygen Concentration	5584	5583	117 = mg/L
21	Dissolved Oxygen % Saturation	5591	5590	177 = % saturation
22	Nitrate (NO_3^-)	5598	5597	117 = mg/L
23	Ammonium (NH_4^+)	5605	5604	117 = mg/L
24	Chloride (Cl)	5612	5611	117 = mg/L
25	Turbidity	5619	5618	194 = NTU
26	Battery Voltage	5626	5625	163 = Volts

ID	Parameter Name	Holding Register Number	Holding Register Address	Default Units
30	Oxygen Partial Pressure	5654	5653	26 = torr
31	Total Suspended Solids	5661	5660	117 = mg/L
32	External Voltage	5668	5667	163 = Volts
33	Battery Capacity (remaining)	5675	5674	241 = %
34	Rhodamine WT Concentration	5682	5681	118 = µg/L
35	Rhodamine WT Fluorescence Intensity	5689	5688	257 = RFU
36	Chloride (Cl ⁻) mV	5696	5695	162 = mV
37	Nitrate as Nitrogen (NO ₃ as N) Concentration	5703	5702	117 = mg/L
38	Nitrate (NO ₃ ⁻) mV	5710	5709	162 = mV
39	Ammonium as Nitrogen (NH ₄ as N) Concentration	5717	5716	117 = mg/L
40	Ammonium (NH ₄) mV	5724	5723	162 = mg/L
41	Ammonia as Nitrogen (NH ₃ as N) Concentration	5731	5730	117 = mg/L
42	Total Ammonia as Nitrogen (NH ₃ as N) Concentration	5738	5737	117 = mg/L
48	Eh	5780	5779	162 = mg/L
49	Velocity	5787	5786	118 = µg/L
50	Chlorophyll-a Concentration	5894	5793	118 = µg/L
51	Chlorophyll-a Fluorescence Intensity	5801	5800	257 = RFU
54	Blue Green Algae-Phycocyanin Concentration	5822	5821	118 = µg/L
55	Blue Green Algae-Phycocyanin Fluorescence Intensity	5829	5828	257 = RFU
58	Blue Green Algae-Phycocerythrin Concentration	5850	5849	118 = µg/L
59	Blue Green Algae-Phycocerythrin Fluorescence Intensity	5857	5856	257 = RFU

Appendix C: Unit IDs

ID	Abbreviation	Units
Temperature		
1	C	Celsius
2	F	Fahrenheit
3	K	Kelvin
Pressure, Barometric Pressure (17-32)		
17	PSI	Pounds per square inch
18	Pa	Pascals
19	kPa	Kilopascals
20	Bar	Bars
21	mBar	Millibars
22	mmHg	Millimeters of Mercury (0° C)
23	inHg	Inches of Mercury (0° C)
24	cmH ₂ O	Centimeters of water (4° C)
25	inH ₂ O	Inches of water (4° C)
26	Torr	Torr
27	atm	Standard atmosphere
Distance/Length (33-48)		
33	mm	Millimeters
34	cm	Centimeters
35	m	Meters
36	km	Kilometers
37	in	Inches
38	ft	Feet
Coordinates (49-64)		
49	deg	Degrees
50	min	Minutes
51	sec	Seconds
Conductivity (65-80)		
65	μS/cm	Microsiemens per centimeter
66	mS/cm	Millisiemens per centimeter
Resistivity (81-96)		
81	ohm-cm	Ohm-centimeters
Salinity (97-112)		
97	PSU	Practical salinity units
98	ppt	Parts per thousand salinity
Concentration (113-128)		
113	ppm	Parts per million
114	ppt	Parts per thousand
115		(available)
116		(available)
117	mg/L	Milligrams per liter
118	μg/L	Micrograms per liter
119	---	(deprecated, no longer available)

120	g/L	Grams per liter
121	ppb	Parts per billion
Density		
129	g/cm ³	Grams per cubic centimeter
pH		
145	pH	pH
Voltage (161-176)		
161	μV	Microvolts
162	mV	Millivolts
163	V	Volts
Dissolved Oxygen (DO) % Saturation (177-192)		
177	% sat	Percent saturation
Turbidity (193-208)		
193	FNU	Formazin nephelometric units
194	NTU	Nephelometric turbidity units
195	FTU	Formazin turbidity units
Flow (209-224)		
209	ft ³ /s	Cubic feet per second
210		(available)
211		(available)
212	ft ³ /day	Cubic feet per day
213	gal/s	Gallons per second
214	gal/m	Gallons per minute
215	gal/hr	Gallons per hour
216	MGD	Millions of gallons per day
217	m ³ /sec	Cubic meters per second
218		(available)
219	m ³ /hr	Cubic meters per hour
220		(available)
221	L/s	Liters per second
222	ML/day	Millions of liters per day
223	mL/min	Milliliters per minute
224	kL/day	Thousands of liters per day
Volume (225-240)		
225	ft ³	Cubic feet
226	gal	Gallons
227	Mgal	Millions of gallons
228	m ³	Cubic meters
229	L	Liters
230	acre-ft	Acre feet
231	mL	Milliliters
232	ML	Millions of liters
233	kL	Thousands of liters
234	acre-in	Acre inches
% (241-256)		
241	%	Percent

Fluorescence		
257	RFU	Relative fluorescence units
Low-Flow (273-288)		
273	ml/sec	Milliliters per second
274	ml/hr	Milliliters per hour
275	l/min	Liters per minute
276	l/hr	Liters per hour
Current (289-304)		
289	μA	Microamps
290	mA	Milliamps
291	A	Amps
Velocity		
305	ft/s	Feet per second
306	m/s	Meters per second

Appendix D: Register Data Formats

The Modbus protocol specification requires any multiple-byte data type to be transmitted in Big Endian order, or most significant byte (MSB) first. In-Situ devices shall use the following register data formats.

ID	Type	Size (Registers)	Description
2	Unsigned Short	1	2 bytes, 1 register, MSB first
5	Float	2	4 bytes, 2 registers. IEEE floating point format

IMPORTANT: ENSURE THAT THE PANEL RETAINING THUMB SCREWS (NUTS) ARE TIGHT BEFORE USE.

Part Numbers
 1403 Detach-WLM probe
 1408 1408 DP-EP
 3250 TScrewsx2

Item
 Detachable WLM probe
 DrawDown Electronic Panel
 Thumbscrews Set (2)

Figure 3
 Unique hanger and tape protector supports the meter on the casing and protects the tape from sharp edges on the well casing.

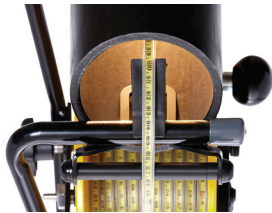
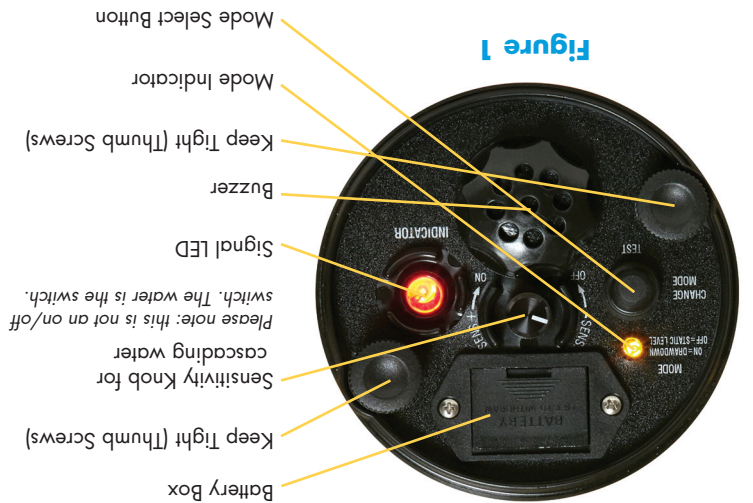


Figure 2
 To test entire system, Hold the center pin on the probe against the stud on the back of the screw on the frame. The buzzer will sound if the system is okay. **Make sure sensitivity dial is turned fully clockwise.**



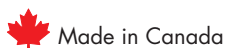
WE ALSO CARRY:

- Data Loggers
- Interface Meters
- Conductivity Meters
- Temperature Meters
- Well Casing Indicators
- Well Depth Indicators

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Please visit our website www.heroninstruments.com for more information on the complete Heron line.



HERON INSTRUMENTS
 Groundwater Monitoring INC.

dipper-T Water Level Meter with drawDown

Operating and Maintenance Instructions



www.heroninstruments.com

dipper-T Water Level Meter Instructions

General Care of the dipper-T

The dipper-T is a high quality, robust unit that will give many years of reliable service if these recommendations are followed:

- Avoid sharp edged casing, use the tape guide on the unit.
- Take care to avoid the tape becoming entangled with other equipment in boreholes or wells, use stilling pipes when possible.
- **DO NOT** use the dipper-T as a guide to backfilling, bentonite sealing or sand packing in wells. This type of material falls through the water column at a much slower rate than the dipper-T probe and can result in a trapped probe and tape.
- Neatly rewind and clean the tape after each use.
Refer to: [Cleaning the dipper-T](#)

Warranty is conditional upon adherence to these guidelines

Equipment Check

Before taking the unit into the field, carry out these simple tests with the sensitivity knob turned fully clockwise (see Figure 1 panel controls), and the two panel retaining thumb screws (nuts) are tight.

The sensitivity knob is used to adjust the unit's response to varying conductivities to maintain a sharp distinctive signal. Turn the knob clockwise for low conductivity (pure) water and anti clockwise for high conductivity (dissolved minerals) water.

NOTE: There is no on/off switch on the instrument. If using in **DRAWDOWN** mode, return the unit to **STATIC** mode to turn the meter off. The **dipper-T** consumes no power in **STATIC** mode when not in water.

To check the condition of the battery and circuit, press the **CHANGE MODE** button.

The unit should sound and the **INDICATOR** light will come on. If the unit does not respond, replace the battery and try again.

To test the entire unit from the panel to the probe, short the probe across the stand off screw and the pin on the axle as shown in (Figure 2), the unit should sound as above.

Use in the Field

The **dipper-T** will operate in two modes, **STATIC** level and **DRAWDOWN**. In the **STATIC** mode, the unit will be silent until it touches the ground water. In **DRAWDOWN** mode, the unit will be sounding and the light will be on until it touches water.

Reel the tape down the well carefully avoiding the edge of the casing. Hang the **dipper-T** on the casing when possible and run the tape over the guide on the frame leg to avoid cuts and nicks to the tape. Swivel the probe holder on the frame to allow the tape free movement down the well. (See Figure 3)

When the unit sounds (in **STATIC** mode) carefully measure the depth to water indicated on the tape from your reference point. Raise and lower the tape in and out of the water to get a consistent result.

NOTE: The inverted triangle on the probe holder serves as a datum point indicating "top of casing". In wells that have cascading water that may give false readings, reduce the sensitivity by turning the sensitivity knob anti-clockwise.

The **dipper-T** probe is depth rated to the full length of the tape.

DO NOT use the **dipper-T** to measure the progress of back filling wells. The tape and probe may become stuck in the backfill material.

DO NOT allow the tape to "freewheel" down the well. It may become caught in other equipment in the well.

When rewinding the tape remove as much water and debris as possible from the tape and the probe.

Cleaning the dipper-T

Always clean the meters after use in the field to maintain optimal performance and extend the life of the unit.

The **dipper-T** may be cleaned with any mild household dishwashing detergent and rinsed with water.

DO NOT use abrasives, partially halogenated hydrocarbons or ketones to clean the reel. If the electronic panel is removed first, the reel and tape can be washed with a power washer.

Remove the panel retaining thumb screws (nuts) to release the panel. Take care not to lose the thumb screws as the unit will not work without them.

For longer units with aluminium reel discs, the panel is connected to the tapes with male and female push connectors.

Troubleshooting the dipper-T

Q. No sound or light when the unit is tested.

A. Refer to **Equipment Check** and follow procedures. Change the battery. Switch from **STATIC** to **DRAWDOWN** and back. If the unit is in **DRAWDOWN** mode and it sounds, put the probe in water and the sound should stop.

Q. Equipment checked, panel okay but probe not working.

A. Make sure both panel retaining thumb screws (nuts) are tight.

Q. Every thing okay, but the probe is still not working.

A. Carry out full continuity test, as shown in (Figure 2).

Q. Still not working in STATIC mode.

A. Adjust the sensitivity setting. Check the probe connection to the tape. Carefully remove the probe, wipe the male connector and replace the probe, tighten fully (do not lose the O-ring). If the unit still does not work check all the connections inside the hub. (Inside the hub polarity is not an issue as the current is AC.)

Q. The instrument continues to sound when not in water.

A. Press the **MODE** button. Dry the probe with a clean cloth if the water is saline. Remove the probe and dry the male connector. If at this stage the unit stops sounding, replace the probe. If the instrument starts sounding again there is a short in the probe.

Contact Heron Instruments or your supplier if you cannot isolate the problem.

Warranty (5 years, probe 1 year)

Heron Instruments Inc. warrants to repair or replace any such defective equipment or part (determined to our satisfaction to have a defect in workmanship or original material) upon receipt and inspection of such defective equipment to Heron Instruments Inc. with all shipping pre-paid by the user.

In no event shall Heron be liable for any direct, indirect or consequential damages, abuse, acts of third parties (rental equipment), environmental conditions or other expenses which may arise in connection with such defective equipment.

This warranty shall not apply to damage of equipment caused by improper installation, usage, storage, alteration or inadequate care.

Heron Warranty coverage does not extend to the following:

- Tape, bag or batteries used with the product
- Products used as rental equipment
- Products contaminated by materials which are shown to be hazardous and; as such, have rendered the unit unserviceable
- Parts failure due to neglect in cleaning or servicing
- Failure of parts caused by misuse

When returning a product for service, please contact Heron Instruments in advance:

- by telephone at **1-800-331-2032 or 905-628-4999**
- by email at **info@heroninstruments.com**

Warranty is conditional upon adherence to these guidelines.



ATTACHMENT B

U.S. EPA Region 5 Analytical Services Branch
PFAS Sampling Fact Sheet

and

PFAS-Specific Sampling Precautions,
Procedures, and Requirements document

EPA Region 5 Analytical Services Branch

PFAS Sampling Fact Sheet

Aqueous Sampling Instructions

- Collect a grab sample of approximately 5 mL (i.e. 5 mL ± 1 mL) in a pre-weighed 15 mL polypropylene tube (e.g. Celltreat part #229411 or equivalent). Additional tubes must be collected for replicates and matrix QC (i.e. laboratory duplicates and matrix spikes). Do **NOT** fill the entire tube, only to the 5 mL hash mark. Do **NOT** sub-sample in the field.
 - Minimum # of tubes required per sample location = 3
 - Minimum # of tubes required for a sample location including matrix QC = 6
- If requested, the laboratory can provide pre-weighed tubes for the field as well as polished reagent water for equipment, field, and trip blanks. Otherwise, field personnel must pre-weigh sample tubes before collecting samples and provide those weights to the laboratory.
 - Pre-weighed tubes provided by the laboratory will have the individual weights written on each tube. Do **NOT** add additional markings or stickers to the tubes.
 - The laboratory encourages field personnel to include equipment, field, and trip blanks with sampling sets.
- Collect at least the minimum # of tubes required for each sampling location and place each set of tubes per sampling location in a clear plastic sealable bag. Label the bag with all the sample information, **NOT** the individual tubes.
- When completing the chain of custody, each bag of tubes must be listed as individual line items. Each line is considered an individual sample by the laboratory.
 - If matrix QC analysis is being requested for a specific sampling location, indicate that on the individual sample line on the chain of custody. Do **NOT** add additional line items on the chain of custody for matrix QC. See Figure 1.
- When packing the samples in a cooler, keep the tubes in the plastic bags and pack the bags amongst adequate amounts of ice. Do **NOT** use a tube rack or otherwise remove the tubes from the bags.
- The temperature of the sample tubes must be < 6°C when received by the laboratory.

Solids Sampling Instructions

- Collect a grab sample in a 50 mL polypropylene tube (e.g. Celltreat part #229421 or equivalent), filling the entire tube.
 - Minimum # of tubes required per sample location = 2
- If requested, the laboratory can provide polished reagent water for equipment, field, and trip blanks. The laboratory encourages field personnel to include equipment, field, and trip blanks with sampling sets.
- Label each sample tube with all the sample information. Label all sample replicate tubes identically as the laboratory considers them to be a single sample.
- When completing the chain of custody, each sampling location must be listed as individual line items. Each line is considered an individual sample by the laboratory.
 - If matrix QC analysis is being requested for a specific sampling location, indicate that on the individual sample line on the chain of custody. Do **NOT** add additional line items on the chain of custody for matrix QC. See Figure 1.
- When packing the samples in a cooler, place the tubes in clear plastic sealable bags and pack the sample bags amongst adequate amounts of ice. Do **NOT** use a tube rack or otherwise remove the tubes from the bags.
- The temperature of the sample tubes must be < 6°C when received by the laboratory.

Region 5 Analytical Services Branch

PFAS Sampling Fact Sheet

Prohibited Field Materials

Below are common sources of PFAS contamination in the field that should be avoided when sampling. The laboratory encourages field personnel to include equipment, field, and trip blanks with sampling sets.

- Waterproof, water-resistant, or stain-resistant clothing/products
- Certain personal care products (e.g. cosmetics and lotions)
- Certain insect repellants and sunscreens
- Water or oil-resistant paper (e.g. fast food wrappers)
- PTFE- or FEP-containing products (e.g. Teflon®)
- PVDF- or ETFE-containing products (e.g. certain kinds of tubing)
- Latex gloves
- Plastic clipboards, binders, or hardcover spiral books
- Post-it® notes
- Recycled paper products (e.g. paper towels and notebook paper)
- Chemical (blue) ice packs
- Disposable glass pipettes
- Aluminum foil
- Kim® wipes

Figure 1. Sample Line on Chain of Custody

STA. NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION	CON-TAINERS	Analyte				TAG NUMBERS
							P	F	A	S	
S01	1/14/20	12:00		X	Sample #1	6	X				matrix QC sample

Disclaimer: The mention of trade names or commercial products in this document does not constitute an official EPA endorsement or rejection.

Attachment 2. PFAS-Specific Sampling Precautions, Procedures, and Requirements

The collection of environmental samples for PFAS analysis requires specific sampling considerations. Due to the ubiquitous nature of PFAS compounds that can occur in a wide variety of materials and very low, parts-per-trillion (ppt) screening levels, it is very important to follow sampling requirements listed in this section to reduce chances of external contamination sources, as well as preventing cross-contamination between sample locations which would jeopardize field sample results.

PFAS compounds can be found in many different materials that are commonly used or worn by field samplers. The most notable PFAS containing material is Teflon[®] that can be found in a variety of textiles and sampling equipment components. For example, polytetrafluoroethylene products (tubing, sample containers, and sampling tools) are often used in sampling; however, since these products can contain PFAS, they cannot be used in sampling for PFAS. In addition, many consumer goods brought to a sampling site may contain PFAS that can contaminate samples. Field sampling and laboratory hygiene protocols are critical to ensuring that testing results reflect actual PFAS levels in the analyzed media. The Interstate Technology and Regulatory Council (ITRC) has summarized site characterization, sampling precautions, and analytical method issues and options through their fact sheet series:

https://pfas-dev.itrcweb.org/wp-content/uploads/2020/10/sampling_analytical_508_2020Aug_Final.pdf

Table A-1, below, provides a list of potential materials that could contain PFAS compounds. Note that it may not be possible to avoid all "do not use" materials listed in Table A-1. In these cases, field blank sampling can be focused on materials and equipment when its use cannot be avoided. As such, field blank samples should be used to assess the relative impact to field samples and will assist in determining if field materials and equipment contain measurable PFAS compounds.

It is highly recommended that the user of this SAP/QAPP determine if there are state-specific PFAS standard operating procedures, also known as "SOPs" in the specific state that sampling will be conducted. Some states, such as Michigan and California, have developed very detailed groundwater, soil, and drinking water sampling guidance documents that are specific to PFAS. State derived guidance documents, such as these, can be a useful resource for additional groundwater, soil, drinking water, and general PFAS sampling procedures and can complement the general SOPs outlined in this SAP/QAPP.

As an example, Michigan guidance documents can be found at:

https://www.michigan.gov/documents/pfasresponse/Groundwater_PFAS_Sampling_Guidance_637871_7.pdf

https://www.michigan.gov/documents/pfasresponse/Soil_PFAS_Sampling_Guidance_639407_7.pdf

https://www.michigan.gov/documents/pfasresponse/Residential_Well_PFAS_Sampling_Guidance_634601_7.pdf

https://www.waterboards.ca.gov/pfas/docs/sept_2020_pfas_sampling_guidelines.pdf

The user of this template should verify with the selected analytical laboratories for the specific sample shipping guidelines for PFAS samples. Specifically, the following actions should be followed during sample shipping:

- Regular wet-ice should be used to cool and maintain the samples to the appropriate temperature requirements;
- Double bag ice in low density polyethylene (LDPE) resealable storage bags if needed;
- Samples should be bagged in Ziploc[®] (or similar bags) to organize them and keep them dry from melting ice;
- Chain of custody (COC) forms should be bagged in Ziploc[®] (or similar bags) so the forms do not directly touch ice or melted water;
- Bagged COC and other forms should be taped to the inside of the cooler lid;
- A clearly labeled temperature blank sample (high density polyethylene [HDPE] or polypropylene bottle filled with tap water) will be placed in each sample cooler;
- The cooler with ice, samples, and forms should be taped closed with a custody seal and shipped by overnight courier; and
- Sample cooler should be shipped as soon as possible (e.g., overnight) to ensure the samples arrive within the analytical holding time specified by the lab.

Table A-1. List of Materials that May Contain Per- and Polyfluoroalkyl Substances (PFAS) and Should be Avoided and Respective Replacements. Compiled from information provided by Michigan Department of Environmental Quality (2018) and ITRC (2020).	
Materials that Should be Avoided	Acceptable Replacements (when possible)
Teflon [®] , polytetrafluoroethylene (PTFE) - component of a variety of water and stain proof materials, lubricants, hose/tubing lining	High density polyethylene (HDPE) - bottles/storage containers; most 5-gallon buckets
Hostaflon [®] , fluorinated ethylene-propylene (FEP) - component of a variety of electrical equipment, pipes, labware, and liquid containment vessels	Polypropylene - bottles/storage containers/bottle caps, baling twine, packaging tape Acetate - soil core liners, films, textiles, some absorbency products, packaging tape
Tefzel [®] , ethylene tetrafluoroethylene (ETFE) - component of certain kinds of tubing/piping, wire coatings, protecting films	Silicon - tubing/seals/sealants Stainless steel - pump bodies, samplers, mixing bowls, storage containers
Neoflon [®] , polychlorotrifluoroethylene (PCTFE) - component of valves, seals gaskets and food packaging materials	Polyvinyl chloride (PVC) - plumbing, cable insulation, imitation leather, scrub brushes, some sheathing/drop cloths, bladder pump bodies
Low-density polyethylene (LDPE) - common component of bottles, plastic bags, and storage containers	Glass - bottles/storage containers, mixing bowls; Note that PFAS compounds will readily adsorb to glass and can be difficult to remove; Therefore, direct contact of sample media to glass should be avoided
Kynar [®] , polyvinylidene fluoride (PVDF) - component of certain kinds of tubing, aluminum foil, batteries	
Waterproof, water-resistant, or stain-resistant clothing/products (synthetic and leather); These garments are usually advertised as waterproof, water-repellant, or dirt and/or stain-resistant characteristics; This also includes any spray coatings (i.e. Scotchgard [™]) that are applied to any garments or shoes/boots	Well-laundered clothing, defined as clothing that has been washed 6 or more times after purchase (not laundered with water, dirt, and/or stain-resistant garments), made of non-treated synthetic or natural fibers (preferably cotton); Polyurethane or PVC boots/rain coats are viable alternative to water-resistant textiles

Tyvek [®] suits	
Personal care products (e.g. cosmetics and lotions)	Do not use; the only allowable insect repellants are OFF Deep Woods and Sawyer Permethrin
Certain insect repellants	
Fabric softener - laundry wash and drying	
Popup tents	Avoid when possible, set up away from sample collection/preparation areas
Certain sunscreens	Banana Boat, Meijer Lotion Broad Spectrum, and Neutrogena Ultra-Sheer Dry-Touch, and Coppertone sunscreens can be used but not in the sample collection or processing areas; Wash hands thoroughly after application before sampling
Latex gloves	Powderless nitrile gloves
Water or oil-resistant paper, containers, and cups (e.g. fast food wrappers, butcher paper, aluminum foil)	Do not eat in sampling collection/preparation areas Bottled water and hydration drinks (i.e. Gatorade [®] and Powerade [®]) can be consumed but not where samples are being collected
Ziploc [®] bags (or similar)	Usually made of LPDE (which is a do not use component); However, it is not possible to not use Ziploc [®] bags; It is okay to use this item as long as sample material (such as soil) does not come in contact with the bags: It is also okay to use Ziploc [®] bags to bag sample bottles, wet ice, and chain of custody forms when shipping samples
Plastic clipboards, binders, or hardcover spiral books	Aluminum, HDPE, or Masonite field clipboards
Post-it [®] notes	Sharpies [®] , regular pens, and graphite pencils
Rite in the Rain [®] products (paper, notebooks, or pens)	Regular loose-leaf paper (non-waterproof) with little to no post-consumer content Laundered cloth rags (no fabric softener)
Recycled paper products (e.g. paper towels and notebook paper)	
Aluminum foil	Do not use
Kim [®] wipes	Laundered cloth rags (no fabric softener)
Decon 90 [®] decontamination soap	Alconox [®] , Citranox [®] , and/or Liquinox [®]
Wash/rinse water that may be	Commercially available deionized water in an
Chemical (blue) ice packs	Regular wet ice doubled bagged (Ziploc [®]); wet ice made from municipal tap water



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Reference: SOP1001

Low Flow Groundwater Sampling Activities

This Standard Operating Procedure (SOP) supports a method which minimizes the amount of impact the purging process has on the groundwater chemistry during sample collection and to minimize the volume of groundwater that is being purged and disposed.

Prior to conducting the field work, information on the construction of the well and well development should be obtained and that information factored into the site specific sampling procedure. Stabilization of the water-quality indicator parameters is the criterion for sample collection. The specific information on what took place during purging must be recorded in the field notebook or in the groundwater sampling log.

Equipment

Depth-to-water measuring device - An electronic water-level indicator with marked intervals of 0.01 foot.

Interface probe for determination of liquid products (NAPL) presence, if needed.

Sampling pump - Adjustable rate, peristaltic pump

Tubing - food-grade, polyethylene tubing.

Multi-Parameter meter with flow-through-cell - water-quality indicator parameters which must be monitored include pH, ORP, DO, turbidity, specific conductance, and temperature. The multi-parameter meter should be calibrated with appropriate calibration waters prior to sampling each day. The inlet of the flow cell must be located near the bottom of the flow cell and the outlet near the top.

Decontamination Supplies - documented source of distilled water, detergent/non-phosphate soap, spray bottles, paper towel, brushes, and buckets or decontamination tubes for pumps.

Sample bottles, sample preservation supplies, labels, and chain of custody forms.

Well construction data and field data and water quality data from the previous sampling event.

Field notebook, groundwater sampling logs.

Personal protective equipment (PPE) specified in a site Health and Safety Plan.

A 5-gallon pail or container to contain the purged groundwater and for ultimate transport to a 55-gallon steel drum.

Materials of the sampling equipment (bladders, pumps, tubing, and other equipment that comes in contact with the sample) should be limited to stainless steel, glass, and other inert material. This will reduce the chance of the sampling materials to alter the groundwater where concentrations of the site contaminants are expected to be near the detection limits.

Sampling Procedures

Sampling locations must begin at the monitoring well with the least contamination, generally up-gradient or furthest from the site or suspected source. Then proceed systematically to the monitoring wells with the most contaminated groundwater. Check and record the condition of the monitoring well for damage or evidence of tampering. Lay out polyethylene sheeting around the well to



HYDE ENVIRONMENTAL, INC.

Reference: SOP1001

Low Flow Groundwater Sampling Activities

minimize the likelihood of contamination of sampling/purging equipment from the soil. Place monitoring, purging and sampling equipment on the sheeting. Unlock monitoring well. Record location, time, date, and appropriate information in a field logbook or on the groundwater sampling log. Remove inner casing cap.

Refer to the available monitoring well information and record the depth of the pump or tubing intake in the field logbook or groundwater sampling log. Measure the water level (water level must be measured to nearest 0.01 feet) and record information on the groundwater sampling log, leave water level indicator probe in the monitoring well. Connect the downhole tubing to clean, silicone tubing placed in the peristaltic pump to a flow-through cell. The discharge line from the flow-through cell must be directed to a container to contain the purge water during the purging and sampling of the monitoring well. Start pumping the well at a low flow rate (0.2 to 0.5 liter per minute) and slowly increase the speed. Check water level. Maintain a steady flow rate while maintaining a drawdown of less than 0.33 feet. If drawdown is greater than 0.33 feet lower the flow rate. 0.33 feet is a goal to help guide with the flow rate adjustment. It should be noted that this goal may be difficult to achieve under some circumstances due to geologic heterogeneities within the screened interval, and may require adjustment based on site-specific conditions and personal experience. Continue purging, monitor and record water level and pump rate every five (5) minutes to 10 during purging. During the purging, a minimum of one tubing volume (including the volume of water in the pump and flow cell) must be purged prior to recording the water-quality indicator parameters; turbidity, DO, specific conductance, pH, ORP, and temperature.

The stabilization criterion is based on three (3) successive readings of the water quality field parameters; the following are the criteria which must be used: Parameter Stabilization Criteria Reference pH ± 0.1 pH units, specific electrical conductance (SEC) $\pm 3\%$ FS/cm, ORP ± 10 millivolts, turbidity $\pm 10\%$ NTUs (when turbidity is greater than 10 NTUs), DO ± 0.3 milligrams per liter. Once the criteria have been successfully met indicating that the water quality indicator parameters have stabilized, then sample collection can take place.

If a stabilized drawdown in the well can't be maintained at 0.33 feet and the water level is approaching the bottom of the screened interval, reduce the flow rate or turn the pump off (for 15 minutes) and allow for recovery. Under no circumstances should the well be pumped dry. Begin pumping at a lower flow rate, if the water draws-down again turn pump off and allow for recovery. If two (2) tubing volumes (including the volume of water in the pump and flow cell) have been removed during purging then sampling can proceed next time the pump is turned on. This information should be noted in the field notebook or groundwater sampling log with a recommendation for a different purging and sampling procedure. Maintain the same pumping rate or reduce slightly for sampling (0.2 to 0.5 liter per minute) in order to minimize disturbance of the water column. Disconnect the pump's tubing from the flow-through-cell so that the samples are collected from the pump's discharge tubing. All sample containers should be filled with minimal turbulence by allowing the groundwater to flow from the tubing gently down the inside of the container.

After sample collection, disconnect the tubing that extends from the monitoring well and close and lock the well.



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Reference: SOP1001

Low Flow Groundwater Sampling Activities

Decontamination Procedures

Decontamination procedures for the water level meter and the water quality field parameter sensors. The electronic water-level indicator probe/steel tape and the water-quality field parameter sensors will be decontaminated by the following procedures: 1. The water-level meter will be hand washed with phosphate free detergent and a scrubber, then thoroughly rinsed with distilled water. 2. Water quality field parameter sensors and flow-through cell will be rinsed with distilled water between sampling locations. No other decontamination procedures are necessary or recommended for these probes since they are sensitive. After the sampling event, the flow cell and sensors must be cleaned and maintained per the manufacturer's requirements.

Quality Control

Quality control (QC) samples must be collected to verify that sample collection and handling procedures were performed adequately and that they have not compromised the quality of the groundwater samples. The appropriate EPA program guidance must be consulted in preparing the field QC sample requirements for the site-specific Quality Assurance Project Plan (QAPP). Primary areas of concern for quality assurance (QA) in the collection of representative groundwater samples include:

Obtaining a groundwater sample that is representative of the aquifer or zone of interest in the aquifer. Verification is based on the field log documenting that the field water-quality parameters stabilized during the purging of the well, prior to sample collection.

Ensuring that the purging and sampling devices are made of materials, and utilized in a manner, which will not interact with or alter the analyses.

Ensuring that results generated by these procedures are reproducible; therefore, the sampling scheme should incorporate co-located samples (duplicates).

Preventing cross-contamination. Sampling should proceed from least to most contaminated wells, if known. Field equipment blanks should be incorporated for all sampling and purging equipment, and decontamination of the equipment is therefore required.

Properly preserving, packaging, and shipping samples. All field QC samples must be prepared the same as regular investigation samples with regard to sample volume, containers, and preservation. The chain of custody procedures for the QC samples will be identical to the field groundwater samples.

The following are QC samples which may be collected during the sampling event:

Equipment/Rinsate Blank: A sample that is collected by pouring over or running analyte-free water through the sample collection equipment after decontamination and before sample collection. The sample is collected in the appropriate sample container with the proper preservative, identical to the samples. This represents background contamination resulting from the field equipment, sampling procedure, sample container, preservative, and shipment.



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Reference: SOP1001

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Field Blank: In the field, analyte-free water is collected into a sample container with preservatives. The sample containers are the same lot used for the environmental samples. This evaluates contamination introduced from the sample container(s) with applicable preservatives.

Field Replicate/Duplicate: Two (2) or more samples collected at the same sampling location. Field replicates should be samples collected side by side or by collecting one sample and immediately collecting the second sample. Field replicates represent the precision of the whole method, site heterogeneity, field sampling and the laboratory analysis.

Field Split Samples: Two (2) or more representative subsamples taken from one environmental sample in the field. Prior to splitting, the environmental sample is homogenized to correct for sample heterogeneity that would adversely impact data comparability. Field split samples are usually analyzed by different laboratories (interlaboratory comparison) or by the same laboratory (intra-laboratory comparison). Field splits are used to assess sample handling procedures from field to laboratory and laboratory's comparability.

Laboratory QC Samples: Additional samples will be collected for the laboratory's quality control: matrix spike (MS), matrix spike duplicate (MSD), laboratory duplicates, etc.

Shipping Container Temperature Blank: A water sample that is transported to the laboratory to measure the temperature of the samples in the cooler.

Trip Blank: A sample collected at the laboratory using analyte free water in the appropriate sample container with the proper preservative, taken out to the field, and returned to the laboratory for analysis without being opened. Trip blanks are generally for volatile organic compounds, low level metals, and gasoline range hydrocarbon samples. Used to assess contamination introduced during sample transport.

Health and Safety

Depending on the site-specific contaminants, various protective programs must be implemented prior to sampling the first well. The site Health and Safety Plan should be reviewed with specific emphasis placed on the protection program planned for the sampling tasks. Standard safe operating practices should be followed, such as minimizing contact with potential contaminants in both the liquid and vapor phase through the use of appropriate personal protective equipment. Avoid skin contact with, and incidental ingestion of, purge water. Use protective gloves and splash protection. At a minimum, skin protection will be afforded by disposable protective clothing, such as nitrile gloves or Tyvek®. Physical hazards associated with well sampling include lifting injuries, use of sharp objects for cutting discharge hose, heat and cold stress, and slip, trip, and fall conditions.

Post-Sampling Procedures

Several activities need to be completed and documented once groundwater sampling has been completed. These activities include, but are not limited to: 1. Ensure that all field equipment has been decontaminated and returned to proper storage location. Once the individual field equipment has been decontaminated, tag it with date of cleaning, site name, and name of individual responsible, all



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sample paperwork should be processed, all field data should be compiled for site records, and all analytical data when processed by the analytical laboratory, should be verified against field sheets to ensure all data has been returned to sampler.

* Parts of this SOP were collected from U.S. EPA Low Stress (Low Flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells, September 19, 2017

Reviewed/Approved

By (typed): James C. Lindemann

Signature:

A handwritten signature in black ink, appearing to read 'James C. Lindemann', is written over a horizontal line.

Title: President, Hyde Environmental, Inc.

Date: November 10, 2022



HYDE ENVIRONMENTAL, INC.

Reference: SOP1003

Investigation Derived Waste Handling

This Standard Operating Procedure (SOP) presents guidance for the management of investigation-derived wastes (IDWs).

IDWs are discarded materials resulting from field activities such as sampling, surveying, drilling, excavation, and decontamination processes. IDWs may include purged groundwater, solutions from decontaminating sampling equipment; and other wastes or supplies used in sampling. Personal protective equipment (PPE) and other solid waste (paper towels, plastic sheeting, etc) are not considered IDW and will be disposed as solid waste.

Predetermine staging areas for IDW containers (i.e. 5-gallon pail, 55-gallon steel drum). Stage containerized IDW awaiting results of chemical analysis at a pre-determined, secure location on the site. A label shall be applied to each drum using indelible marking. Labeling or marking requirements for IDW include project name, generation date, location of waste origin, and contents (i.e., purge water). Store containers such that the labels can be easily read. Determine the methods and personnel required to safely transport IDW containers to the staging area before field mobilization. Handling and transport equipment will be consistent with the associated weight for both lifting and transporting. All 55-gallon drums will be stored at a centralized on-site location that is readily accessible for vehicular pick-up. Drums will be closed during storage and be in good condition.

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HYDE ENVIRONMENTAL, INC.

Reference: SOP1002

Groundwater Sampling Data Verification and Validation

This Standard Operating Procedure (SOP) is applicable to data verification and validation as described in the following. An example data verification/validation checklist is attached (*IDQTF, UFP-QAPP Manual VI, March 2005*).

Field-Generated Data - Field-generated data are recorded in field logbooks by field sampling personnel in real time. Water level measurements and field parameters will be collected as a key element of the low-flow groundwater sampling process. Prior to collecting a groundwater sample, field-generated data will be verified to determine if stabilization criteria were met and that there were no uncommon conditions noted.

Laboratory-Generated Data - The laboratory provides electronic copies of the deliverable via secured web site. The data are entered into Hyde's database (Microsoft Excel table) directly from the lab reports by Hyde, and the data entry is checked by a separate individual from Hyde. Hyde does not perform data transformation or calculations that would alter the data values received from the laboratory. The analytical data report is then reviewed by a Hyde data reviewer/validator, who are independent of individuals who are directly involved in the sample collection process, to perform data verification/data validation and data assessment in accordance with the procedures described below. If any corrections need to be made to the deliverable, they will be made at that time, and a corrected deliverable will be resent by the laboratory.

Data verification and validation will be performed to ensure that the data are defensible and of known quality. Validation will be independent of both the project laboratory and individuals who are directly involved in the sample collection process. Data validation will include a review of the laboratory data report, including the narrative, analytical results, method and instrument quality control results, chain-of-custody sheets, raw data, and laboratory logs. It also includes an evaluation of quality assurance/quality control (QA/QC) samples (i.e. field duplicate, equipment/rinsate blank), and reporting limit adequacy. Data verification does not include review of any raw data or laboratory logs, but includes the other elements cited above. The verification and validation process will result in a conclusion about the usability of the data.

Verification and validation criteria, along with a list of data qualifiers will be as specified in the laboratory's quality manual. These qualifiers are generally based on the National Functional Guidelines for Data Review, but with added suffixes to provide additional information regarding the control limit that was exceeded. Data verification will be prepared by an individual under the direction of the QA Manager.

Data values that are significantly different from the population are referred to as "outliers". Outliers can result in improper sampling or analytical methodology, matrix interferences,



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Groundwater Sampling Data Verification and Validation

errors in data transcription, or real (but extreme) changes in analytical parameters. Errors found during data verification/validation will be identified and corrected prior to submittal of the project report. Analytical results that are rejected will be discussed with the USEPA on a case-by-case basis. If wells are re-sampled due to rejected data, only the new data will be presented in reports and used in decision making. If sampling data is rejected, but no re-sampling is performed, the data will still be presented in reports (with an "R" flag) and with explicit cautionary remarks regarding any use of the data.

Precision and accuracy will be adequately assessed through the data verification and validation process as described in the previous section. Sensitivity of the data will be evaluated through verification that the laboratory met the required reporting limits. Representativeness and comparability will be achieved through adherence to the QAPP-specified field and laboratory analytical procedures.

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